Monitoring and Control of Biological Textile Wastewater Treatment Using Artificial Neural Networks

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A dissertation submitted to the University of Glamorgan
in part fulfilment for the award of the degree of Doctor of Philosophy

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July 2002

To Henrique

Certificate of Research

This is to certify that, except where specific reference is made, the work described in this thesis is the result of the candidate. Neither this thesis, nor any part of it, has been presented, or is currently submitted, in candidature for any degree at any other University.

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Abstract

This thesis is concerned with the development of an artificial neural network based control scheme (ANNBCS) to improve the performance of a combined anaerobic and aerobic treatment process for textile industrial effluents. The ANNBCS acquired the required input data from on-line sensors, processed this information and when necessary, suggested suitable remedial action(s) for the treatment process. The objective of the ANNBCS was to take remedial actions that would ensure consistent treatment efficiency whilst meeting discharge consents and reducing operation costs.

The most appropriate types of artificial neural networks (ANNs) were selected for use in the control scheme from tests on a range of ANNs. The analysis was carried out with data that was obtained from a fluidised bed anaerobic digester fed with a synthetic baker's yeast wastewater (from another project). The data reflected various operating conditions of the digester such as: steady state, sudden changes in the organic load and sensor failure conditions. The networks that were investigated included the linear, backpropagation (BP), radial basis function (RBF), Elman, and self-organising map (SOM). The following criteria were used to select the best performing ANN: (i) accuracy of the network predictions; (ii) time required for the necessary training; (iii) the size of the training data. The off-line predictions made by each ANN were accurate enough to be used although a feedforward (FF) multi-layer Perceptron (MLP) network trained with a BP algorithm proved to be the most suitable candidate. The control scheme also incorporated a SOM whose function was to classify the incoming data before passing the information to an appropriately trained BP network.

A comprehensive set of experiments were conducted on a 30 l up-flow anaerobic sludge blanket (UASB) reactor, in conjunction with a 20 l aerobic tank, and a 3.75 l aerobic settler using a cotton simulated textile effluent (STE). The STE included among other components, a sizing agent (potato starch) and a reactive red azo dye. The experiments were designed to define the most appropriate on-line measurements and also remedial actions to be taken by the ANNBCS. The experiments consisted of operating both processes systematically under varying organic and colour load conditions.

Part of the data gathered from the described experiments was used to train and test off-line, in a computing environment, four control schemes in a progressive manner in order to see which one would better cope with sensor loss. Preliminary results demonstrated that a hybrid structure containing a learning vector quantization (LVQ) (replacing the SOM) followed by a series of BP networks was the most efficient of those tested at dealing with different load conditions whilst being least influenced by sensor failure.

Subsequent to the comprehensive set of experiments described above, the ANNBCSs were tested on-line. One experiment controlled a colour step change in load and BA for the UASB reactor (i.e. LVQ + BPs) and the second controlled an organic step change in load for the aerobic stage. In the last case only BP networks were used since there was no need for a classification network. Further evaluation of the ANNBCS capabilities, namely the response to an organic step change in load, took place in simulation using neural network auto-regressive exogenous (NNARX) models built to represent the UASB reactor during particular organic and colour loads. This testing further demonstrated the robustness of the ANNBCS.

Acknowledgements

I would like to acknowledge Dr. Steve Wilcox (Director of Studies), my supervisors Prof. Freda Hawkes, Prof. Dennis Hawkes, and also my colleagues Dr. Richard Dinsdale, Dr. Alex Chong, and Mr. Premier, for their invaluable guidance and help throughout the research project.

I would also like to thank my family and friends, because without their patience and support I would not have overcome the frustrations of this working period. I would like to thank my son for allowing me to be absent of his life for a period of 1.5 years.

I would like to show my appreciation to everyone at the School of Technology, especially to Prof. John Ward for his help on financial matters during part of the writing-up stage, to Mr. Tony Evans for doing the engineering drawings of the filters and to Mr. Gareth Betteney for his advise on electronic matters. Within this School I would like to express my heartfelt appreciation to all colleagues at the lodge and at the Wastewater Treatment Laboratory namely Miss Helen Forsey, Miss Lesley Farley, Miss Claire Furlong, Dr. Sarah Martin, and in particular to Miss Kirsty Veitch for assisting my work during my maternity leave and Dr. Cliona O'Neill for providing the OECD waste and also the STE for most of the duration of this work.

I am also in debt to the staff from the School of Applied Sciences, namely, Mr. Iauen for his help in electronic matters, Mr. Norman for performing the X-ray analyses and Mr. Michail Morfano for his help in taking the microscopic photographs.

This work was funded by the European Commission under its Environment and Climate Programme (ENV4-CT95-0064). The author would like to express their gratitude to BPB Paperboard Davidson Mill, Aberdeen for the provision of anaerobic granules and the Welsh Water for provision of activated sludge.

Nomenclature

ADMI	American Dye Manufacturers' Institute
AI	Artificial Intelligence
ANN(s)	Artificial Neural Network(s)
ANNBCS(s)	Artificial Neural Network Based Control Scheme(s)
APHA	American Public Health Association
ARX	Auto-Regressive eXogenous
ASM(s)	Activated Sludge Model(s)
ASSE(s)	Average Sum Squared Error(s)
b	Bias matrix
BA	Bicarbonate Alkalinity
BOD	Biological Oxygen Demand (standard 5 days measurement)
BOD_{st}	Short-time BOD
BP	Back-Propagation
BS	British Standard
B_v	Volumetric loading rate
CI	Colour Index
COD	Chemical Oxygen Demand
CSTR	Completely Stirred Tank Reactor
DI	DeIonised
DNA	DeoxyriboNucleic Acid
DO	Dissolved Oxygen
ES(s)	Expert System(s)
EU	European Union
F	Transfer function of the neurones in each network layer
F:M ratio	Food to Microorganisms Ratio
FAS	Ferrous Ammonium Sulphate
FF	Feed-Forward
FFN(s)	Feed-Forward Network(s)
FID(s)	Flame Ionisation Detector(s)
GAC	Granular Activated Carbon
GC	Gas Chromatograph
GMI	Gas Measurement Instruments
HPLC	High Performance Liquid Chromatography
HRT(s)	Hydraulic Retention Time(s)
IAWQ	International Association on Water Quality
IC	Inorganic Carbon
ICA	Instrumentation, Control and Automation
LVQ	Learning Vector Quantization
MIMO	Multi-Input Multi-Output
MISO	Multi-Input Single-Output
MLP	Multi-Layer Perceptron
MLSS	Mixed Liquor Suspended Solids
MS	Mass Spectrometry
n	number of samples
n_a	Number of past ouput data
n_b	Number of past input data
NDIR	Non-Dispersive Infra Red
n_k	Number of delay(s) associated with an input
NNARX	Neural Network Auto-Regressive eXogenous
NSSE	Normalised Sum Squared Error
OD(s)	Optical Density(s)
OECD	Organisation for Economic Cooperation and Development

ORP	Oxidation Reduction Potential
OUR(s)	Oxygen Uptake Rate(s)
p	Partial pressure
PAC	Powered Activated Carbon
PC(s)	Personal Computer(s)
PID	Proportional Integral Derivative
PRBS	Pseudo Random Binary Signal
RAS	Return Activated Sludge
RBF	Radial Basis Function
RBFN	Radial Basis Function Network
RN(s)	Recurrent Network(s)
RODTOX	Rapid Oxygen Demand and Toxicity Tester
RPM	Revolution(s) Per Minute
RTC	Real Time Control
SCA	Specific Catalase Activity
sd	Standard Deviation
SEM	Scanning Electron Microscopy
SISO	Single-Input Single-Output
SOM	Self Organising Map
SOUR	Specific Oxygen Uptake Rate
SRT	Sludge Retention Time
SS	Suspended Solids
SSE(s)	Sum Squared Error(s)
STE(s)	Simulated Textile Effluent(s)
STP	Standard Temperature and Pressure
SVI	Sludge Volume Index
t	Time expressed in terms of the sampling period
TA	Total Alkalinity
TC	Total Carbon
TCD(s)	Thermal Conductivity Detector(s)
TCU	True Colour Units
TOC	Total Organic Carbon
TOD	Total Oxygen Demand
TS	Total Solids
TSS	Total Suspended Solids
TVA	Total Volatile Fatty Acids Alkalinity
TVFA	Total Volatile Fatty Acids
u	Input vector of the network architecture
UASB	Upflow Anaerobic Sludge Blanket
UV	Ultra Violet
VFA(s)	Volatile Fatty Acids(s)
VI	Virtual Instrument
VS	Volatile Solids
VSS	Volatile Suspended Solids
w	Weight matrix
WAS	Waste Activated Sludge
WW(s)	WasteWater(s)
WWT	WasteWater Treatment
WWTP(s)	WasteWater Treatment Plant(s)
y	Output vector of the network architecture

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1. INTRODUCTION

This Chapter contains four sections. It describes what are the main problems, which are evident from the literature, and draws the main objectives of the work in order to try to minimise the difficulties encountered in such areas of study. It also summarises the context of this work within the European Union (EU) project. Finally, it summarises the structure of the thesis.

1.1. Problem Definition

Increasing industrial and domestic water consumption is leading to potential water shortages and consequently increasing costs within many European countries. A particularly good example is the textile industry where large volumes of water are used (Burkinshaw and Graham, 1995) and discharged to the environment. This has, in part, led to the need for higher effluent standards by the EU. This makes the textile industry a good candidate for the development of water recycling and emission abatement systems.

In many textile factories the wastewater (WW) flow is variable and intermittent and may only occur during weekdays. The composition of the WW from the textile industry is determined by the processes (Correia *et al.,* 1994), fibre type and chemicals used and therefore varies considerably, making this waste difficult to characterise. Generally, it consists of large volumes containing high concentrations of organic and inorganic chemicals and is typically coloured due to the presence of dyes (Altinbas *et al.,* 1995).

Colour removal from textile WWs is one of the most difficult problems for environmental engineers when designing a treatment process (Lin and Lin, 1993). This is because the wastewater treatment (WWT) process is designed for steady loads and changes that occur in the colour concentration lead to the inefficient treatment of the WW, which is very visible (McCurdy *et al.*, 1992).

Biotreatment processes, namely anaerobic and aerobic, have been successfully used to treat WW from this industry (e.g. Basibuyuk and Forster, 1997). Anaerobic digestion is useful in the treatment of textile WW as it removes some organic pollution and decolourises the commonly used azo dyes (Delée *et al.*, 1998). Azo dyes are reduced in the anaerobic stage while acting as electron acceptors in the degradation of other WW compounds. Usually the starch commonly used in sizing cotton textiles provides the electron (Carliell *et al.*, 1995). An aerobic stage is needed to remove more organic compounds including aromatic amines generated from azo bond reduction in the anaerobic stage.

There are three main advantages to be gained from controlling biotreatment processes. Reduction of capital costs, running costs and consistent compliance with discharge consents. However, there are some difficulties that are encountered when controlling these systems:

1. Lack of on-line reliable sensors for the WWT industry and fast remedial actions to be applied in case of treatment failure have been experienced. The unreliability of on-line sensors comes from their need for on-line sampling and filtration, and quite large maintenance requirements (Jaconbsen and Jensen, 1998; Steyer *et al.*, 2002).

2. It is well known that anaerobic digesters as a biological process are difficult to control over long periods of time, largely because of the complex and poorly known, non-linear, time varying dynamics (Emmanouilides and Petrou, 1997; Moletta *et al.*, 1994). However, parameters such as the mixing rate, the bacterial culture, nutrients, pH and buffering capacity must be maintained in order to ensure the treatment efficiency (Speece, 1996). Similarly, activated sludge processes have also encountered control problems due to their non-linear and dynamic nature (Tyagi *et al.*, 1993; Spanjers *et al.*, 1998).

A conventional on-off or proportional integral derivative (PID) control system would fail catastrophically if the sensor monitoring the controlled variable, such as pH, fails and special provision should be made for this. In many applications sensors are reliable but as mentioned earlier, in WWT sensor failure can be a regular occurrence and so the control system must be robust enough to continue operating in such circumstances. Ideally the control system would perform at the same efficiency, however, with some information lost to the input of the controller when a sensor fails. What should be possible to achieve is for the control system performance to degrade smoothly, thus allowing some time for plant personnel to repair or replace the failed sensor and so restore the monitoring capability.

An Artificial Neural Network (ANN) should be an ideal candidate to control such a biological system due to such features as non-linear transfer functions, no need for a full knowledge of the biochemistry process, ability to generalise thus permitting training by specific examples and some toleration to sensor loss. An ANN uses a distributed representation of the external world and exhibits graceful degradation in performance when the network encounters a problem outside the range of experience. The ANN approach requires no explicit encoding of knowledge as in an Expert System (ES), which makes a neural approach well suited to applications in which knowledge extraction is difficult or in cases where the interrelationships between process parameters are hard to model. Despite the potential of ANNs they are still not widely used in biotechnology (Collins, 1990) partly because it is not possible to ask the network how it arrived at a particular conclusion. The 'black-box' approach is often criticised especially when used as a model, although the nature of a mathematically based model may also have deficiencies. Mathematical models can also be empirical when they need to include 'correction factors' to make them fit reality more closely.

ANNs have been successfully used for a number of chemical engineering applications including sensor data analysis and fault detection. There is evidence in the literature that ANNs could be ideal for use in WWTPs, both anaerobic and aerobic systems, as reviewed in the Chapter 2.

1.2. Aims and Objectives of the Work

The main aim of the work presented in this thesis was to develop an ANN based control scheme (ANNBCS) to improve the performance of two biological treatment processes (i.e. anaerobic and aerobic) for textile industrial effluents including situations of colour and organic overloads and also sensor failure conditions. This ANNBCS would use data from appropriate on-line instrumentation and would output suitable remedial actions to the treatment process(es). Other objectives were inherent to this project, such as:

- Undertake a detailed investigation of the plant by monitoring on-line and off-line the performance when treating a simulated textile effluent (STE). These include the assessment of a few parameters.
- Define appropriate remedial actions.
- Test a range of different ANNs in order to select the most appropriate one(s) to be applied in an ANNBCS.
- Test on-line the performance of an ANNBCS when controlling the biological treatment processes.

1.3. EU Based Project

This work was undertaken as part of a much larger EU project entitled 'Integrated water recycling and emission abatement in the textile industry'. The project included the participation of nine main partners from six European countries (i.e. Austria, Belgium, Italy, France, Portugal, and the UK). The overall project objectives were to develop an integrated process for the provision of recycled water of assured quality with minimum emissions. Three treatment modules, which could be used individually or together were developed, namely a: combined sorption/anaerobic digestion stage for heavy metal and dye removal; sensor protected aerobic stage; and finally a module for holistic polishing (granular activated carbon (GAC) adsorption or membrane filtration). The project also included an assessment of the economic and environmental aspects of the process in the different member states. The main goal of the project was to attain a reduction in clean water intake to the textile industry by as much has 50-75 %, and of final discharge of sludge to landfill by up to 60 % in addition of a recovery of heavy metals of up to 80 %. This was to minimise emission discharge and significantly reduce the water brought in from outside the company. The work presented in this thesis contributed to the development of an ANNBCS for the industrial textile WWT using two biological treatment processes: anaerobic and aerobic.

1.4. Structure of the Thesis

Chapter 2 of this thesis provides a critical review of the literature published on areas of the treatment process biochemistry, instrumentation, conventional control approaches versus artificial intelligence (AI) for the modelling and control of biological treatment systems, and ANNs applied for the detection of failure conditions. Chapter 3 describes the apparatus and procedures used to carry out the work and also includes the experimental design for the five Experimental Phases. Chapter 4 presents the data collected during four Experimental Phases and the importance of each monitoring parameter when applied in a control scheme is highlighted. Chapter 5 discusses the selection of an appropriate ANN for control, the development of the ANNBCS and its application on-line to the anaerobic and aerobic stages. Chapter 6 presents the use of neural network auto-regressive exogenous (NNARX) models that represent the upflow anaerobic sludge blanket (UASB) reactor in a computer simulation. It also includes a further evaluation of the ANNBCS when controlling the UASB reactor models for situations not viable to be tested in the real laboratory scale plant. Finally, Chapter 7 draws the main conclusions and defines areas for further work.

2. LITERATURE REVIEW

This Chapter reviews WW pollution from the textile industry and the importance of pollution control and biological treatment methods. The biochemistry and microbiology of anaerobic and aerobic processes are briefly described, covering also a review on the main monitoring parameters used in order to control such treatment processes. Finally, the use of AI mostly ANNs as a tool to model and control such processes is also reviewed.

2.1. Pollution from the Textile Industry

It was estimated that in Europe the textile industry consumed 2 – 5 % of the total industrial water consumption and was therefore among the world's ten largest industrial consumers of water (Durig, 1981). Water consumption for industrial processes varied between 3 – 9 l kg^{-1} for cotton desizing to 334 - 835 l kg^{-1} for wool washing (Correia *et al.*, 1994). Considering both volume and effluent composition, the WW discharged by the textile industry was rated the most polluting among all industrial sectors (Reid, 1996). This makes it a good candidate for the development of water recycling and emission abatement systems.

Cotton is the world's principal fibre type, it accounts for approximately 50 % of the 40 million tonnes world fibre consumption (Holme, 1997). Collishaw *et al.* (1992) cited that the cotton consumption in 2000 would increase to 23 million tonnes. It can be assumed that dye usage would rise in proportion, increasing the discharge of colour and other related pollution.

A history of dyes in the textile industry can be found on the Internet (Druding, 1998). Some 3000 dyes have been used, however, the total number of formulations was approximately 7000 (Laing, 1991). Modern textile dyes are required to have a high degree of chemical and photolytic stability (Easton, 1995) so that it maintains its structure and colour and to resist breakdown due to time and exposure to sunlight, water, soap (McCurdy *et al.*, 1992), washing, bleach and perspiration (Travis, 1993) among other factors. Dyes may be classified according to their chemical structure (e.g. azo and anthaquinone), or by their usage or application method (e.g. reactive and disperse) (Kirk-Othmer, 1993). Very often, both terminologies are used. However, classification by application is the principal system adopted by the Colour Index (CI) (Renfrew and Taylor, 1990). A generic name and then a CI constituent number based on the chemical structure is assigned e.g. CI Acid Blue 80. Dyes can also be named by their commercial trade name, which is usually made up of three parts; a trademark to designate the manufacturer and the dye class, colour, and a series of letters and numbers used as a code for precise definition e.g. Procion Red H-E7B. There are about 12 classes of chromogenic groups, the most common being the azo type (Kirk-Othmer, 1993) accounting for 60-70 % of all textile dyestuffs produced (Carliell *et al.,* 1995). Therefore, the importance of this chemical class in textile dyeing is apparent. Azo dyes are coloured chemical compounds containing one or more azo chromogens (-N=N-). Reactive dyes, which belong to the azo chemical type combined with different types of reactive groups, made possible to achieve extremely high 'washfastness' properties by relatively simple dyeing methods (Kirk-Othmer, 1993). Their chemical structures are much simpler, their absorption spectra show narrower absorption bands, and the effects of the dye are brighter (Siegel, 1972). Reactive dyes are the most used e.g. in Loughborough Catchment and Wanlip Catchment 60.8 and 66.7 % of the dyes were reactive from 8 different classes of dyes, respectively (Churchley, 1994).

The dyeing of cotton with reactive dyes occurs by exhaustion of the dye onto the cloth in the presence of an electrolyte such as salt (sodium chloride or sodium sulphate) at neutral pH; fixation of the dye to the fibres at an alkaline pH; and washing of the cloth to remove electrolyte, alkali and unfixed dye (Shamey and Nobbs, 1997). The degree of fixation for different dye and fibre combinations varies considerably. For the reactive class, cellulose fibre there is a 50 – 90 % of fixation and 10 - 50 % loss to the effluent (Easton, 1995), comparing with 98 % of dye fixation in wool. Also, as mentioned earlier in many textile factories the WW flow is variable and intermittent and may only occur during weekdays and the textile WW is difficult to characterise (Correia *et al.,* 1994). The textile process includes normally sizing and desizing, weaving, scouring, bleaching, mercerising, carbonising, fulling, dyeing and finishing. The large number of compounds contained in textile effluent was demonstrated by Alaimo *et al.* (1990) who identified 314 compounds. Cotton processing effluents are highly coloured and have high concentrations of total dissolved solids (high concentration of salts), high biological oxygen demand (BOD) and high chemical oxygen demand (COD) (Correia *et al.*, 1994). Most dyes exhibit low BOD and low toxicity, and thus tend to be less polluting than substances such as sizing agents (mainly modified starch) and waxes and impurities from raw cotton (Durig, 1981). The use of acetic acid does not contribute to increased salinity but this component can account for 50-90 % of the dyehouse organic load (Laing, 1991). The major pollutant types identified in the textile WW, along with their main origin in the textile manufacturing process were summarised by Delée *et al.* (1998) (Table 2.1). Their general impact on the biological treatment is also presented.

Table 2.1 - Major pollutant in textile WWs, their origin and major impact in biological treatment (from Delée *et al.*, 1998)

Pollutants	**Major chemical types**	**Main origin**	**Impact on biological treatment**
Organic load	Starches, enzymes, fats, greases, waxes, surfactants Acetic acid	Desizing Scouring Washing Dyeing	High demand on aeration systems. Activated sludge bulking problems
Colour	Dyes; scoured wool impurities	Dyeing Scouring	Insufficient removal in bioreactors
Nutrients (N, P)	Ammonium salts, urea, phosphate-based buffers and sequestrants	Dyeing	Not removed in anaerobic processes Increased complexity and sensitivity of aerobic processes (biological nutrient removal required)
pH and salt effects	NaOH, mineral/organic acids, sodium chloride, silicate, sulphate, carbonate	Scouring Desizing Bleaching Mercerizing Dyeing Neutralisation	Inhibition/collapse of bioreactors
Sulphur	Sulphate, sulphide and hydrosulphite salts, sulphuric acid	Dyeing	Sulphate-reduction in anaerobic reactors
Toxicants	Heavy metals, reducing agents (e.g. sulphide), oxidising agents (e.g. chlorite, peroxide, dichromate, persulphate), biocides, quaternary ammonium salts	Desizing Bleaching Dyeing Finishing	Inhibition of sensitive microbial groups (nitrifiers, methanogens) in bioreactors
Refractory organics	Surfactants, dyes, resins, synthetic sizes, chlorinated organic compounds, carrier organic solvents	Scouring Desizing Bleaching Dyeing Washing Finishing	Insufficient removal in bioreactors. Possible accumulation in biomass aggregates/films, leading to inhibition

High dye concentrations in watercourses can affect the transmittance of light and thus may affect aquatic flora and fauna (Diaper *et al.*, 1996). Dyes exhibit low toxicity to mammals and aquatic organisms (Churchley, 1998). However, they may be of concern when the treated effluent is used as a supply of drinking water (Lloyd *et al.*, 1992). Also, the eye can detect concentrations of 0.005 mg l^{-1} of reactive dye in clean river water, hence consent levels for the discharge of colour to receiving waters are normally applied for aesthetic reasons and not for prevention of toxicity (Durig, 1981). Most complaints from the public concerning colour tend to refer to water containing red dyes (Smith, 1996), usually linked to the presence of reactive azo dyes in the water (Carliell *et al.*, 1995).

All textile companies are under pressure from increasing legislation and higher water and sewage charges both to clean up their effluents and to minimise water usage (Watson, 1991). Most water companies have been passing the problem of dye removal back to the dyehouse operator (Diaper *et al.*, 1996). This trend is likely to increase, as the legislation becomes stricter. For all these reasons on-site effluent treatment is more attractive. Holme (1997) stated that within the dyeing and finishing industry Germany had the highest environmental costs (10.1 % of total cost from which 50 % were attributed to the effluent treatment) followed by the UK (6.9 %).

2.2. Treatment Methods for Textile Effluents

Several textile WWT methods have been reviewed (Laing, 1991; Steenken-Richter and Kermer, 1992; Correia *et al.*, 1994; Cooper *et al.*, 1994; Hazel, 1995; Perkins, 1996; Churchley, 1998). These authors have compared the different treatment technologies based on parameters such as: performance according to standards, capital and running costs, volume capability, speed of decolourisation, potential for reutilisation of the effluent, and the various limitations and drawbacks, namely the impact on the environment.

Colour is one of the hardest compounds, in the textile WW, to remove (Lin and Lin, 1993; Boe *et al.*, 1993; Cowey, 1998). Therefore, recycling of exhausted dyebaths offers potential for reduced dyeing costs by lowering the consumption of water, chemicals and energy as well as reduced treatment costs. However, recycling is difficult in the case of the reactive dyeing of cotton as the dyeing behaviour of the residual dye differs from that of the fresh reactive dye (Burkinshaw and Graham, 1995). Removal of dyes and their intermediates can often be achieved only through a combination of treatments (McCurdy *et al.,* 1992). Hazel (1995) studied 30 different treatments and concluded that no simple technique would remove colour from a complex dye waste mixture to meet consent conditions. Thirty different methods of colour removal were also evaluated, and it was concluded that many of the techniques available required technological improvement and cost reduction to become commercially viable, although decolourisation is achievable using one or a combination of: adsorption, filtration, precipitation, chemical degradation, photodegradation, and biodegradation (Willmott *et al.*, 1998). Vandevivere *et al.* (1998) reviewed the performance using different treatment methods (Table 2.2).

Table 2.2 – Evaluation of various technologies for the treatment of textile effluents (from Vandevivere *et al.*, 1998)

Process	**Stage**	**Status**	**Performance**	**Limitations**
Fenton oxidation	Pre-treatment	Several full-scale plants in S. Africa	Full decolourisation; low capital and running costs	
Electrolysis	Pre-treatment	Pilot-scale	Full decolourisation; cheap	Foaming and electrode lifespan
Foam flotation	Pre-treatment	Laboratory-scale	Removes 90% colour and 40% COD; cheap, compact	
Filtration	Main or post-treatment	Extensive use in S. Africa	High performance; reuse of water, salts and heat	Handling and disposal of concentrate stream
Biodegradation				
Activated sludge	Main treatment	Widely used	Removes bulk COD, N	High residual COD, N, colour, surfactants
Sequential anaerobic/aerobic	Main treatment	Very few reports	Better removal of COD, colour, and toxicants	High residual colour and COD
Fixed-bed	Main treatment	Some pilot trials in China	Better removal of COD, colour	
Fungi/H_2O_2	Main treat.	Laboratory-scale	Full decolourisation	
Coagulation/flocculation	Pre- main or post-treatment	Extensive use	Full decolourisation; water reuse	Not always effective; sludge disposal
Ozone	Post-treatment	Full-scale	Full decolourisation; water reuse	Expensive; aldehydes formed
Sorption (carbon, clay, biomass)	Pre- or post-treatment	Laboratory or full-scale, depending on sorbent type	New sorbents are effective and cheap; water reuse	High disposal or regeneration costs
Photocatalysis	Post-treatment	Pilot-scale	Near-complete colour removal; detoxication	Only as final polishing step

2.3. Biological Treatment of Textile Effluents

Vandevivere *et al.* (1998) stated that since dyes are intentionally designed to resist degradation, it is no surprise that little dye degradation occurs in activated sludge systems. Many anaerobic bacteria, but only a few aerobic bacteria, were capable of azo dye reduction (Chung and Stevens, 1993). Several WWTPs based upon anaerobic and aerobic bacterial action have been developed to treat textile industry WW. Also the combination of biological treatments has been considered e.g. sequential anaerobic-aerobic degradation. Only biotechnological solutions can offer complete destruction of the dyestuff, with a co-reduction in BOD and COD (Willmott *et al.*, 1998). Athanasopoulos (1990) cited that acclimatisation describes the bacteria 'getting used to' a particular waste and as the time passes, the biomass may become active towards the waste with the generation of enzymes and/or metabolic pathways. However, biomass can also lose acclimatisation during plant shutdowns. Pretreatment of textile wastes before biological treatment could include any or all of the following: screening, sedimentation, equalisation, neutralisation, chrome reduction, coagulation or any of the other physical-chemical treatments. The performance of biological treatment depends on the BOD:COD ratio.

2.3.1. Treatment of Textiles Effluents Using Anaerobic Systems

Despite numerous laboratory-scale trials demonstrating the potential of anaerobic treatment for colour removal, large-scale installations equipped with anaerobic pre-treatment do not generally achieve full decolourisation (Vandevivere *et al.*, 1998; Cowey, 1998; Delée *et al.*, 1998). Using an anaerobic fixed bed reactor an almost complete decolourization of many dyes, an efficient COD removal and a digestion of substances that were refractory under aerobic conditions were obtained by Minke and Rott (1999). WW from cotton yarn and fabric finishing was treated in an anaerobic expanded bed reactor at 35 °C up to a COD loading of 0.63 kg m^{-3} day^{-1} (Athanasopoulos, 1992). The COD removal varied from 50 to 87 % with a CH_4 production of 70-80 %. A full-scale plant for full recycling of scouring water in operation at a Spanish wool combing plant consists of an anaerobic plant with 80 % COD removal followed by a distillation process (Wool Record, 1996).

COD removal can be easily inhibited by textile effluents (Athanasopoulos, 1992). Some anaerobic decolourisation of dyes may be due to adsorption of the dyes to the biomass. However, most decolourisation of dyes by anaerobic treatment is considered to occur by means of biological degradation. Anaerobic decolourisation was found to range from 0 to over 99 % (Delée *et al.*, 1998). Some dyes in the same class gave very different results in terms of anaerobic colour removal, which may be explained in terms of dye structure. Azo dyes are reduced and hence decolourised i.e. the chromophore is destroyed when acting as electron acceptors for the microbial electron transport chain. This is a four electron process which proceeds through two stages (Carliell *et al.*, 1994). The first reaction gives rise to a colourless compound which is unstable and may revert to its original coloured form under oxidising conditions, or it can be further reduced under anaerobic conditions to form stable colourless compounds i.e. stable aromatic amines (see equation below). It is believed that in many cases decolouration of reactive azo dyes under anaerobic conditions is due to the action of azo reductase enzymes (Willmott *et al.*, 1998).

$$R_1\text{-}N{=}N\text{-}R_2 + 4e^- + 4H^+ \xrightarrow{\text{Azo reductase}} R_1\text{-}NH_2 + R_2NH_2$$

Where R_1 and R_2 are aromatic components in dye molecules

A source of labile carbon is required as a source of reduction equivalents for dye decolourisation to occur (Carliell *et al.*, 1996; Razo-Flores *et al.*, 1997). Concentrations of 5 g l^{-1} of glucose, glycerol and lactose have been found to give colour removals of 82, 71 and 71 % respectively of 0.5 g l^{-1} Remazol Black B, while starch gave rise to 52 % decolourisation (Nigam *et al.*, 1996). However, starch is the only one of these co-substrates typically found in cotton effluent. Razo-Flores *et al.* (1997) stated that Mordant Orange 1 and Azodisalicylate were completely reduced and decolourised in continuous UASB reactors in the presence of co-substrates.

Carliell *et al.* (1994) proposed a range of anaerobic degradation products for Procion Red H-E7B which might theoretically be formed. It was confirmed by Carliell *et al.* (1995) that 2-aminonaphthalene-1,5,-disulphonic acid was present after anaerobic digestion of the dye thus showing that the azo bond had been cleaved. Brown and Hamburger (1987) confirmed that the aromatic primary amine metabolites from the reductive cleavage of azo bonds were formed. Dyes are not normally cytotoxic, mutagenic or carcinogenic, but the amines formed by anaerobic digestion may possess these characteristics (Zaoyan *et al.*, 1992; FitzGerald and Bishop, 1995). Therefore, further treatment is essential prior to discharge e.g. aerobic stage.

2.3.2. Treatment of Textiles Effluents Using Aerobic Systems

The aerobic treatment methods applicable to textile WW follow in order of increasing retention time: (1) trickling filters; (2) activated sludge; (3) rotating biological disks; (4) extended aeration; (5) lagoons; and (6) aquatic plants (Athanasopoulos, 1990).

Dyes do not inhibit activated sludge in the concentrations normally found in WW (Durig, 1981). The removal of dyes by sorption during biological treatment is referred to as bioelimination (Laing, 1991), the adsorbed dyes being destroyed during sludge disposal (Pierce, 1994). Sorption is influenced by factors such as pH but the extent of removal varies from dye to dye (Laing, 1991). Churchley (1994) explained that it is likely that on plants continuously or regularly treating dyewaste the adsorption sites will be rapidly occupied if the dye is strongly adsorbed. Renewal of adsorption sites will be by creation of new sludge flocs, de-sorption of the dye or biodegradation of the dye. With normal sludge ages the creation of new flocs is too slow to remove dyes adequately, and de-sorption leads to re-solubilisation of colour. Therefore, biodegradation of dyes in a sewage treatment activated sludge plant is slow (Churchley, 1994). Field *et al.* (1995) reported that some authors found that aerobic bacteria took 100 to 400 generations to adapt to the cleavage of carboxylated azo dyes. Goronszy and Tomas (1992) stated that the ability to remove azo dyes by activated sludge depends on the molecular structure and on the type, number and position of the substitution radicals in the ring structures. Reactive dyes are not readily degraded under aerobic conditions (Easton, 1995; Waters, 1995) and therefore the effluents are in most cases coloured upon leaving the plant. Laing (1991) reported that colour removal from textile effluents by activated sludge processes ranged from 10 to 80 %, typically being below 50 %. A number of authors reported elimination of reactive dyes by adsorption onto sludge ranging from 0–30 % (e.g. Pierce, 1994; Waters, 1995). Therefore treatment of reactive dyes in activated sludge plants results in highly coloured waste passing through the activated sludge plant to the receiving waters.

The by-products of the azo bond cleavage, aromatic amines, which are not further metabolised under anaerobic conditions are readily biodegraded in an aerobic environment (Field *et al.*, 1995). Domestic sewage buffers the pH of industrial effluent, dilutes inhibitory materials (Kroiss and Muller, 1999), and provides nutrients such as N and P which may be too low in the trade effluent to allow adequate bacterial activity (Churchley, 1994) and can therefore enhance degradation of textile effluents. One study found that textile effluent should be mixed with at least 50 % domestic sewage to compensate for nutrient deficiency in the former and hence produce a good quality final effluent by means of activated sludge treatment (Abo-Elela *et al.*, 1988).

2.3.3. Combined Anaerobic-Aerobic Treatment for Textile Effluents

Delée *et al.* (1998) concluded that the anaerobic stage requires aerobic post-treatment in order to complete the mineralisation of some pollutants (azo bond reduction products – aromatic amines, surfactants, residual BOD and remove nutrients). On the other hand, this aerobic post-stage benefits from the anaerobic pre-treatment in several ways, including the protection against BOD and toxic shock loads, the possibility of reductive decolourisation and dechlorination, the minimisation of foaming (Bortone *et al.*, 1995) and bulking problems and the reduction of aeration and sludge disposal costs. Anaerobic pre-treatment offers several potential advantages such as better removal of colour, adsorbable organic halogens, and heavy metals (Rigoni-Stern *et al.*, 1996).

Combinations of anaerobic and aerobic treatment have been carried out by a number of authors including Boe *et al.* (1993) and Basibuyuk and Forster (1997). Jianrong *et al.* (1994) achieved 90 % COD reduction and 96 % colour reduction in a laboratory-scale UASB reactor (8 h hydraulic retention time (HRT)) followed by an activated sludge reactor (6 h HRT) fed with a deeply coloured high strength effluent. The greatest fraction of the colour and COD reduction occurred in the UASB reactor. FitzGerald and Bishop (1995) implemented a laboratory scale sequential anaerobic-aerobic system to study mineralisation of three sufonated azo dyes. Results indicated good dye decolourisation levels greater than 65 % in the first stage and there was very little additional decolourisation in the second stage. COD removal in all three reactors of approximately 85 % in the first stage, with very little additional COD removal in the second stage. Zaoyan *et al.* (1992) operated a large pilot plant (anaerobic followed by an aerobic stage) at a textile dyeing mill which used reactive and other dye classes on polyester/cotton fabric. The sequential system achieved a higher colour removal (72 %) than the aerobic system alone (<60 %) but the final effluent was still deeply coloured. A sequential anaerobic-aerobic full-scale plant was operating at a major dyehouse in Hong Kong (Easton, 1995).

O'Neill *et al.* (2000a) used a combined anaerobic-aerobic treatment (overall HRT 1.8 days) to treat a STE with a reactive azo dye (Procion Red H-E7B) and starch. Most colour removal occurred in the UASB reactor and the BOD:COD ratio of the reactor effluent increased by up to 47 %. A maximum of 77 % colour removal overall was achieved with starch and dye concentrations of 3.8 and 0.15 g l^{-1}, giving a final true colour of 0.21 true colour units (TCU). However, at 3.8 g l^{-1} starch, volatile fatty acid (VFA) levels in the UASB reactor rose, while at 2.9 g l^{-1} starch they did not. O'Neill *et al.* (2000a) recommended that if colour removal efficiency decreases, carbohydrate should be added to the anaerobic reactor at a maximum sludge loading rate between 0.11 and 0.15 kg COD kg^{-1} volatile solids (VS) d^{-1}. The results obtained by O'Neill *et al.* (2000b) showed that azo-dye degradation actually occurred in the anaerobic stage forming aromatic by-products, which were removed by subsequent aerobic treatment. Some aerobic COD removal was likely to be attributable to degradation of these products. Similarly, Field *et al.* (1995) found that the anaerobic treatment could provide 97 % decolourisation and 60 % COD removal, and subsequent aerobic treatment removed an additional 30 % COD, thought to be due to the removal of aromatic amines.

2.4. Biological Wastewater Treatment – Need for Monitoring and Control

The use of on-line monitors providing information on key process parameters is crucial if WWTPs are to be operated more effectively. 'Sensors are the eyes and ears of the control system' as they tell us what is the process doing (Olsson and Newell, 1999). Information from such monitors would allow refinement of models and the application of process control strategies. A monitoring system for a biotreatment process must combine available on-line data, intermittent off-line measurements and the best available understanding of the mechanisms controlling process behaviour. The control system interfaces with the plant through sensors and actuators (e.g. valves and pumps). Sensors are the weakest part of the chain in real time control (RTC) of WWTPs, they are behind the hard and software technology (Lynggaard-Jensen *et al.*, 1996). Steyer *et al.* (2002) stated that the WWT field suffers from a major lack of sensors both reliable and highly informative. For example TOC, COD and VFA sensors are recognized as fragile measuring devices. Steyer *et al.* (2001) used a modified Fourier Transform Infrared spectrometer connected to an ultra-filtration membrane to provide precise measurements of soluble COD, TOC and VFA concentration as well as total alkalinity (TA) and partial alkalinity. Although sensor technology is improving (e.g. Vanrolleghem, 1995; Steyer *et al.*, 2002), in the harsh environment, maintenance will always be of concern (Nyberg *et al.*, 1996). Many of the commercially available sensors require sampling and filtration (Lynggaard-Jensen *et al.*, 1996). Also, sensors transmit noisy signals and therefore advanced signal processing and estimation are required. Desirable features of an on-line sensor are: measure a significant parameter; be low cost; need infrequent maintenance, not foul, periodic automatic re-calibration and internal diagnostics.

2.4.1. Anaerobic Digestion

Gujer and Zehnder (1983) suggested that the process of anaerobic digestion has six main metabolic stages, however, Mosey (1983) simplified the process into three main bioconversions: hydrolysis and acidogenesis, acetogenesis and methanogenesis. Hydrolysis of high molecular weight carbohydrates, fats and/or proteins into soluble polymers by means of the enzymatic action of hydrolytic fermentative bacteria and the conversion of these polymers into organic acids, alcohols, H_2 and CO_2. Organic acids and alcohols are then converted to acetic acid by the H_2-producing acetogenic bacteria and finally methanogenic bacteria convert acetic acid and H_2 gas into CO_2 and CH_4. The acetoclasic methanogens are the most important, since acetic acid is the principal VFA formed by anaerobic digestion (Rozzi *et al.*, 1997) and approximately 70 % of CH_4 production is attributable to acetic acid degradation and the rest via the reduction of CO_2 by hydrogen (Jeris and McCarty, 1965). Methanogens are often considered the key class of microorganisms in anaerobic biotechnology, as they are the most sensitive ones. The theoretical CH_4 production is 0.35 l CH_4 g^{-1} COD removed at standard temperature and pressure (STP) (Olthof and Oleszkiewicz, 1982). The quantity of COD converted to CH_4 depends on factors such as the food to microorganisms (F:M) ratio, temperature, the organisms present and HRT (Speece, 1996) among others. Imbalances of the bacterial populations can lead to build-up of degradation products, which can cause inhibition of the process (Chynoweth *et al.*, 1994). The stability and efficiency of the industrial anaerobic digestion process relies upon the balance between the degradation of organic waste to hydrogen, formate, acetate, and C3 to C5 VFAs and the conversion of these fermentation products to CH_4 and CO_2 (Cord-Ruwisch *et al.*, 1997). Converti *et al.* (1993) proved the existence of an organic loading rate threshold for any effluent, beyond which local surpluses of substrate cause consistent increases in the concentration of VFAs, which may behave as competitive inhibitors of anaerobic digestion. Factors such as residence time, bicarbonate alkalinity (BA), influent substrate concentration, digested sludge return and loading frequency affect reactor stability (Graef and Andrews, 1974). Digester stress conditions (e.g. hydraulic or organic overload and entry of toxic compounds) can cause an imbalance between VFA production and consumption resulting in the accumulation of VFA (e.g. Harper and Pohland, 1986). In poorly buffered digesters, this eventually results in digester acidification ($pH < 6.2$), and in the inhibition of the methanogenic bacteria (Mosey, 1983; Switzenbaum *et al.*, 1990). This can result in total digester failure and death of the methanogenic bacterial population. Several weeks to several months are necessary for the reactor to recover (Steyer *et al.*, 2000). Such failures cause severe environmental and economic problems for the WWT authority. Therefore, there is a need for a sensitive, accurate process control parameter which can act as an early warning indicator of potential digester failure under conditions of small perturbations of organic overload and gradual overloading conditions (Cord-Ruwisch *et al.*, 1997).

Anaerobic treatment has been used since the beginning of the 20th century for the treatment of organic solids from domestic sewage, as it is a treatment process that achieves a high degree of waste stabilisation (Holder *et al.*, 1975). The main barrier to the use of anaerobic treatment of industrial waste was said to be the lack of experience with the process (Switzenbaum, 1995). Advances in reactor design (i.e. high rate reactors with an immobilised biomass - Mousa and Forster, 1999) and increased understanding of the process (Moletta, 1995), among other things, have led to the more widespread use of anaerobic digestion, which is increasingly being used to treat industrial wastes. The start-up of new full-scale installations can be made within a few weeks, sometimes even a few days (Lettinga, 1995). However, these high-rate systems became highly sensitive to toxic materials and have a general tendency for instability (Speece, 1996; Lester and Birkett, 1999) and its control is even more challenging when operating at short HRT where early detection of failure becomes a prime objective. In industrial situations, few indicators of digester performance are usually monitored on-line (Hawkes *et al.*, 1994; Steyer *et al.*, 2002). Most anaerobic digesters avoid instability by retaining the maximal amount of bacterial biomass and operating below their design limits, thus trading relative stability off against reduced cost-effectiveness (Hawkes and Hawkes, 1994). Also, Vanrolleghem (1995) stated that there was also a lack of manipulation variables and actuators. Heinzle *et al.* (1993) reviewed control actions that have been applied to the anaerobic process. Plant operators have used control actions for anaerobic digesters such as reduction of the digester feed, addition of a base, recycling the sludge back to the digester, biogas stripping of CO_2 (Graef and Andrews, 1974) and H_2S (for high sulphate WWs) and re-circulate the scrubbed gas to the digester (Weiland and Rozzi, 1991). The choice of the control action depends on the characteristics of the WW stream to be treated and possibly on the reactor configuration.

2.4.2. Activated Sludge Process

Activated sludge treatment was developed in the UK in 1914 (Crites and Tchobanoglous, 1998) and is the most widely used biological system for WWT (Lester and Birkett, 1999). The system must be continuously aerated to ensure survival of the microorganisms, which is an energy-intensive process. Lester and Birkett (1999) observed that many different species have been reported to be present namely, heterotrophs, autotrophs, yeasts, algae, fungi, filamentous bacteria and protozoa. The existence of the microbial cells in the form of flocs permits them to be consolidated in a separate secondary sedimentation tank. The mixture of biomass and WW is maintained in suspension by the turbulence created by mechanical aerators or diffusers for air or pure oxygen. The process can be considered to be a single-stage continuous culture system employing biomass feedback (Lester and Birkett, 1999).

Aerobic treatments are suitable for treatment of effluents with a COD of 40 - 4000 mg l^{-1} (Thampi, 1998). According to Lin and Peng (1995), activated sludge processes are inadequate for treatment when the COD of textile effluent exceeds 1200 mg l^{-1}. Effluent from well operated activated sludge plants usually have a BOD and total suspended solids (TSS) concentrations of < 10 mg l^{-1}. Nutrients may need to be added to industrial effluents to enable biological treatment (Laing, 1991). Generally they need BOD:N:P of 100:5:1 or 100:3.5:0.6 if dosage control has been used (Möbius, 1991). Other nutrients are required by most biological systems such as: substantial quantities of sodium, potassium, calcium, chloride, sulphate and bicarbonate and trace quantities of iron, copper, manganese, boron, molybdenum, cobalt, and iodine (Metcalf and Eddy, Inc., 1991).

One of the most common problems encountered in the operation of an activated sludge plant is the development of bulking sludge with poor settling characteristics and poor compatibility (Metcalf and Eddy, Inc., 1991). Over 50 % of UK plants have experienced bulking conditions (Lester and Birkett, 1999). The most predominant form of sludge bulking is caused by the growth of filamentous organisms or organisms that can grow in a filamentous form under adverse conditions related namely to (1) the physical and chemical characteristics of the WW (nature, flow, strength, pH, temperature and nutrient content); (2) design limitations (air supply capacity, clarifier design, return sludge-pumping capacity limitations, or poor mixing); (3) plant operation (low dissolved oxygen (DO) in the aeration tank, insufficient nutrients, widely varying organic waste loading, low F:M ratio, and insufficient soluble BOD gradient) (Metcalf and Eddy, Inc., 1991). These authors stated that at high F:M ratios high growth is achieved resulting in growth of filamentous microorganisms - substances such as starch and acetic acid, which have high BOD, may lead to bulking problems. Also, at low F:M ratios bacterial growth cannot be sustained and endogenous metabolism or auto-oxidation occurs where weaker cells die and become a source of food for the remaining cells. The non-biodegradable cell capsules and viable cells then form a pin-point floc which does not settle properly (Lester and Birkett, 1999). Therefore, a balance between the two extremes must be achieved. Chlorine and hydrogen peroxide may be used to provide temporary help to control the filamentous organisms (Metcalf and Eddy, Inc., 1991).

When properly designed and operated, the activated sludge process is capable of high-quality performance and can meet or exceed most effluent requirements. However, it has the reputation of being difficult to operate and can exhibit process failure. As well it requires more energy than the other biological processes used in WWT. To control the activated sludge process, quality variability should be minimised and the effluent quality maintained below effluent standards, followed by minimisaton of costs (Andrews, 1992). The principal factors used in activated sludge process control are 1) maintaining DO levels in the aeration tank(s), (2) regulating the amount of return activated sludge (RAS), and 3) controlling the waste activated sludge (WAS). RAS is important in maintaining the mixed liquor suspended solids (MLSS) concentration and the WAS is important in maintaining a constant F:M ratio and in controlling the mean cell residence time, often referred to as the sludge age (Low and Chase, 1999). Andrews (1992) referred also to the addition of carbon source and chemical dosage for pH adjustment. Control of nutrient addition has also been performed (Lynggaard-Jensen *et al.*, 1996).

2.5. Monitoring the Anaerobic Treatment Process

An anaerobic digester must be considered as a complex system where mechanical, microbiological and physicochemical aspects are closely linked to form a viable unit. Kotzé *et al.* (1969) stated that no single parameter can be used as a control measure of the process of anaerobic digestion as the degradation of organic matter is brought about by a heterogeneous microbial population. Process variables to be used in anaerobic process control can be defined as (a) *stability indicators* - those parameters which should be monitored to assure an early warning of oncoming unstable conditions; and (b) *control variables* - those parameters which should be monitored to keep or restore stable operating conditions. Graef and Andrews (1974) evaluated the following parameters as potential *stability indicators*: rate of CH_4 production, substrate concentration, total VFAs (TVFA), CO_2 partial pressure (*p)*, pH, BA, organism concentration, specific growth rate, dry gas flow rate, total VFA alkalinity (TVA):TA, dissolved CO_2 and unionised acetic acid. They selected the first four as these offered the greatest potential from the standpoint of indicating stability, however, none of these variables were effective in predicting process failure i.e. the rate of CH_4 production decreased along with a simultaneous decrease in pH or increase in $\%CO_2$

and TVFA concentration, for hydraulic, organic and toxic overloading. Two or more of these four variables could provide information needed for monitoring digesters in the field. Archer (1983) stated that the performance of digesters can only be assessed by interpretation of measurable parameters such as pH_2, redox potential, pH, VFAs, temperature, gas production, biomass content and feed rate composition. Heinzle *et al.* (1993) stated that the reactor may be characterised by its VFA levels, CH_4:CO_2 ratio and total gas production rate.

Switzenbaum *et al.* (1990) observed that some of the more commonly used indicators were VFAs:BA ratio; gas production rates and gas composition (CH_4 and CO_2); pH, and VS and COD reductions. The authors stated that usually several of these have been monitored together for supplementary information. Commonly used indicators such as VFA:BA ratio, CH_4 productivity, pH, usually detect process upsets once they are underway (Guiot *et al.*, 1995). Switzenbaum *et al.* (1990) reviewed the off and on-line measurable variables with special emphasis for trace intermediate gas monitoring which has shown some potential to be an ideal indicator i.e. it should be correlated to the metabolic status of the process, function on-line in real-time and be possible to monitor with a reliable, robust and 'easy-to-handle' instrument. The anaerobic digestion process was pictured as a three-phase process (solid-liquid-gas) where each phase is closely related to the other two (Figure 2.1).

Giraldo *et al.* (1990) presented the theoretical relaxation time for different substances such as H_2 in the biogas, CO_2, CH_4, CO, acetate and propionic. The authors found that H_2 had a relaxation time of only 14 seconds, which made it the fastest response parameter together with CO. CO_2, acetate and propionate came after with a relaxation time of 1, 2 and 4 h, respectively and only after CH_4 with 2 days. Moletta *et al.* (1994) found that an organic overload was associated with an accumulation of VFAs, a decrease in pH, an increase in gaseous H_2; an increase in the %CO_2 and corresponding decrease in CH_4, an increased rate of gas production, and a decrease in total organic carbon (TOC) removal. They also found that the detection time of H_2, TOC and VFAs variations was less than 30 minutes, while that of the pH, gas production rate and composition was ~1 hour after the beginning of the increased loading. These authors suggested a combination of pH, biogas production and H_2 as indicators. While Mathiot *et al.* (1992) proposed for an automatic control system the following parameters: liquid phase - VFAs, pH, BA, COD, TOC and dissolved H_2; gaseous phase - production rate and composition of gas (CH_4, CO_2, H_2, CO).

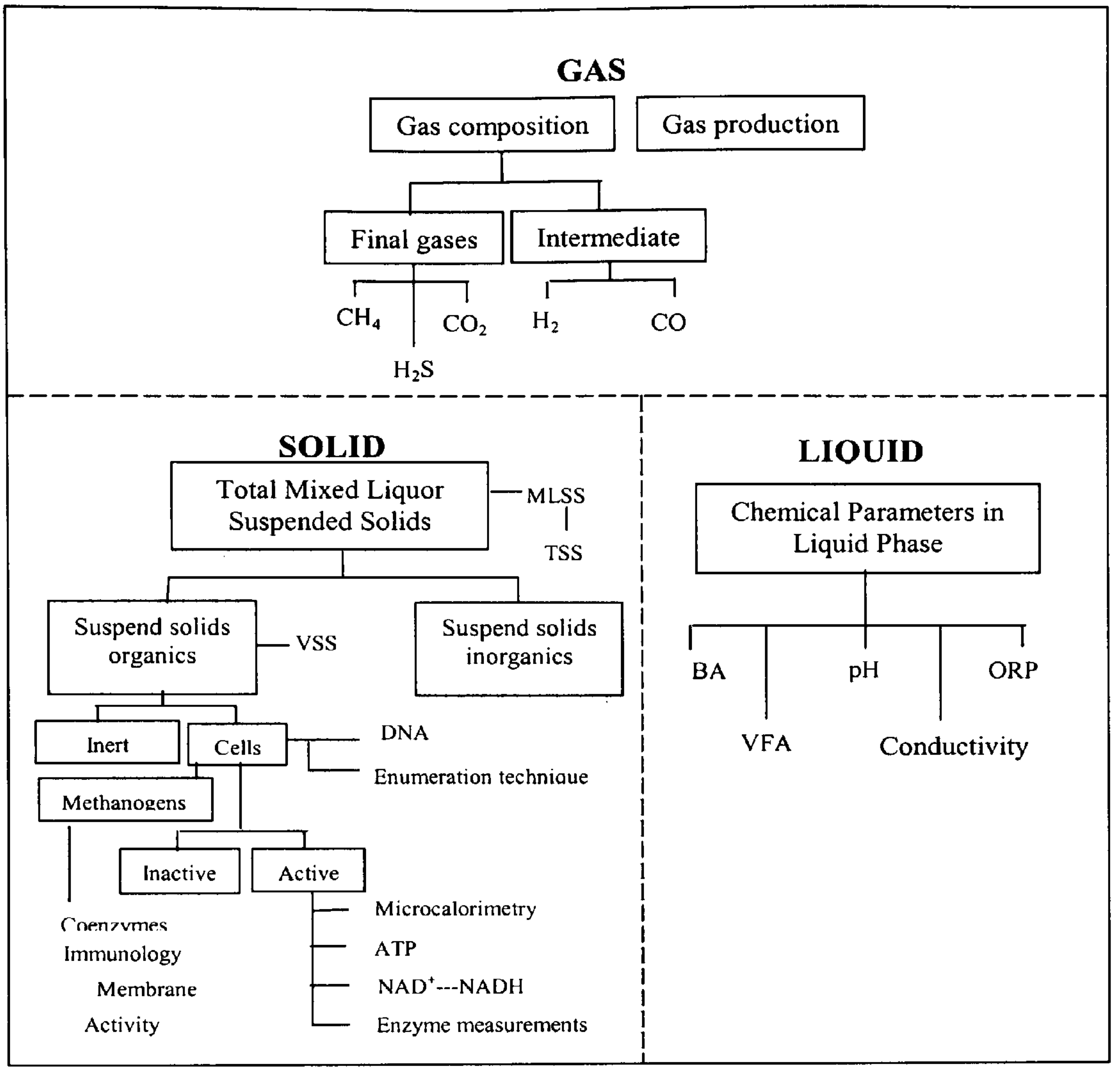


Figure 2.1 - Characterisation tests for the 3-phases of anaerobic digesters (modified from Switzenbaum *et al.* (1990))

Temperature - Environmental factor affecting digester operation

Temperature represents one of the essential factors affecting the WW fermentation rate (Lester and Birkett, 1999). Anaerobic digesters can be operated optimally within mesophilic (25-40 °C) or thermophilic (50-70 °C) temperature ranges (Lester and Birkett, 1999). Methanogens grow optimally at temperatures of 35 °C (Switzenbaum *et al.*, 1990) or 37 °C (Soto *et al.*, 1992). Clearly that a change in operation temperature of a digester will significantly affect the performance, due partly to a change in kinetic parameters but also to changes in the microbial population (Lawrence and McCarty, 1969). After an increase in temperature, Ahring *et al.* (1995) found that CH_4 production almost ceased and did not resume even 10 days later. Peck *et al.* (1986) showed that individual VFA concentrations responded differently during a decrease in the operating temperature of a mesophilic reactor. An order of sensitivity was established: i-butyrate i-valerate i-caproate > propionate > n-valerate n-caproate > acetate n-butyrate. Those to the left accumulated most rapidly in the temperature stressed digester, and were removed least quickly during the recovery period. They recommended that the temperature should be raised back up to the normal operating temperature as soon as possible. Mathiot *et al.* (1992) noted that after the temperature had returned to its normal value, gas production increased immediately, but its CO_2 and CH_4 content took a slightly longer time to go back to their normal values.

2.5.1. Solid Phase Characterisation

Solid phase is the combination of non-soluble materials immersed in the liquid phase. This mixture is composed of organic (inert organic solids and cells) and inorganic solids. Switzenbaum *et al.* (1990) stated that active cell measurements and their metabolic status were very important parameters in defining control strategies as chemical parameters in the liquid phase provide little information about the metabolic status of the microorganisms. However, solid mesurement techniques shown in Figure 2.1 are generally elaborate, time consuming and difficult to use in RTC (Switzenbaum *et al.*, 1990; Fell, 1999), therefore only reference to total solids (TS) and VS measurements is performed here.

Solids

In general, TS or VS measurements are most often used to estimate active biomass, but they do not distinguish between living and dead organisms (Chung and Neethling, 1988). VS have been shown to be significantly related to the deoxyribonuccleic Acid (DNA) contained by living cells (Hattingh and Siebert, 1967). By increasing the VS content of a digester, the amount of substrate may be increased with a resulting overall digestion capacity (Kotzé *et al.*, 1969). VS are not a suitable method for determination of reactor performance during rapid changes in substrate strength or composition due to the slow growth rate of anaerobic bacteria.

Turbidity has been used to measure SS in anaerobic digestion (Vanrolleghem, 1995). Three measuring principles have found application: optical measurements, absorption of ultrasound and gamma rays. Vanrolleghem (1995) stated that attention must be granted to fouling, to the gas bubbles as they may contribute significantly to light scattering, and to colour in the water as it may contribute to the light absorption. For which Vanrolleghem offered some advice on sensor location and usage of pulsing IR-light instead of white light, to help eliminating colour interference.

2.5.2. Liquid Phase Characterisation

Parameters used to characterise the chemical status of the liquid phase are commonly used. Control variables should, preferably, refer to the liquid phase, instead of the gas phase, as the environment to be controlled is the mixed liquor, which contains the anaerobic microorganisms (e.g. Rozzi *et al.*, 1985). On-line monitoring of most of the liquid phase parameters can be implemented, but calibration and maintenance problems make their long-term performance difficult. Chemical or optical probes in the digester effluent can foul, in some cases rapidly, as reported for standard pH probes by Hawkes *et al.* (1992). Conductivity is a measure of the ability of a solution to carry an electric current. Oxidation-reduction potential (ORP), or redox potential, is an indication of the oxidation state of a specific monitored system. ORP should reportedly be below -450 to -500 mV for azo dye reduction to occur (Carliell *et al.*, 1995). ORP measurements are relatively simple and quite accurate (Olsson and Newell, 1999). However, according to FitzGerald (1994), it is insensitive and slow to react, and therefore unsuitable as a measurement for rapid digester control.

Bicarbonate Alkalinity (BA)

It is essential to understand the buffering action since many chemical and biological reactions in WWT are pH dependent and rely on pH control. Alkalinity of a WW is a measure of its capacity to neutralise acids, in other words to absorb hydrogen ions without a significant pH change (Olsson and Newell, 1999).

The incentive to measure the dissolved CO_2 and bicarbonate content of the mixed liquor is because imbalance of anaerobic digesters (due to accumulation of VFAs) cannot easily be detected on the basis of pH measurements alone, especially when the alkalinity of the mixed liquor is high (Rozzi *et al.*, 1994; Hawkes *et al.*, 1992) as alkalinity must be destroyed to a large extent before pH drops significantly. Neither the off-gas CO_2 fraction nor the digester pH changes respond quickly with the onset of digester stress (Ripley *et al.*, 1986). If VFA production/consumption balance becomes too severe the buffering capacity will become inadequate, the pH drops and the reactor 'sours' and methanogenic bacteria become inhibited (Hawkes *et al.*, 1993). Since the alkalinity is mainly due to the bicarbonate buffer (sodium and calcium bicarbonate), it has been proposed since the early sixties that its measurement can be used in control strategies for anaerobic digestion (McCarty *et al.*, 1964). The development of models of the anaerobic system by Graef and Andrews (1974) to investigate the merits of various parameters in process control showed that BA had a significant effect on the stability of the anaerobic bacteria. Rozzi *et al.* (1994) described the start-up and operation of anaerobic digesters with automatic bicarbonate control. The system without control became unstable and sour conditions developed. The advantages of BA monitoring have been reported by Rozzi *et al.* (1985) and a review of the methods/instruments, which have been used to determine BA in complex solutions, was given by Guwy (1995). BA is the buffer capacity provided through neutralisation of CO_2 by the cations released from the breakdown of VFAs forming bicarbonate. The decrease in BA concentration during overloading or inhibition is proportionate to the increase in TVFA concentration provided other weak anions are either negligible or constant, i.e. every mol l^{-1} of VFA that is allowed to build up will destroy (and replace) an equivalent concentration of bicarbonate.

The bicarbonate ion provides buffer capacity over an approximate pH range from 5.3 to 7.3 (Stumm and Morgan, 1981). For historic reasons alkalinity is measured as mg of $CaCO_3$ l^{-1} to a specified pH. Suggested concentrations of bicarbonate range from a recommended minimum of 1000 up to 5000 mg l^{-1} as $CaCO_3$ (Holder *et al.*, 1975; Hobson and Wheatley, 1993) to maintain the reactor pH above 7, while for Jenkins *et al.* (1983) the safe BA level is between 2000-5000 mg $CaCO_3$ l^{-1}. Brovko and Chen (1977) recommended even a BA of 3500-5000 mg $CaCO_3$ l^{-1}. The naturally occurring buffering capacity in anaerobic digesters can vary considerably depending on the waste type. In an anaerobic digester that is working well the VFA:BA is normally 0.3 or less (Ross *et al.*, 1992). If the ratio increases above this, the system is deemed to be unstable (Carliell *et al.*, 1996).

The pH endpoint in the alkalinity titration is the subject of some dispute, values of 5.75 (Jenkins *et al.*, 1983), 4.3 (Ripley *et al.*, 1986), and 4.0 (McCarty *et al.*, 1964) have been suggested in the past. The definition of alkalinity is somewhat circular since selection of a pH endpoint and, consequently, the value obtained for the alkalinity of a sample depends upon prior knowledge of the alkalinity (Powell and Archer, 1989). The buffering capacity of acetic and propionic acids is a useless part of the alkalinity in anaerobic digestion that operates in the pH range 6.5-7.5 (Jenkins *et al.* 1983). Therefore, the distinction between BA and TA is of critical importance. BA refers to the TA minus the TVA. It is common practice in the operation of an anaerobic reactor to use the VFA:TA ratio as a control parameter (Speece, 1996).

The on-line monitoring of BA has had a crucial bearing on the use of BA (and conversely, with the difficulty in monitoring VFAs) as an automatic control variable. Only recently automated bicarbonate monitors have been developed and applied in practice. Guwy *et al.* (1994) described a BA measuring prototype device where a continuous stream of substrate was saturated with gaseous CO_2, acidified by the addition of excess acid, and the rate of CO_2 evolution, proportional to the concentration of bicarbonate/carbonate in the liquid flow, continuously measured by a sensitive gas meter. The instrument was robust and its response was satisfactory for WWT process control applications, with linearity and accuracy of the order of 7 % in the range 5 to 50 mM HCO_3^- and a response time in the order of 30 minutes. Later, Hawkes *et al.* (1995) concluded that the on-line BA monitor was successfully used to monitor and to control an anaerobic digester operating on molasses WW, during organic overloads, using 'on-off' and ANN strategies.

McCarty *et al.* (1964) advised the use of sodium bicarbonate for pH control as it is relatively in-expensive, it does not react with CO_2 to create a vacuum in the digester, and there is little danger that it will raise the pH to undesirable levels. It is also quite soluble and can be dissolved prior to addition to the digester for more effective mixing. Di Pinto *et al.* (1990) used a proportional control law to maintain constant bicarbonate concentration in the effluent by addition of sodium carbonate addition.

Volatile Fatty Acids (VFAs) concentration

All organic acids contain the carboxyl group written as *–COOH,* they are weak, ionise poorly and all have sharp penetrating odours (Olsson and Newell, 1999). Acids with up to nine carbons are liquids but those with longer chains are greasy solids, hence the common name *fatty acid.* VFAs are the most important intermediates in the anaerobic digestion process. VFA concentrations have been monitored for a long time as potential process performance indicators (i.e. acetic, propionic, iso and n-butyric and iso and n-valeric acids). In principle, the concentration of an individual VFA (especially acetic, propionic and butyric acid) can be considered as the best control parameters in the liquid phase, as they give indications about the metabolic state of two among the most delicate microbial groups: the obligate H_2 producing acetogens and the acetoclastic methanogens (Weiland and Rozzi, 1991). For example Dinsdale *et al.* (1997) defined 'steady state' when during at least 3 HRTs TVFA levels remained at a constant low level. In a well equilibrated process, VFAs should either not exist or at least not accumulate. The accumulation of VFAs in digestion mixed liquors never appeared beneficial either to the CH_4 productivities or to the reliability with time of the digestion system. The accumulation of acetate, either transient or stable with time, usually appears harmless. Acetic acid is usually considered to be the predominant VFA present, comprising 50 to 90 % of the total (DiLallo and Albertson, 1961). A high VFA level is the result and not the cause of the digester imbalance (McCarty *et al.*, 1964). Although, VFAs response is not as rapid as that of H_2 (FitzGerald, 1994), monitoring the individual VFA concentration would be a good basis for a process control strategy. A mathematical model developed by Mosey (1983) proposed that the increase in pH_2 as a result of overloading the system would produce a larger increase in propionic acid than acetic acid. The precise cause of high VFAs can be difficult to determine as for example the symptoms of toxicity and of trace metal deficiency are often relatively similar (Speece, 1996).

Anaerobic digestion has been monitored off-line for the determination of the VFA content of the digester supernatant, using several laboratory/instrumental procedures. This is either by titrimetry or colorimetric analysis of TVFA (Montgomery *et al.*, 1962), gas chromatography determination of the individual VFAs (Barnfield *et al.*, 1978), mass spectrometry (MS) or high performance liquid chromatography (HPLC). Using the GC technique, VFAs are normally measured using thermal conductivity detectors (TCDs) or flame ionisation detectors (FIDs). For on-line monitoring the effluent injected must be free from SSs, which is seldom true. Automation of filtration or extraction is difficult, expensive and requires regular maintenance. VFAs can also be measured accurately using MS but the cost is usually prohibitive. Gas chormatography and HPLC methods are complex and require skilled personnel and therefore they are unsuited to automation for on-line monitoring and control of the process (Collins and Paskins, 1987). Various researchers have developed on-line techniques for TVFA measurements e.g Dilallo and Albertson (1961); Powell and Archer (1989); Rozzi *et al.* (1985), but no full success was achieved. Cruwys *et al.* (2002) developed an optimised procedure for the routine analysis of VFAs in WWs using static headspace gas chromatography with a FID. Nordberg *et al.* (2000) used electronic gas sensors and near infrared spectroscopy for determination of volatile compounds in the head space with success.

Asinari di San Marzano *et al.* (1981) suggested that individual VFA concentration increased disproportionately during process instability. Propionate was suggested as the best indicator by Kaspar and Wuhrmann (1978). Propionate and butyrate are important intermediates in the anaerobic degradation of complex matter (Schmidt and Ahring, 1993). Propionic acid is believed to be the most toxic VFA appearing in anaerobic digestion (Hanaki *et al.*, 1994). Methanogenic populations were demonstrated to be inhibited at propionic acid concentrations in excess of 1000 mg l^{-1}, while they could tolerate acetic and butyric acids up to 10 000 mg l^{-1} (cited by Inanc *et al.*, 1999). High concentrations of acetate and H_2 inhibit the conversion of propionic acid to those end products. Such inhibition leads to a build-up of VFAs, which leads to a decrease in pH if the buffering capacity of the system is exceeded, and inhibition increases with decreasing pH. Additionally, the unionised VFAs are able to enter the cytoplasmic membrane of the bacteria and uncouple the process of adenosine triphosphate synthesis (Zoetemeyer *et al.*, 1982). VFA accumulation therefore reflects a kinetic uncoupling between acid production and consumption and is typical for stress situations (Ahring *et al.*, 1995). Any transient state must be stopped and replaced by a steady-state where the F:M ratio should not be exceeded. Propionic acid control may be very useful and can be equivalent for example to control for pH and dissolved H_2, depending on the conditions (Ryhiner *et al.*, 1993). Monitoring propionic acid concentration has been used for controlling overloads of a completely stirred tank reactor (CSTR) anaerobic reactor fed on WWs of a citric acid factory (Renard *et al.*, 1991). TVFA concentration monitoring, although it does not discriminate the behaviour of the different bacterial populations, can also be exploited for process control.

It is not feasible to define an absolute VFA level indicating the state of the process. Anaerobic systems have their own normal levels of VFA, determined by the composition of the substrate digested or by operating conditions (initial pH and alkalinity) (Lester and Birkett, 1999). Buswell (1959) stated that the overall upper limit of 2000 mg VFA l^{-1} was over emphasised. He preferred the use of sudden changes in a constant value of the VFA content, as a control parameter, rather than setting levels for 'safe' digestion. Ahring *et al.* (1995) suggested that the relative change in VFA, not the absolute concentrations as the way forward with a combination of iso and n-butyrate being the best indicator of stress caused by hydraulic and organic loading and temperature increase. Hill (1982) suggested that a propionate:acetate ratio < 1.4 was an indicator of stability. Schroder and de Haast (1988) stated that in a normally operating anaerobic system the concentration of VFA within the reactor, expressed as acetic acid, should not exceed 500 mg l^{-1} and should normally be less than 250 mg l^{-1}. However, Pullammanappallil *et al.* (1998) found that VFA and pH levels cannot be used reliably to predict digester imbalance as the microbial consortia was able to tolerate propionic acid concentration of 3000 mg l^{-1} and pH levels of 6.1.

pH

The parameters pH, alkalinity and VFA content are closely inter-related (Kotzé *et al.*, 1969). Actually, pH control is in effect really BA control. pH control is significant as methanogens growth is severely inhibited below a pH of 6.2 with an optimum growth rate in the range 6.6 to 7.6, and tolerance up to pH 8 (McCarty *et al.*, 1964). pH of 7.0±0.1 has been traditionally recommended. The occurrence of low pH is the result of a well developed imbalance and as such is not useful as an early warning indicator (Switzenbaum *et al.*, 1990).

If acidogenesis proceeds too rapidly compared with acetoclastic methanogenesis, for example following a rapid change in load, H_2 and VFAs accumulate and the fermenter may fail. Inhibition of methanogenesis by acidification can be prevented by efficient pH control in the digester and so under conditions of shock loading coupled to pH control production of organic acids will exceed methanogenesis and the excess acids will appear in the digester effluent until the system becomes balanced again. Without pH control, fermenter failure is the consequence of continued overproduction of VFAs which gives rise to a 'stuck' or 'sour' reactor. pH controls also the fraction of undissociated fatty acids that are thought to freely permeate the cellular membrane of microorganisms after permeating the membrane, the fatty acids internally dissociate, thus lowering the cytoplasmic pH and affecting bacterial metabolism (Zoetemeyer *et al.*, 1982). Therefore, immediate action to add alkalinity is required. Bubbling with N_2 in a pH controlled system enhances methanogenesis due to reduction in inhibition by CO_2 (Hansson, 1979). Removal of CO_2 from carbonate-buffered systems could also be used to rectify mild acidification and might be considered as an alternative to addition of alkali where cation toxicity is a problem (Archer, 1983).

In-line pH monitoring of an anaerobic digester would be more useful in the operation process than off-line due to the loss of CO_2. If the sample is allowed to stand exposed to the air for a few minutes CO_2 will escape, causing the pH to rise (Speece, 1996). pH measurements are simple and inexpensive (pH electrode). Brovko and Chen (1977) stated that the measurement of pH does not itself constitute an adequate control procedure in digester maintenance because it is a logarithmic rather than arithmetic function, pH is not sufficiently sensitive to rather large fluctuations in alkalinity concentrations. For example, while alkalinity decreased from 3600 to 2250 mg l^{-1}, the pH value moved only from 7.1 to 6.9 hardly more than the error involved in its measurement. Also drift of the signal requires frequent re-calibration and fouling makes necessary frequent washing and other cleaning (e.g. ultrasonic) systems or multiple probe systems.

The most commonly used type of pH probe is a combination electrode that consists of a glass and a reference electrode. The nature of the reference junction within the reference electrode limits the use of pH probes in anaerobic digester environments to wastes with low fat and protein concentrations, which could otherwise contaminate the reference junction. The problem is exacerbated by the high solid content and dissolved sulphide compounds present in an anaerobic digester and therefore, conventional combination electrodes operate in a reactor for only a short time before failure. Xerolyte combined pH electrodes (Ingold, BHD Poole, UK) are less susceptible to fouling as they have a stiff polymer mass containing KCl but free from AgCl. They also have an aperture diaphragm, which allows accurate operation for 10 days without need for calibration. Guwy *et al.* (1997b) found that with the upflow velocity in the reactor the Ingold Xerolyte electrodes performed well.

Control of digester stability using pH as a control variable was shown to be effective by for example Rozzi (1984), Denac *et al.* (1990), Di Pinto *et al.* (1990) and Hawkes *et al.* (1995). The pH control of the feed is still used in many digesters, and this is typically a wrong application of control procedures (Weiland and Rozzi, 1991).

Dissolved H_2 concentration

Hydrogen is formed during the breakdown of complex organic matter to VFAs and again during the further conversion of these acids to acetic acid and CO_2 (Mosey and Fernandes, 1989). Because of the unfavourable energetics, oxidation of propionate and butyrate is only possible if products are removed efficiently by the methanogens, resulting in a low pH_2 (Thauer *et al.*, 1977). A pH_2 below ca. 10^{-4} atm is necessary for degradation of propionate and butyrate, respectively (McCarty and Smith, 1986). Such low pH_2 in methanogenic systems are achieved by 'interspecies H_2 transfer' from H_2-producing bacteria to H_2-oxidising methanogens (Wolin, 1975). A rise in pH_2 is inhibitory to production of more H_2 and acetate from VFAs so that organic acids could then only be removed if their concentrations relative to acetate were very high, but as acetate removal is rate limiting this is prevented and fermenters fail due to acidogenesis. Fluctuations in H_2 or levels of VFAs occur normally in fermenters. For instance, a rise in acetate concentration should not be detrimental (Asinari di San Marzano *et al.*, 1981) provided that the operational pH is maintained. Hydrogen is rarely detected in fully functional methanogenic digesters unless the system has been disturbed by an increase in feed load (Archer, 1983), for e.g. a pulse addition of organic toxicants (Hickey *et al.*, 1987) and a pulse addition of heavy metals (Hickey *et al.*, 1989). Various researchers have stated that in general, the behaviour of H_2 appears to depend heavily on the energetic content of the substrate(s), reactor type (and feeding regime) and how well the system was buffered and type of upset (Hickey *et al.*, 1991).

It was initially thought that H_2 transfer occurred between the bacteria and a H_2-pool. However, from the H_2 turn over rates and the H_2-pool (dissolved H_2) size it was calculated that up to 95 % of the H_2 was passed directly to the H_2 utilising bacteria (Conrad *et al.*, 1985). These researchers then proposed that the transfer mechanism was dependant on the juxtaposition of the syntrophic bacterial association (H_2 producing acetogens and methanogens) and that the H_2 produced is utilised before it is able to equilibrate with the H_2-

pool. This transfer would require close contact and it would be an advantage for the anaerobic bacteria to adhere together in flocs, biofilms or granules for this to occur. The high bacterial cell densities in the granules minimise the distances between bacteria and maximise interspecies transfer of acetate, formate, and H_2 between syntrophic fatty acid degraders and methanogens (Conrad *et al.*, 1985; Pauss *et al.*, 1990). These syntrophic reactions generate a very low ORP in the digester, which favours the anaerobes (Fell, 1999).

Hydrogen is being considered as an ideal indicator preceding a noticeable build up of VFAs thus anticipating the failure (Switzenbaum *et al.* 1990). Concentrations and pressures of H_2 are reported in a variety of units. Wolin and Miller (1982) concluded that H_2 was not only a product and a substrate in anaerobic fermentations, but also an important regulator. Hydrogen is a key intermediate in methanogenesis and since the beginning of the 1980s several researchers investigated how to use H_2 concentration (either in the gas phase or dissolved in solution) in anaerobic process control (e.g. Archer *et al.*, 1986; McCarty and Smith, 1986; Collins and Paskins, 1987; Whitmore *et al.*, 1987; Dochain *et al.*, 1991; Guiot *et al.*, 1995). A control strategy using H_2 concentration, VFA and BA have been proposed by Powell and Archer (1989).

According to kinetic considerations based on the Michaelis-Menten model the relationship between the loading rate and the H_2 concentration is expected to be non-linear, based on the assumption that there is a maximum H_2 uptake capacity of the H_2 consuming methanogenic bacteria (Cord-Ruwisch *et al.*, 1997). However, these authors found a quasi-linear relationship between H_2 concentration and loading rate that could possibly be due to the lower energetics of the homoacetogenic H_2 consumption, which need higher H_2 concentrations than methanogenic bacteria for efficient H_2 uptake (Cord-Ruwisch *et al.*, 1988). According to traditional kinetic models, the H_2 concentration without loading should be zero. The reason for the residual pH_2 of 2 to 3 Pa at a feed rate of zero can be explained by the fact that a threshold value for H_2 consumption exists below which methanogenic bacteria are incapable of H_2 degradation (Cord-Ruwisch *et al.*, 1988) and is dependent on the energetic conditions (such as product concentrations, pH, and temperature) (Hoh and Cord-Ruwisch, 1996). Furthermore, as long as the sludge is active, there will be some H_2 production (Cord-Ruwisch *et al.*, 1997).

A number of methods of measuring H_2 in a gas phase or as a gas dissolved in a liquid have been developed, and a review of these methods, in the context of anaerobic digesters, has been published by Pauss *et al.* (1990). Pauss *et al.* (1990) and Pauss and Guiot (1993) emphasised the need to measure H_2 concentration in the liquid rather than the gas phase. This is because H_2 mass-transfer coefficients in anaerobic digesters are much smaller than those typically found in aerobic fermentations (Pauss and Guiot, 1993). However, as long as the H_2 mass-transfer rate satisfactorily correlates with the operating conditions of the reactor, H_2 could be monitored through measurement of its gas content, which is cheaper and more pragmatic than measurement in the bulk liquid (Pauss *et al.*, 1990). Dissolved H_2 in an anaerobic digestion process was continuously measured by a voltametric membrane electrode (Kuroda *et al.*, 1991). The sensor was not affected by several compounds in the anaerobic digestion media except for sulphide. Strong and Cord-Ruwisch (1995) used an inexpensive amperometric dissolved H_2 probe to determine the onset of digester failure by substrate overloading. Later, Cord-Ruwisch *et al.* (1997) used an on-line method for measuring dissolved H_2 in a semi-permeable membrane, situated within the biomass of the laboratory anaerobic digester, using trace reduction gas analysis. The advantages of this new technique over the previous H_2-electrode based control system (Strong and Cord-Ruwisch, 1995) are greater sensitivity, robustness, no interference by other gases, and a stable monitoring baseline.

2.5.3. Gas Phase Characterisation

Anaerobic digester gas from a well functioning unit will always contain CO_2 and CH_4; the ratio is significant as it eliminates the effect of other gases. It will also contain varying concentrations of H_2 and CO, depending on the health of the system. If compounds containing organic or inorganic N and S are present, these will result in production of N_2 and H_2S, respectively. Approximately 70 % of the WW organic component is anaerobically degraded to CH_4 while the remaining 30 % consists of CO_2 with associate trace concentrations of H_2, CO and H_2S gas (Gujer and Zehnder, 1983). The gas concentrations in the biogas constitute a sensitive diagnostic tool, indicating the biochemical condition of the fermentation. The total volume of gas produced is also significant. This will vary depending on the organic loading, biomass capability at a given time and also the operating temperature.

Much of the work performed over the past decade for on-line control, has focused on gas phase characterisation (Hickey *et al.*, 1991). The use of gas phase indicators for real-time data acquisition have been investigated as they have the advantage of significantly faster response times to stress of the anaerobic microorganisms than liquid phase indicators as well as being less susceptible to probe fouling (Switzenbaum *et al.*, 1990; Hickey and Switzenbaum, 1991). Ryhiner *et al.* (1993) and Ehlinger *et al.* (1994) referred that gas phase analysis gave the most reliable and inexpensive results in an industrial environment. However, Ehlinger *et al.* (1994) found that no single gas phase parameter was sufficient for control, only in combination, toxic and organic events could be detected. Mathiot *et al.* (1992) stated that gaseous H_2, CH_4, CO_2 and gas production rate could be used in conjunction as an algorithm for the control of a digester. The following two sections describe the monitoring of gas production rate and gas composition.

Biogas production rate

The methanogenic bacteria have the highest doubling time and therefore are the slowest to adapt to overloadings. This causes a rise in VFAs, accompanied by a drop in total gas production. However, the gas flow rate response to inhibitors is inconsistent because it depends on the type of toxicant (Pullammanappallil, 1993). If the toxicant inhibits the growth rate of the acidogens and methanogens then gas production rate would drop, but if it inhibits only methanogens the gas production rate increases because the CO_2 consumption rate by the methanogens is decreased (Pullammanappallil, 1993). In FitzGerald (1994) work the irregularities in the gas flows correlated with the feed running out; foam blocking lines; and a split in the peristaltic pump tube.

Results of temperature shocks to anaerobic digesters, carried out by Peck *et al.* (1986) showed biogas production rate to be a good indicator of process stability. Kidby and Nedwell (1991) showed that the rate of biogas production responded rapidly to increased hydraulic loading in anaerobic digesters operating on raw settled primary sludge. However, they also stated that gas production rate was not a useful stand alone process control variable, as without the knowledge of organic input to the system or biogas composition for example, the cause of the process instability would be difficult to determine. As the initial increase in gas production during overloading is a function of the CO_2 produced by the destruction of bicarbonate, therefore it is more appropriate to monitor variations in BA or %CO_2 rather than the gas production rate (Rozzi *et al.,* 1985).

A variety of gas measurement systems are currently available for measuring gas production in anaerobic digestion at laboratory scale. Guwy (1995) reviewed some of the gas flow monitoring techniques. For example, Guwy *et al.* (1995) developed a precise low flow gas meter using the more sensitive pressure transducer at that time available and a valve of known characteristics. They developed an on-line high precision gas meter giving reproducible and accurate measurements (0.5 %) at low (<5 cm^3 min^{-1}) and irregular gas flows with a low back pressure, and with an analogue output interfacing to a data acquisition system.

Gas composition

The response time for gas composition monitoring depends on the head-space volume, rate of biogas production and gas solubility (Hawkes *et al.* 1995). The composition of the biogas is possibly a more useful indication of the anaerobic digester status than the biogas production rate as it reveals information about the methanogenic activity. The major and trace gas components of biogas are reviewed in the following.

Biogas CH_4 and CO_2 concentrations

Theory indicates that the rate of CH_4 production is directly related to the condition of the digestion process. In cases of hydraulic, organic and toxic loading the rate drops sharply as failure ensues. However, Graef and Andrews (1974) reported from their simulations that the rate of CH_4 production exhibited a temporary increase for both hydraulic and organic overloading, yet in the case of toxic overloading it demonstrated a definite decline, which suggested that this rate may be a diagnostic of toxic failure. Ryhiner *et al.* (1993) stated also that when the pH drops, this would initially result in an apparent decrease in CH_4 production caused by an increase in gas-liquid mass transfer, caused by the release of CO_2. Methane concentration depends on the liquid flow rate which makes its control in the gas phase unsuitable for anaerobic digestion and it could only be used as a controlled variable if the set point was to be varied depending on CH_4 content corrected for the liquid feed rates (Ryhiner *et al.*, 1993). The CH_4 produced does not undergo any chemical reaction and being slightly soluble is transported quantitatively into the gas phase (Andrews and Graef, 1971). Also, von

Sachs *et al.* (2000) showed that CH_4 production rate response was fast because of the low solubility of CH_4 in water.

Graef and Andrews (1974) findings indicated that the CO_2 composition can fluctuate substantially even when the increase do not approach digester failure. This fluctuation results from the dual sources of CO_2 and the rate of gas transfer between the liquid and gas phase. They explained that the balance either remains in solution as dissolved CO_2 and carbonic acid or reacts with a base to form bicarbonate ions. The solubility of CO_2 is a function of its partial pressure, however, the quantity of CO_2 converted to bicarbonate and carbonate ions depends on its partial pressure as well as the pH and base concentration of the solution. If the partial pressure and/or pH decreases, CO_2 will enter the gas phase because of chemical and physical release from the solution (Graef and Andrews, 1974). The suitability of $\%CO_2$ as a control variable is questionable, as significant variations in pCO_2 are dependant on the CO_2 'stored' in the liquid phase as bicarbonates. For this reason, Rozzi *et al.* (1985) reported that although pCO_2 can be a good instability parameter, a control system using the addition of alkali, based on the pCO_2 would be detrimental if it were to postpone and amplify the ‘surge’ of CO_2 due to bicarbonate decomposition. Variations in biogas CO_2 concentration due to organic overloads have been determined by Denac *et al.* (1988), Hawkes *et al.* (1992) and Guwy *et al.* (1995). Unfortunately, CO_2 and CH_4 variations are significant only after the imbalance is well developed (Switzenbaum *et al.*, 1990). More recently, Merkel and Krauth (1999) studied the mass transfer of CO_2 in anaerobic reactors under dynamic substrate loading conditions and found that it was governed by the liquid phase resistance with the volumetric mass transfer coefficient being a function of the actual biogas production rate. The authors stated that equilibrium of CO_2 concentration between the two phases could not be assumed, so that in carbon balances even under steady-state conditions of oversaturation must be considered, in which the pH in the reactor is shifted towards lower values. For control of anaerobic digestion, the $CH_4:CO_2$ ratio presents a rapid and sensitive parameter, however, no dogmatic rule can be laid down concerning a definite ratio or the volume of gas, which should be produced (Kotzé *et al.*, 1969). Any decline in rate of gas production from a constant value accompanied by a change in the $CH_4:CO_2$ ratio is indicative of unbalanced conditions (Kotzé *et al.*, 1969) and must be immediately investigated. A change in this ratio is partially caused by the referred increase in CO_2 concentration.

Specific gas analysers monitor the content of a component directly. Typically, infrared absorption on-line measurements are used to determine CO_2 and CH_4 (Mathiot *et al.*, 1992), which are less expensive than gas chromatography techniques described by Denac *et al.* (1988) and TOC methods of CO_2 determinations (Rozzi and Brunetti, 1980). Calorimetry can quantify CH_4, which is a relatively inexpensive detection method that can provide in-line analysis (Fell, 1999).

Biogas trace gases – H_2, CO and H_2S

Hickey *et al.* (1987) examined the response of the anaerobic digestion process to inhibition induced by the pulse addition of four organic toxicants. CO_2, CH_4 and H_2 in the gas were monitored with a gas chromatograph (GC). Severe inhibition of CH_4 production (> 70 % CH_4 reduction) resulted in a rapid accumulation of H_2 in the gaseous headspace. Their results indicated that monitoring H_2 in conjunction with conventional process indicators should improve digester monitoring and could provide more rapid indication of process upsets due to toxic shocks.

There has been an increasing level of interest in measuring H_2 concentration in the gas space of anaerobic digesters as a possible method of controlling the substrate loading rate (Archer *et al.*, 1986; Harper and Pohland, 1986; Kidby and Nedwell, 1991; Mosey, 1983; Whitmore *et al.*, 1987). This is because H_2 is believed to be responsible for 1/3 of the electron transfer between fermenting and methanogenic bacteria (Archer *et al.*, 1986). It has been shown that gaseous H_2 is extremely sensitive, and responds strongly to any disturbance to the system, and also to increases in organic loading rate (FitzGerald, 1994). An increase in gaseous H_2 has been observed only 10 minutes after shockloading (Mathiot *et al.*, 1992). After an organic overload, H_2 concentration in the gas increased by 350 % in 30 minutes which makes it a very important parameter for the control of anaerobic digestion of highly degradable WW (Moletta *et al.*, 1994). Puñal *et al.* (1999) found H_2 concentration a quite sensitive variable, and also a very fast response variable, but it must be analysed together with parameters such as CH_4 composition or gas flow rate. Fell (1999) has chosen biogas H_2 as a control parameter for his research because it could rapidly detect fluctuations in toxicant concentration of instant coffee processing effluent (pyrazine), it was non-invasive and had the advantage of not being susceptible to probe fouling. Furthermore, equipment to measure H_2 in biogas was commercially available at low-cost. However, he found that H_2 concentration decreased to normal operational levels before the digester had achieved steady-state operation according to parameters such as the VFA:BA balance. He concluded that H_2 concentration was advantageous as a rapid detection control parameter and could be useful in combination with other parameters, such as COD and VFA which did not respond as quickly to inhibition, but were better indicators of longer term digester instability.

The H_2 concentration in the biogas is usually determined by a GC using specific detectors e.g. the mercuric-mercuric oxide detector. However, on-line GC analysis is sophisticated and expensive which restricts the use of such technique. Accurate, inexpensive and rapid on-line measurement of low H_2 concentration (detection limit 0.1 ppm) has been made possible by the development of an instrument, the Gas Measurement Instruments (GMI) Exhaled Hydrogen Monitor (GMI Ltd. Renfrew, Scotland), for investigation of inborn mal-adsorption of sugars in humans. The instrument has been widely used since its introduction in 1980 to investigate the H_2 concentration in the biogas of anaerobic digesters as originally suggested by Mosey (1982). The instrument usage in anaerobic digestion was reviewed by Collins and Paskins (1987). It is a polarographic instrument and is cross sensitive to H_2S and mercaptans that can occur in biogas from anaerobic digester. Its cross sensitivity to H_2S makes it essential to strip the H_2S from the biogas before introducing it into the monitor. Different chemicals have been used for scrubbing H_2S from the biogas before entering the GMI Exhaled Hydrogen Monitor: copper sulphate (Mosey and Fernanades, 1989; Guwy *et al.*, 1997a); lead (Mosey and Fernandes, 1984) and zinc (Fell, 1999) acetate crystals. Measurement of H_2 in the biogas has the attraction of simplicity compared to the dissolved H_2 but with the disadvantage of the transfer rate of H_2 from liquid to the gas phase (Archer *et al.*, 1986).

Mosey and Fernandes (1989) concluded that for digesters treating milk sugars H_2 served as an excellent 'event marker'. With industrial waste digesters it might also prove to be of use to detect fluctuations in WW strength at constant flow rate, as an alarm indicator for toxicity, or as feedback control signal to adjust the speed of the pump to suit the strength of the WW. Although it responded quickly (<10 minutes) to changes in the substrate flow rate and composition, its increase did not necessarily mean that inhibition of the process has reached a critical point (Mosey and Fernandes, 1989). These researchers showed that large changes in H_2 concentration could occur without any significant changes in bacterial performance.

Various researchers have proposed various H_2 management schemes (such as stripping) to improve reactor performance (e.g. Poels *et al.*, 1985). Some studies with artificial elevation of H_2 concentration in anaerobic reactors did not show any noticeable effect on propionic acid accumulation (fixed bed reactor - Denac *et al.*, 1988; CSTR - Inanc *et al.*, 1999).

During an overload of an anaerobic filter, pH_2 in the biogas and gas flow rate were the first parameters to change and the coefficients varied around 50 and 4 – 5, respectively (Moletta, 1989). During the same experiment, pH variations were small and TOC was multiplied by 3, however both occurred later. The variations in gas composition, in VFAs and BA were the last factors detected. Guwy *et al.* (1997a) concluded that the use of biogas H_2 as a control parameter would be questionable especially if the pre-acidification of the feed to the digester was variable. However, they did also state that the rapid response and ease of on-line measurement of H_2 support its use in digester control along with other parameters, which can be measured on-line. This agreed with Switzenbaum *et al.* (1990) who stated that H_2 is limited as a stand-alone indicator as it is not always apparent why its concentration has changed. Kidby and Nedwell (1991) used the GMI instrument on a CSTR sewage sludge digester to determine whether H_2 concentration could provide advanced warning of digester failure for a HRT reduction from 20 to 8 d. At the onset of digester failure a marked decrease in the volume and CH_4 content of the biogas was accompanied by an increase in VFA production and a concomitant decrease in pH. During this period, H_2 accumulation in the biogas was not evident. An increase in H_2 was noted, however, well after reactor failure had occurred. Therefore, they considered that H_2 concentration in the biogas could not be used as an indicator of incipient digester failure, at least when volumetrically overloaded. However, in a pilot-scale anaerobic contact system, H_2 concentration measured with the GMI instrument rose rapidly in response to volumetric shocks even when no VFA accumulation was observed (Archer *et al.*, 1986).

Hickey *et al.* (1989) found that CO demonstrated a characteristic response to heavy metal and organic toxicant induced inhibition. Hickey and Switzenbaum (1990) found that CO concentrations in the biogas were regulated by ORP and that an increase in CO happened in response to increased acetate or organic loading. They used GC for CO and H_2 determination. CO was found to vary between 300 to 2000 ppb and H_2 ranged from 14-100 ppm. They noted that the levels of gaseous CO in anaerobic digester samples were directly related to the acetate concentrations and inversely related to CH_4 concentration. They concluded that analysis of H_2 and CO together should give information on the metabolic status of both of the terminal methanogenic steps in the degradation process. Puñal *et al.* (1999) concluded that the monitoring of CO concentration did not permit the prediction of destabilisation of the bioreactor.

Little has been published on H_2S monitoring. Sulphide volatilisation is a function of many digester operational parameters including pH, sulphate loading rate, metal concentration and biogas production rate (McFarland and Jewell, 1989). The incorporation of H_2S gas as a parameter into a control strategy may be of use for wastes, which contain high concentrations of influent sulphur. Sarner *et al.* (1988) used a gas washing system, containing sodium sulphide and sodium carbonate, to scrub H_2S from biogas produced from a full scale anaerobic digester, which was able to maintain H_2S levels below 100 ppm. H_2S measurement in the gas phase may be performed by monitoring the reaction of sulphide with Pb-strip, subsequently, the black PbS that is produced, is quantified by colorimetry (Escoffier *et al.*, 1992).

2.6. Monitoring the Aerobic Treatment Process

The activated sludge process is required to meet effluent standards while keeping investment, sludge production and energy consumption as low as possible (Spanjers *et al.*, 1998). A problem inherent in achieving the aims apart from the monitoring difficulties is that the activated sludge process is highly dynamic because of variations in influent flow rate, concentration and composition and sometimes the presence of toxic substances. Therefore, control strategies must be applied that can cope with the dynamic character of the activated sludge process and with the following objectives: growing the right biomass population, maintaining good mixing, adequate loading and DO concentration, adequate airflow, good settling properties, avoid clarifier overload and avoid denitrification in clarifier (International Association on Water Quality - IAWQ, 1995). Consequently, common control strategies manipulate the WAS, RAS and aeration capacity. Such strategies have been based on measurements of MLSS, DO and more recently respirometry.

2.6.1. pH

Nitrification releases protons, which must be buffered by sewage as excess acid destroys activated sludge (Teichgraber, 1990). Remedies are enhancing denitrification, reducing influent nitrogen, limiting nitrification and/or dosage of alkali for which exact dosage control and careful choice of alkali are required (Teichgraber, 1990). Conversely, Beaubien and Jolicoeur (1985), controlled pH at around 8 using sulphuric acid, as the influent was very alkaline.

2.6.2. Dissolved Oxygen (DO)

DO is one of the most important and useful measurement in activated sludge processes and is also the basis for the BOD and oxygen uptake rate (OUR) tests. Olsson and Andrews (1978) demonstrated that the DO profile reflected both, the hydraulic and organic loadings, reaction rate, and degree of completion of the reactions in the activated sludge process. Metcalf and Eddy, Inc. (1991) discussed the role of oxygen in the activated sludge process. They stated that the oxygen supply must be adequate to 1) satisfy the BOD of the waste, 2) satisfy the endogenous respiration by the sludge organisms, 3) provide adequate mixing, and 4) maintain a minimum DO concentration throughout the aeration tank. To meet the sustained peak organic loadings, it is recommended that the aeration equipment be designed with a safety factor of at least two times the average BOD load. In practice, the DO concentration in the aeration tank should be maintained at about 1.5 to 4 mg l^{-1} in all areas of the aeration tank; 2 mg l^{-1} is a commonly used value. Values above 4 mg l^{-1} do not improve operations significantly (Low and Chase, 1999), but increase the aeration costs considerably (Lee *et al.*, 1998). Aeration accounts typically for more than 50 % of the total plant energy requirements (Groves *et al.,* 1992).

The solubility of oxygen in water is directly proportional to the pressure it exerts on the water. At a given pressure solubility of oxygen varies greatly with water temperature and to a lesser degree with salinity. A typical DO saturation value is 9.2 mg l^{-1} at 20 °C (assuming zero chloride concentration) (Olsson and Newell, 1999). DO is widely measured on-line using a DO probe (membrane electrode) composed of two solid metal electrodes in contact with a salt solution that is separated from the water sample by a selective membrane. The probe has also a sensor for measuring temperature. With membrane-covered electrode systems the problems of impurities are minimised because the sensing element is protected by an oxygen-permeable plastic membrane that serves as a diffusion barrier against impurities (Olsson and Newell, 1999). DO automatic control strategies are being employed in many full-scale activated sludge WWTPs, with success. DO dynamics are fast, reliable sensors exist for measuring DO, and the manipulated variable (air flow rate) can be easily adjusted (Barnett, 1992).

2.6.3. MLSS, Volatile Suspended Solids (VSS), Turbidity and Settling Properties

The concentration of biomass in the aeration tank has been expressed as the MLSS. Since MLSS incorporates inorganic solids, a closer approximation to the biotic component may be obtained by using VSS. Between 40 and 85 % of the MLSS is typically volatile. Neither the MLSS nor the VSS are necessarily directly related to the number or mass of viable microbial cells present i.e. they include both live and dead cells, suspended volatile matter, etc. (Lester and Birkett, 1999). Beaubien and Jolicoeur (1985) maintained MLSS around 3.5 g l^{-1}.

Turbidity, the scattering and absorption of light by particulate matter, is one of the most commonly measured physical parameters in WWTPs (Johnson, 1998). Turbidimeters fall into two basic categories i.e. absorptiometers, which measure the absorption of light through a sample, and nephelometers, which measure the amount of light scattered at one or more angles to the incident beam (Briggs and Grattan, 1992). Some of the turbidimeters have built-in compensation for colour of the sample, fouling of optical surface, and/or mechanical means of cleaning the surfaces, and in some instances the optical elements are not in contact with the sample at all (Briggs and Grattan, 1992).

Increasing attention is being drawn to final clarification as the unit operation in activated sludge WWT that is critical to overall treatment performance. Vanrolleghem *et al.* (1996) reviewed a number of automated systems for quantification of settling properties. Different measuring principles are used in these so-called settlometers (Severin *et al.*, 1985). The traditional way of quantifying sludge settleability was by measuring the sludge volume index (SVI). Various approaches to designing on-line settlometers have been reported. One approach is to install a measuring system that tracks the sludge blanket or concentration profiles in the full scale clarifier. Another methodology consisted of using a down-scaled version of the final settler in a measuring system and performing experiments in this model reactor using a moving optical system (Vanrollenghem *et al.*, 1996). Parallel work was carried out using image analysis to reveal the relation between sludge floc structure and settling properties (Grijspeerdt and Verstraete, 1996). Using image processing a bulking index has been proposed to predict the occurrence of bulking phenomena in the activated sludge (Olsson and Newell, 1999).

2.6.4. Respirometry

In aerobic suspended growth effluent treatment systems, the level of MLSS is maintained by recycling at a level sufficient to treat the incoming organic load, but there is a lack of reliable on-line information on which to control the RAS. Respirometry is the measurement and interpretation of the respiration rate of activated sludge and it has been generating much interest for monitoring and control of activated sludge plants (Baeza *et al.*, 2002). The respiration rate or the OUR is the amount of O_2 consumed by the microorganisms measured per unit volume and unit time, and can be taken as a measure of the biological activity. High OURs indicate high biological activity, and vice-versa. OUR is most valuable for plant operations when combined with VSS data as the specific oxygen uptake rate (SOUR). OUR reflects two of the most important biochemical processes in a WWTP: biomass growth and substrate consumption (Olsson and Newell, 1999). The types of respirometer in common use to measure OUR depend on oxygen electrodes, infrared detection of evolved oxygen, gas flow or pressure measurements. The measuring principles and the significance of the results are reviewed by Mahendraker and Viraraghavan (1995) and Spanjers *et al.* (1996). OUR is known to reflect the extent of microbial activity in breaking down organic matter and SOUR for a given sludge type has been shown to be a good indicator of active cell biomass and to vary with substrate concentration according to the Michaelis-Menten equation, thereby allowing prediction of final effluent quality to be made during transient loading conditions (Huang and Cheng, 1984). On-line applications of respirometry have been reported. The respirometric methods are relatively simple, fast, and economical (Mahendraker and Viraraghavan, 1995). To collect respiration rate data several respirometric measuring principles developed in the past are at the disposal of the modeller. These principles can be classified into a limited number of basic principles according to only two criteria: (1) the phase where O_2 is measured, gas or liquid; (2) the flow regime of either gas or liquid phase, flowing or static (Spanjers *et al.*, 1996). In all cases, the respiration rate is obtained from O_2 mass balances over these phases. The work described by Vanrolleghem and Spanjers (1998) concerned a new hybrid respirometric principle obtained by combining the two different respirometric principles mentioned above, which was found to be particularly suited to sludge and WW characterisation in the context of activated sludge process models. On-line respirometry can be used to estimate the organic load to the plant (Brouwer *et al.,* 1994), warn of incoming toxicity (Vanrolleghem *et al.*, 1994), predict biodegradability of WWs, and predict effluent quality. Sometimes the term BOD monitor is used for respirometers, however, this is not to be confused with 5 days standard BOD since the monitor measures oxygen consumption using a biomass adapted to the WW typically during a few minutes (Olsson and Newell, 1999). The rapid oxygen demand and toxicity tester (RODTOX) can determine potential toxicity towards the aerobic process stage (Rozzi *et al.* 1999) and also the strength of the influent as short-term BOD (BOD_{st}) (Grijspeerdt *et al.*, 1995). More details about the RODTOX can be found elsewhere (Vanrolleghem *et al.*, 1994). Respirometric techniques can be used for simulation of process models and be used for on-line control of activated sludge plants (Mahendraker and Viraraghavan, 1995). Low and Chase (1999) found OUR a useful control variable, however, there has been some controversy as to the utility of this variable. This is due partly to inadequate measuring techniques, difficulties in locating the instrument in the process and partly to a lack of understanding of the information content of respirometric data (IAWQ, 1995). Currently about 50 % of the commercial respirometer brands are based on a DO sensor (Spanjers *et al.*, 1998). Spanjers *et al.* (1998) published a book on the principles of respirometry in control of the activated sludge process. The work described in detail subjects such as the measuring principles, modelling respiration, the controller structures and manipulated variables. Typical examples of control objectives are: keep the MLSS concentration at 3 g l^{-1}, let the DO follow a changing desired value and keep the actual respiration rate at 42 mg l^{-1} h^{-1}. Recently, Baeza *et al.* (2002) stated that their control system using in-line fast OUR measurements and ANN models was able to increase 10 % of the N removal in a nutrient removal plant.

2.6.5. Biomass Activity

In activated sludge plants, the ratio of active to inactive solids can change through changes in raw waste characteristics or sludge retention time (SRT) as such biomass can be separated into vital biomass and necromass. Thus both MLSS and VSS may misrepresent the active biomass concentration (Austin *et al.*, 1994). Viable cells can be stained with vital dyes and thus be differentiated from dead cells, allowing an on-line estimation of viable biomass and the cell morphology can also be analysed (Bittner *et al.*, 1998). Sonnleitner *et al.* (1992) presented a review on biomass determination and stated that on-line estimation of the biomass concentration is no longer a matter of comfort but it is essential for control. On-line measurements of the activity of activated sludge biomass may allow efficient operation of WWTPs. For off-line determination techniques such as sonication followed by microscopic analysis, cytometry and also by indirect methods such as degydrogenase activity have been used. However, some methods do not provide the viability of the cells and/or they are very laborious, time-consuming, difficult to calibrate and questionable to validate. A series of sensors and methods that can be automated have appeared in the last decades. Many of them rely on optical measuring principles, others exploit filtration characteristics, density changes of the suspension as a consequence of cells, or (di)electrical properties of suspended cells and even exploiting the heat generated during growth and other metabolic activities of organisms, which is also proportional to the amount of active cells in a reaction system (cited by Sonnleitner *et al.*, 1992).

A relationship between activities of enzymes, particularly those important to oxidative substrate removal, in activated and microbial numbers has been suggested. Biochemical activities of oxidation pond contents sampled throughout a year showed that variation in the activity of catalase and phosphatase seemed to be directly related to bacterial number (Hosetti and Patil, 1988). Also Richards *et al.* (1984) demonstrated good correlations between various enzymatic activities and respiration rate in studies on 14 activated sludge plants. Catalases are a type of hydroperoxidase which scavenges H_2O_2 and protect cells from damage caused by reactive oxygen species. Catalase is an iron porphyrin protein, which is reduced by H_2O_2 and reoxidised by O_2. This enzyme is produced in living cells and decomposes the toxic excretory product H_2O_2 into water and oxygen (Hosetti and Frost, 1998). Catalase activity is widespread in plants (Kimborough *et al.*, 1997) and animal cells, with its detection being a parameter in bacterial classification schemes. Catalse activity is routinely monitored as disappearance of H_2O_2 followed spectrophotometrically (Aebi, 1974) or by titration (Scott and Hammer, 1961). The measurement of catalase activity indicates the biological activity and organic strength of the wastes. A study by Hosetti and Frost (1998) revealed that the catalase measurement can be used as an alternative to BOD in monitoring activated sludge plant effluents. Catalase tests require only a short period of time for completion and are effective over a wide range of temperature and pH. A novel monitor by Guwy *et al.* (1998) for measuring biomass catalase activity, which can be used on-line follows oxygen evolution from H_2O_2 by catalase activity in activated sludge. During studies performed by the authors, the biomass activity determined by the monitor correlated well with the respiration rates and should provide a simple and robust alternative to OUR measurements in activated sludge plants. The monitor provided a robust, low maintenance and non-invasive on-line measurement of a parameter related to active aerobic cell mass, unaffected by fouling of a sensor, colour of the medium or the presence of inert particulate matter.

2.7. Performance Related Parameters for Biotreatment Processes

This section reviews parameters normally associated with the performance of the biotreatment processes i.e. organic strength, colour and aromatic amines. These paramaters are common to both biotreatment processes: anaerobic and aerobic.

2.7.1. Organic strength

The main aim of biological treatment processes is to reduce the organic content of the WW in order to be discharged to the environment and not violate discharge requirements set by the regulatory authorities. The oxygen demand is an extremely important measurement of WW quality as it measures the potential for oxygen depletion in the water, and therefore is an important indicator of organic pollution. Deoxygenation of water can seriously adversely affect the ecosystem, and is often used to define the level of pollution in the water (Stephenson and Judd, 1995). It is necessary to assess the overall efficiency of biotreatment processes as this has a direct bearing on the quality of the final effluent and the economy of the process. A decrease in efficiency would be indicative of failure of the process and can therefore serve as a control parameter. However, for example a low COD removal efficiency may be recorded for an effluent which is not giving rise to inhibition, but which simply has a large component of non-biodegradable though chemically oxidisable material. Another parameter of the efficiency is the percentage conversion of the organic content to biogas in anaerobic digestion. A description of the main measurements of organic content in WWs will follow. For each parameter off-line assays and on-line instrumentation is briefly described.

Biological Oxygen Demand (BOD)

BOD is the classical parameter to define the 'strength' of a WW. BOD is a measure of the biodegradable organics levels using suspended microorganisms (Stephenson and Judd, 1995). Measured amounts of a WW diluted with prepared water are placed in a bottle. The dilution water contains a nutrient solution and is saturated with DO. The DO is measured at the start usually with a DO probe. Seed microorganisms are supplied to oxidise the waste organics if sufficient microorganisms are not already present in the WW sample. The primary reaction is metabolism of the organic matter and uptake of DO by bacteria releasing CO_2 and producing an increase in bacterial population. The secondary reaction represents the oxygen used by the protozoa, which consume bacteria in a predator-prey relationship. The standard test has an incubation period of five days (BOD_5) or in some countries seven days (BOD_7) at 20 °C (Olsson and Newell, 1999) and then the DO is measured again and recorded in mg l^{-1}. Depletion of oxygen in the test bottle is directly related to the amount of degradable organic matter. Considering the time for the analysis the BOD test is certainly not suitable for operation/control purposes. Furthermore, BOD is not a single point value but is time dependent, also is not a precise measurement and the reproducibility is quite poor. Tests on real WWs normally show standard deviations of 10-20 % (Olsson and Newell, 1999). However, the regulatory authorities still use the BOD test to assess the organic content of WWs (Stephenson and Judd, 1995). Vanrolleghem *et al.* (1990) developed the RODTOX, an aerobic biosensor for rapid determination of the BOD_{st} (30 minutes) and correlated closely with the standard BOD_5 value at 20 °C.

Chemical Oxygen Demand (COD)

COD is widely used to characterise the organic strength of WWs. The test measures the amount of oxygen required for chemical oxidation of organic matter in the sample to CO_2 and H_2O. The laboratory test procedure is to add a known quantity of standard potassium dichromate solution, sulphuric acid reagent containing silver sulphate, and a measured volume of sample to a flask. This mixture is vaporised at 150 °C and condensed for 2 hours. Most types of organic matter are destroyed in this boiling mixture of chromic and sulphuric acid. After the mixture has been cooled and diluted with distilled water and the condenser has been washed down the dichromate remaining in the specimen is titrated with standard ferrous ammonium sulphate (FAS) using ferroin indicator. Ferrous iron reacts with dichromate ion with an end point colour change from blue-green to reddish brown. A blank sample of distilled water is carried through the same COD testing procedure as the WW sample in order to compensate for any error that may result due to the presence of extraneous organic matter in the reagents. There is no uniform relationship between the COD and BOD of WWs except that the COD value must be greater than the BOD (Olsson and Newell, 1999). An empirical correlation of COD to BOD for a particular WW can be determined (American Public Health Association - APHA, 1989) which is useful as this method takes only ~3 hours.

The COD test, however, cannot distinguish between biodegradable and inert organic matter (Germirli *et al.,* 1991). The COD:BOD ratio provides an estimate of the proportion of biodegradable organic matter present in WWs (Gray, 1989). Baker *et al.* (1999) referred to the several interferences and potential sources for error in the COD test e.g. the volatile compounds are only oxidised to the extent with which they stay in contact with the liquid media (APHA, 1995) and the heat generated from adding sulphuric acid to the flask may drive volatile compounds out of the solution (Wolff, 1975). 'Even a trace of organic matter on the glassware or from the atmosphere may cause gross errors'. Standard Methods recommend that samples should have a COD greater than 50 mg l^{-1} and lower than 250 mg l^{-1}. There are various commercial on-line COD monitors. For example, the RTM Arkon GIMAT COD monitor (RTM Ltd., Cheltenham, UK) that works based on an oxidation by H_2O_2 with a ± 5 % accuracy and a range of 0 – 5000 mg l^{-1}. The short-time Phoenix on-line COD instrument (Envitech Ltd., Cardiff, UK) is similar in principle to the operation of the BIOX 1000. The On-line COD Analyser CT100 (Data Link Instruments) uses ultra violet (UV) spectroscopy and shows good correlations with manually performed COD, it does not need reagent, it works on unfiltered WW, it needs low maintenance, and the results are provided in 30 seconds.

Total Organic Carbon (TOC)

TOC measures the organically bound carbon in a WW sample. Unlike BOD or COD, TOC is independent of the oxidation state of the organic matter and therefore, it does not provide the same kind of information. TOC does not measure other organically bound elements such as nitrogen, hydrogen and inorganics that can contribute to the oxygen demand measured by BOD and COD (Queeney and Hoek, 1989). If an empirical relationship is established between TOC and BOD or COD then it can be used to estimate the accompanying BOD or COD. TOC is an instrumental analytical method, and its central principle is to convert organic carbon to CO_2 and measure this product in the evolving gas phase. Commercially available TOC analysers oxidise organic carbon to CO_2 and work based on the following principles: high temperature catalytic conversion (650-800 °C), UV radiation, chemical oxidants (e.g. persulphate), or combinations of these (Olsson and Newell, 1999). The CO_2 can be measured directly by a non-dispersive infrared (NDIR) analyser or it can be reduced to CH_4 and measured by a FID in a GC. Inorganic carbon (IC) must be eliminated or compensated for since it is usually a very large portion of the total carbon (TC) in a WW sample. The determination of TC and IC with the estimation of TOC by difference is a common procedure. Particulate matter is to be avoided because the retention time in the reaction chamber is insufficient to allow complete combustion. Moreover clogging may be a problem. Pre-filtration of the samples is therefore essential for proper operation. The trend is to replace COD and BOD with TOC measurements, considering that is a rapid technique, requiring less than 8 minutes and produce highly reproducible results (Laing, 1991). Wilson (1997) found a good correlation between TOC and COD for fresh influent (food WW) but lower correlations with 24 hour old influent and a poorer correlation still with effluent COD. However, the relationship between TOC and BOD showed a high correlation for all three types of samples: fresh influent, 24 hour old influent and effluent. Stephenson and Judd (1995) stated that COD:TOC ratios varied from 6.66 to 1.75 for different types of WWs. El-Rehaili (1994) measured TOC using a Dohrmann DC 190 TOC analyser and the author found the following relationship: $BOD<TOC<COD$.

Total Oxygen Demand (TOD)

TOD is a more recently developed high-temperature (900 °C), rapid (5 minutes) combustion method, which makes use of zirconium oxide or platinum lead fuel cell. A constant amount of oxygen is added to a nitrogen carrier gas via a permeation chamber. Within the temperature controlled (60 °C) chamber, oxygen permeates through a length of permeable tubing carrying the nitrogen, yielding a constant oxygen concentration. The sample is introduced to the reaction oven through a valve. During the oxidation of the sample, oxygen is consumed, resulting in a lowering of the oxygen concentration of the carrier stream, which is sensed by the detector. TOD offers the advantage of simplicity of hardware because the analysis requires no reagents and is not affected by IC concentration and does not require acidification or sparging. TOD also offers the advantage of determination of non-carbon substances e.g. ammonia, nitrates, sulphites, iron, and purgeable organics in the sample (Queeney and Hoek, 1989), which can also be a disadvantage. TOD reflects the oxidation state of the chemical compound (Lueck *et al.*, 1981). Stephenson and Judd (1995) stated that, in general, TOD was higher than COD. Queeney and Hoek (1989) used the Ionics Model 7800 TOD Analyser (Ionics, Inc., Watertown, Massachussetts) and were able to derive linear relationships relating TOD to both BOD and COD providing effective on-line control. However, the sample needed filtration and tight maintenance requirements.

Optical Sensing for Measuring Organic Strength

Absorption at particular wavelengths (such as 254 and 280 nm) has been found to correlate well with BOD, COD and TOC values. The UV absorbance at a wavelength of 254 nm has been found by most authors to provide a good correlation with the dissolved organic matter (e.g. MacCraith *et al.*, 1994). In principle, the absorption technique should provide a non-invasive and real-time method for the monitoring of the TOC, which can be correlated to the BOD (cited by Ahmad and Reynolds, 1999). However, instruments based upon absorption require optical components to be in constant contact with the sample. In addition such instrumentation usually requires pre-sample filtration and frequent washing leading to increased maintenance. Ahmad and Reynolds (1999) stated that efforts to utilise this technique for industrial process monitoring have been frustrated by the rapid fouling of the light transmitting windows of the cell. A brief cross-comparison study, focusing upon several optical techniques including UV (254 nm) absorption spectrometry and optical fluorescence (excitation at 340 nm, emission measurement at 420 nm), together with established chemical methods for estimating TOC in a small range of water samples, has been made by MacCraith *et al.* (1994). The work has shown a useful correlation between proprietary measurements and both the near-UV absorption and fluorescence measurements made. The benefits of optical methods for the monitoring of TOC are many, as absorption and fluorescence do not require the addition of reagents or any special preparation of the sample and the processes involved in the monitoring are rapid (MacCraith *et al.*, 1994). Spectroscopic techniques are well established and it has been applied to WW for on-line non-invasive monitoring of WW quality. Ahmad and Reynolds (1999) study have shown that there is a correlation between the BOD value of a WW sample and the fluorescence intensity at 440 nm from the same sample, however, the correlation is expected to be plant site-selective.

2.7.2. Colour

The visible spectrum contains regions' recognised by the human visual system in terms of colour e.g. between 490 and 500 nm is the colour red (cited by Warren, 1994). Since the issue of colour pollution became a major problem and in-depth research into dyehouse effluent treatment started, numerous methods of quantifying the extent of dye pollution have evolved. Here only two methods are referred: the method based on the British Standard (BS) 6068 (1995) and the American Dye Manufacturers' Institute (ADMI) method.

There are a few important colour terms referred to in the BS 6068 (1995). *Colour of water:* optical property that causes the changing of the spectral composition of transmitted visible light. *Apparent colour of water:* colour due to dissolved substances and suspended matter not dissolved; determined in the original water sample without filtration or centrifugation. *True colour of water:* colour due to only dissolved substances; determined after filtration of the water sample through a membrane filter of pore size 0.45 μm. For the determination of the 'true colour' using an optical instrument e.g. spectrophotometer (with an approximately range from 330 to 780 nm), the WW shall be examined for 3 wavelengths in the visible spectrum (436, 525 and 620 nm) and the result is an average of these three. Different colours cause maximum absorption at different wavelengths of the incident radiation. Strongly coloured water samples may need to be diluted before determination. Colours often depend on temperature and pH, therefore, the temperature and pH of the water sample are regularly determined in parallel with optical measurements and these results are reported with the other findings.

Different authors reported the use of distinct wavelengths for measurements, for example Lopez *et al.* (1999) used the 426, 558 and 660 nm wavelengths and Pak and Chang (1999) only the 410 nm as it is the maximum wavelength for absorbance of reactive dye yellow H-E4R. The Environmetal Agency in UK uses a methodology for defining consent limits and assessing compliance. It consists of the following: after filtration the absorbance values of the sample measured in a 1 cm cell normally at 50 nm intervals across the visible spectrum i.e. 400 - 700 nm. However, exceptions occur if intermediate peaks are present (Waters, 1995). From these absorbance values, percentage removals at each wavelength can be calculated, and from this the mean colour removal can be calculated for a particular treatment technique. Consent limits are usually expressed as absolute limits for the wavelength within a particular range. The permitted colour of the discharge is calculated as cited in O'Neill *et al.* (1999a).

Munsell's approach forms the basis for another colour measurement method, the ADMI method (Allen *et al.*, 1973; Laing, 1991). The ADMI colour numbering system has the principle that a light colour has a low value and a darker shade have a higher value. Different colours can have the same value since the Munsell number refers to colour strength rather than any colour in particular (i.e. two different colours could have the same value if they were both different from pure white by the same degree). This ADMI system is more accurate than other colour systems because it is somewhat independent of hue, but most sensitive to red (Michelsen *et al.*, 1993). Therefore, the main advantage to this system is that it accounts for human perception of colour, and therefore it is practical for aesthetic applications. Wavelengths outside the visible spectrum are not considered since they are invisible to the human eye. Carliell *et al.* (1996) attempted to apply ADMI colour measurements to anaerobically digested textile wastes but, due to residual turbidity, the measurements proved unsuccessful. Ganesh *et al.* (1993) successfully applied the ADMI method to textile wastes, reporting decreases from influent to effluent of 1197 and 877 ADMI units in one day in two separate digesters respectively. Other researchers have also used this method for colour monitoring e.g. O'Neill *et al.* (1999a) and Michelsen *et al.* (1993). However, both laboratory procedures and data calculations of the ADMI method are considered very tedious, so it has not been applied to field practice except in laboratory studies. However, the method has already been automated and used for feedback control to adjust the chemical dosing of oxidant for colour removal.

2.7.3. Aromatic Amines

Knowledge of qualitative and quantitative measurements of aromatic amines is important as the efficiency in the biotreatment stages can be assessed. Aromatic amines analyses were performed by HPLC by various authors e.g. Zissi and Cybertos (1996) and O'Neill *et al.* (2000b). HPLC-MS was used by Straub *et al.* (1992) to analyse and characterise azo and diazo dyes. However, other methods have been used. Schmidt *et al.* (1998) used solid phase extraction followed by derivatisation of the analytes to their corresponding iodobenzenes which were then analysed by means of a GC and electron-capture detection. Razo-Flores *et al.* (1997) analysed aromatic amine mixture by GC-MS and also colorimetrically at 440 nm after reacting with 4-dimethylaminobenzaldehyde-HCl according to a method previously described.

2.8. Modelling and Control of Biological Treatment Processes

Garrett (1998) cited that John Andrews, Professor Emeritus at Rice University, has often said 'The course, which is usually titled Design of Water and WW Treatment Plants should instead be titled Design and Operation of Water and WW Treatment Plants with it being understood that operations include the quantitative description of dynamic behaviour and the use of control systems to convert unsatisfactory dynamic behaviour to satisfactory behaviour'. Control of biological processes is a multidisciplinary subject and it is reasonable to expect different approaches to mastering and practising it. Both, a strictly theoretical point of view and an *ad hoc* approach relying on intuition and hands-on experience are needed when designing advanced control systems (Steyer *et al.*, 1995, 2000). Olsson and Newell (1999) published an excellent book on modelling, diagnosis and control of WWTPs. An IAWQ workshop on Instrumentation, Control and Automation (ICA) in WWT (Olsson, 1993) listed the following incentives for ICA: tighter effluent quality standards and charges according to the effluent pollution; possibility for water recycling; decrement in landfill of sludge; constant improvement of sensors; and need for operational models. The same workshop listed also constraints such as: automation has been considered costly and has not been part of the initial design; there is a lack of reliable on-line sensors today; most sensors today do not allow long periods without extensive maintenance and calibration; most software systems are proprietary and are often quite unfriendly for the user.

Dynamic modelling of biotreatment processes has been largely studied and has shown to be a very powerful tool to improve monitoring and control of WWTPs (e.g. Steyer *et al.*, 1995). Forecasting also plays an essential role in the control of dynamic systems (Harremoes *et al.*, 1993). Olsson and Newell (1999) stated that predictive controllers are the most successful of the advanced multivariable controllers in use in the process industries. Raychaudhuri *et al.* (1996) reviewed the growth and development of control engineering, leading to modern adaptive methods and finally to autonomous intelligent control. Methods to model and control plants with linear characteristics and unchanging parameters already exist. However, non-linear plants with time-varying internal parameters are more challenging and the so-called 'adaptive' methods have been developed to address this issue. Also other techniques have evolved with the abundance of powerful personal computers (PCs) that can 'learn' by using AI techniques such as ESs, fuzzy logic, search and genetic algorithms, ANNs, among others. 'Intelligent control' and 'neurocontrol' are terms that are recognised in the literature today as methods distinct from the more 'conventional' control methods, which included 'adaptive' and 'stochastic' approaches for designing control systems (Raychaudhuri *et al.*, 1996). Some researchers believe that a mixture of the so-called 'intelligent' and 'conventional' methods may be the best way to implement autonomous systems.

Adaptive means that a model is capable of adjusting to changes in both input and behaviour of the system (Novotny *et al*, 1990). Adaptive linear control of a non-stationary biological process is possible, however, the stability is only guaranteed for a defined set-point (Weiland and Rozzi, 1991). A non-linear adaptive control strategy overcomes the problems related to the non-linear behaviour of the biological process by external linearisation. It makes possible the construction of linear systems made of the controller and the process, where the non-linear characteristics of each other are 'neutralised' (Weiland and Rozzi, 1991). This type of model works on a similar principle to a moving average (Fell, 1999). The variance allowed in the average is determined using previous data. This way the model is able to differentiate between background variability and significant disturbances likely to damage system performance (Fell, 1999). The key idea of the adaptive control design is to take advantage of what is well known about the dynamics of bioprocesses (basically reaction network and mass balances) while accounting for the uncertainty (mainly the kinetics) (Steyer *et al.*, 1995).

RTC for WWTPs is nowadays considered a desirable goal for medium- to large-sized utilities for attaining better treatment efficiencies and improved compliance with discharge permit limitations (Capodaglio, 1994a). Control techniques such as deterministic models, stochastic models, ESs, fuzzy logic and ANNs were compared by Capodaglio (1994b) (i.e. in terms of speed, accuracy, confidence, adaptability and others) and evaluated against the RTC requirements (i.e. robustness, implementability, cost-factoring, and others).

Deterministic mathematical models rely on differential equations and kinetic parameters and coefficients to describe the process. Although, they may be useful for design and off-line simulation of treatment facilities, they are generally slow, require recalibration if system parameters change, and may be hard to reconfigure if the physical system is modified (Capodaglio, 1994b). Stochastic modelling (synonymous of time series analysis) is the methodology that deals with the study of a set of observations generated sequentially in time (Harremoes *et al.*, 1993). Time series analysis processes of the Box-Jenkins type can be employed with good results for the identification of models of dynamic systems in a RTC scenario (Capodaglio, 1994a). Several applications of stochastic models to forecasting and control of treatment process time series have been reported in the literature. Univariate and multivariate processes were applied to predict daily values of several operational variables at the Green Bay (Wisconsin, USA) WWTP with good results (Capodaglio *et al.*, 1992). These concepts have been incorporated into a prototype RTC system for the control of an activated sludge plant during hydraulic transients (Novotny and Capodaglio, 1992). Stochastic models are fast, forecasts are fairly accurate and confidence bands are provided, may be self-adapting to changing system conditions, depending upon implementation; 'black-box' type of models but they may provide insight of the physical behaviour of the system (Capodaglio, 1994b).

Capodaglio (1994b) evaluated ESs as follows: speed depends on the implementation (magnitude of database and number of rules); depends on the assembling expert's experience; may not be generally applicable to every system or easily adaptable depending on specific software; robust, unless programming is incomplete; it is generally difficult to collect the expert's knowledge. Capodaglio (1994b) concluded that the fuzzy set theory is as follows: robust; more difficult to understand than ESs; easily adaptable if software is user friendly; priorities must be specified as functions; useful for approaching objective at a global level. The same author observed that ANN models have a fast speed of execution; good accuracy and confidence if adequate 'learning' is provided; self-adapting; impossible to extract additional information other than simulation results; and generally robust.

2.9. Conventional Modelling and Control for Biotreatment Systems

This section contains a brief review of the 'conventional' modelling and control systems applied to anaerobic and aerobic treatment systems.

2.9.1. Conventional Modelling and Control for Anaerobic Treatment Systems

The 'tools of the mathematical modeller's trade are the kinetic equations, rate constants, mass-balances and conversion coefficients that he/she uses to describe the process' (McCarty and Mosey, 1991). Mathematical modelling has gained increasing importance for better understanding of anaerobic degradation and assisted in design and operation of anaerobic reactors. Several dynamic anaerobic digestion models exist in the literature (e.g. Graef and Andrews, 1974; Costello *et al.*, 1991a,b; Marsili-Libelli and Beni, 1996), which account in depth for biological degradation of organic matter, pH-calculation and BA balancing. Reference to various mathematical models has already been made in earlier sections.

Mechanistic dynamic models of the anaerobic treatment process have evolved from models based on a single group of bacteria converting a single substrate to CH_4 (Andrews and Graef, 1971), to models based on complex metabolic pathways mediated by several different groups of bacteria that convert degradable organic molecules to a variety of intermediate and end products (e.g. Mosey, 1983; Rozzi *et al.*, 1985). Costello *et al.* (1991a) derived a dynamic model of a single-stage high-rate anaerobic reactor, based on the work of Mosey (1983) and included six groups of bacteria, by defining the biological make-up of the anaerobic ecosystem, the physico-chemical system, and the reactor process. The model included the accumulation of lactic acid under specific process conditions. Product inhibition and pH inhibition of each group of bacteria were represented in the model. Costello *et al.* (1991b) verified the developed model by comparing its predictions with previously reported experimental data.

The classical Michaelis-Menten model is widely used as the basis for modelling of a number of biological systems. Hoh and Cord-Ruwisch (1996) proposed a kinetic model, which allowed for the effects of substrate inhibition without the need to determine a large number of parameters experimentally. A simplified mathematical model for the behaviour of anaerobic digesters under shock loading conditions was derived by Marsili-Libelli and Beni (1996) with a special emphasis in BA. The model was then used with success to simulate other kinds of shock and to assess the design of a bicarbonate dosing PID controller.

Anderson and Yang (1992) mathematical model was able to predict the pH, the concentration of bicarbonate in the effluent, and the percentages of CH_4, CO_2, and H_2S in the biogas from several laboratory-scale reactors of different configurations treating different types of influent. The model showed that the composition of the WW, such as the concentration of VFA, sulphate, nitrogenous compounds, and COD had a profound effect on the chemical demands for pH control in anaerobic digestion.

Modelling and simulation of the anaerobic treatment of whey WW was performed by Ryhiner *et al.* (1993). A model including five groups of organisms, whey substrate, butyric, propionic, and acetic acids, CO_2, H_2 and CH_4 was developed for single- and two-stage anaerobic fluidised bed reactors. It included thermodynamic limitations for the acetogenesis reaction, acid-base equilibria of dissociating compounds (organic acids and CO_2) for variable and constant pH situations, gas-liquid phase mass transfer, and measurement dynamics. The model was adequate to describe both the steady-state and dynamic behaviour of the reactors. Controls of pH, dissolved H_2, and organic acids, by manipulation of the feed flow rate, were successful in simulation and experiment. Simulations were used to determine optimal control parameters for conventional PI or PID control. Simple control using gas analysis results did not give stable control, either in the simulation or in the experiment. A comparison of PI and PID controls showed that because of the negligible delay time the differential part did not give any improvement.

Merkel *et al.* (1999) studied the population dynamics in anaerobic reactors. The effects of changing WW composition and SRT on an anaerobic mixed population were modelled and compared with the results from continuous degradation studies of a lactic, butyric, propionic and acetic acid containing WW. The mathematical model was successful in simulating organic substrate degradation, pH in the reactor, liquid-gas transport of gaseous products and the long-term development of the anaerobic consortia.

A review of control studies applied to real processes can be found in the excellent paper by Heinzle *et al.* (1993). The controller type on-off, P, PI, PID, adaptive, and generic model control were used by various researchers (Heinzle *et al.*, 1993). However, as reviewed by Heinzle *et al.* (1993), most of the applied control algorithms were conventional PID type and comparisons of experimental data with simulation results were rather scarce. A PID algorithm is much more sophisticated than the simple on-off controller and has normally a much improved performance to match. Several questions must be addressed when implementing the controller such as a proper sampling interval and tuning (Olsson, 1992). It is necessary to determine values for the three constants and this determination requires a three-dimensional search in the feature space of the variables to be performed (Wilcox *et al.*, 1994). In some instances it is not always necessary to utilise all the controller terms. Denac *et al.* (1990) successfully regulated the effluent quality of an anaerobic fluidised bed reactor, expressed in TVFA concentration, using an on-off control algorithm, with alkaline consumption as the controlled variable and feed rate as the manipulated variable during an organic overload, and found it gave a reasonably constant effluent quality level. However, the authors stated that in the case of WWs of widely varying composition and alkalinity, an adaptive algorithm may be necessary. Because anaerobic processes are complex, non-linear and non-stationary, conventional control is often unable to regulate this type of process, in a simple and stable manner (Polit *et al.*, 1995). von Sachs *et al.* (2000) proposed a control strategy, a 'conducted' PI algorithm, for the two-stage anaerobic digestion of textile WW containing sodium chloride. The conduction was performed by certain logical rules for specific constraints on the dilution rate. The control law used the relative specific CH_4

production rate as control variable and the dilution rate as manipulated variable and it was successfully tested on a laboratory scale digester.

Non-linear adaptive control algorithms have been developed by various researchers e.g. Bastin and Dochain (1988), Renard *et al.* (1988) and van Breusegem *et al.* (1990). Bastin and Dochain (1990) reviewed the principles of adaptive control methods and their application to bioreactors. Dochain *et al.* (1988) proposed algorithms that did not require any analytical expression for the fermentation parameters (like the specific growth rates) and assumed that the dilution rate was the manipulated variable. They have adaptively controlled the substrate concentration, the BA and the H_2 concentration. They used VFAs, BA and H_2 concentration (also considered by Dochain *et al.*, 1991) as control variables in simulation. As the model is generally non-linear, the model-based control design will result in an adaptive linearising controller (Bastin and Dochain, 1990), in which the on-line estimation of the unknown variables (component concentrations) and parameters (reaction rates and yield coefficients) are incorporated. An adaptive linearising controller based on COD measurements has been designed, theoretically analysed and experimentally validated on pilot anaerobic digesters (e.g. Renard *et al.*, 1988), however, this control solution need on-line COD (or equivalent substrate concentration) measurements. Alternatively, Dochain *et al.* (1991) suggested the use of H_2 as a controlled variable. In this control scheme a four organism Mosey type model was used and the H_2 level was controlled by measuring the inflow glucose, the outflow H_2 gas and the liquid phase H_2 concentration on-line and by manipulating the inflow rate. Numerous simulations tested the controller under a range of conditions, including a comparison with simple PI control and this approach appeared to be a promising way of including some of the model complexities into an adaptive controller design (Heinzle *et al.*, 1993). Adaptive linearizing control strategy has also been tested by van Breusegem *et al.* (1990) through simulations, indicating that BA control would be desirable and successful, by manipulation of the dilution rate. This control allowed maintaining the VFAs level within admissible bounds. Perrier and Dochain (1993) evaluated adaptive non-linear controller designs for various controlled variables for the operation of the anaerobic digestion process. The selected control variables were the COD, propionate concentration and dissolved H_2 concentration, being the manipulated variable the dilution rate. In addition the controller required knowledge of the CH_4 flow rate and influent substrate concentration. Their simulation results have shown that each controller was able to maintain the controlled variable at the desired set-point after a step change in influent substrate concentration.

Polihronakis *et al.* (1993) developed three non-linear adaptive control algorithms for a CSTR type of anaerobic reactor, for control of: substrate concentration, CH_4 production rate and a strategy which is a combined method of the two previous control schemes. They measured gas flow rate, CH_4 and CO_2 % in the gas, pH, and temperature. These algorithms were applied on a municipal WWTP with success. Johnson *et al.* (1995) described the application of an adaptive algorithm for the monitoring and control of industrial effluent fed laboratory-scale anaerobic filters during shock loads. They used deduced on-line COD (from on-line conductivity and turbidity measurements), gas flow and CH_4 to vary the influent pumping rate. The control model used was based on that reported by Renard *et al.* (1988). Fell (1999) later developed an adaptive control strategy for coping with organic load fluctuations and the toxicity effect from some fractions of the instant coffee production WW. The control strategy involved the same on-line measurements as in Johnson *et al.* (1995). The control system was shown to be effective at smoothing out organic load fluctuations to the digesters by controlling the feed pump. However, severe inhibition and failure of the digestion process was demonstrated to occur in the presence of a toxic substance.

Monroy *et al.* (1996) presented the design and implementation of an adaptive controller for anaerobic digesters using a general non-linear model and an uncertainties estimation scheme. The resulting controller was similar in form to standard adaptive controllers and could be tuned analogously. It does not require biogas flow rate measurements only the substrate COD. Implementation of such control strategy was carried out in a pilot-scale digester under changing environmental (temperature, pH) and feed load conditions. They compared the control results with those obtained with a conventional adaptive strategy and found good performance and robustness against changes in the feed load even though gas measurements were not used.

2.9.2. Conventional Modelling and Control for Aerobic Treatment Systems

Dynamic models of enhanced complexity have been developed in order to incorporate a more detailed description of the reactions and phenomena taking place in activated sludge reactors. In comparison there has been a lack of structure in many thickener/clarifier models (Jeppsson and Olsson, 1993). Activated sludge models (ASMs) range from the simple steady-state models (e.g. Eckenfelder, 1980), which are widely utilised, to the more complete and complicated dynamic models, that take into account changes of flowrate, composition and concentration of the influent WW (El-Rehaili, 1994). ASM1 allows for the dynamic simulation of nitrification-denitrification in a variety of activated sludge flow schemes (Henze *et al.*, 1987). It has been proven to be a successful model in many applications for BOD and N removal. However, the complexity of the model and the detailed WW and microorganisms growth and decay data required by the model made it unappealing in the past for utilisation by process designers and operators. ASM1 requires determination of about 35 parameters, coefficients and variables and a large number of them are not routinely measured even by large WW disposal utilities (Grady, 1989) or are of uncertain determination. Chen and Beck (1993) described the development of a multiple-species model (modified from the ASM1) of the activated sludge process (reactor and settler). The model of the system incorporated within a Kalman filter was for on-line forecasting i.e. for predicting the likelihood of bulking occurring; and on-line estimation of the composition of the biomass and the relative growth rates of the floc-forming and filamentous bacteria. Cheng and Ribarova (1999) investigated the feasibility of upgrading a conventional activated sludge process for biological N removal. They used the ASM1 as a base for modelling of the activated sludge system with calibration with data from the plant operation. Very good correlations between measured data and simulation results were achieved, however, they found that characterisation of the actual WW was necessary. Vanrolleghem *et al.* (1999) presented a concise overview of respirometric experiments for the calibration of ASM1.

The ASM2 further accounted for biological phosphorus removal (Henze *et al.*, 1995; Zhao *et al.*, 1999). This model provided detailed biological kinetics and reflected the state of art on the understanding of nitrification, denitrification and biological P removal. The ASM2D, an extension of ASM1 and ASM2, is a model for biological P removal with simultaneous nitrification-denitrification in activated sludge systems (Henze *et al.*, 1999). The ASM3 can predict oxygen consumption, sludge production, nitrification and denitrification of activated sludge systems (Gujer *et al.*, 1999). ASM3 includes storage of organic substrates as a new process and the lysis (decay) process was exchanged for an endogenous respiration process. It is provided in a form, which can be implemented in a PC code without further adjustments. It does not include biological P removal as it is contained in the ASM2.

One of the major reasons for developing dynamic models is for use in process control systems, which are designed to convert unsatisfactory to satisfactory dynamic behaviour. The types of conventional controllers applied to the activated sludge process are on-off, PID, cascade, self tuning, and adaptive (Andrews, 1992). The author also stated that it is usually not necessary to apply complex multi-input multi-output (MIMO) controllers, as the plant can be controlled by many simple controllers instead.

DO concentration is regarded as the most important control parameter in the activated sludge process. Today, DO control is the standard at Scandinavian plants and DO sensors are used in almost 100 % of the full scale plants (Olsson *et al.*, 1998). The control of the pH process plays also an important role in activated sludge process. However, pH control has been a difficult problem due to its non-linearities and time-varying properties (Shinskey, 1994). Many authors proposed non-linear pH control strategies to overcome these drawbacks (Lee *et al.*, 1998). These researchers developed an automatic control for DO and pH. A discrete type auto-tuned PI controller using an auto-regressive exogenous (ARX) model as a process model was developed to maintain the DO concentration in aeration tanks by controlling the speed of surface aerators. Also a non-linear pH controller using the titration curve was used to control the pH of influent WW with NaOH addition. They stated that both controllers were successful and that the electric power consumption of surface aerators was reduced up to 70 % with respect to the full operation when the DO set point was 2 mg l^{-1} and overall improvement of the effluent water quality was achieved by DO control.

Klapwijk *et al.* (1993) presented an integral control strategy for a maximum respiration rate in activated sludge plants for the following control tasks i.e manipulation of the influent flow rate and of the WAS. It also monitored the instantaneous respiration rate for performance information. They concluded that the control strategy based on respiration rate measurements could be used: to have an optimal biomass concentration; to treat the maximum WW; with a minimum amount of air; while at every moment the effluent quality should meet the standards. Later, Brouwer *et al.* (1994) developed a control system for an activated sludge plant based also on respirometry (Spanjers and Klapwijk, 1990). They concluded, by simulations, that controlling the respiration rate by manipulating the WW flow rate was a satisfactory method preventing huge fluctuations in the COD load of the carrousel, and on the WAS flow rate, and a day/night control of the aerators would result in energy cost savings between 11 and 21 % (at night - lower energy costs, the respiration rate was controlled at a higher value).

Lindberg and Carlsoon (1996) stated that the use of adaptive controllers was appropriate to identify unknown or time-varying parameters and to compensate for the lack of direct process measurements. They developed non-linear and set-point control of the DO concentrations in an activated sludge process. Simulations illustrated that a non-linear PI DO controller outperformed a standard PI controller, which was also confirmed in a pilot-scale plant experiment.

2.10. The Use of AI Techniques for Modelling and Control of Biotreatment Processes

The potential of AI was first described in the 1940s, but the computational power required to use it as an on-line control technique was unavailable at that time. Since then the extraordinary advances in the computing field has resulted in the ability to utilise AI technology more thoroughly. However, it was only since 1980s that there has been a large increase in the number of publications on AI applications in a multitude of fields. According to Russel and Norvig (1995), there are numerous definitions of AI and are organised into two different approaches. In a human-centred approach it must be an empirical science, involving hypothesis and experimental confirmation. In a rationalist approach involves a combination of mathematics and engineering. Among AI tools and techniques are data base search techniques, logic and deduction methods, ESs, fuzzy logic, qualitative simulation, and ANNs. The recent development of these approaches in the design and operation of biological processes is reviewed by Shioya *et al.* (1999). The primary attraction of AI techniques is that they are able to represent systems with non-linear characteristics, without the (often) difficult task of dealing with deterministic non-linear mathematics.

Existing modelling strategies may be divided into three categories, namely, 'white-box', 'black-box' and 'grey-box', based on the type of knowledge used for the model development (Bailey, 1998). In the 'white-box' modelling strategy, the model development is mainly driven by the knowledge of the relevant mechanisms and balances. The 'black-box' model is, on the other hand, mainly driven by measured data obtained from the process e.g. ANN models (Shioya *et al.*, 1999). However, ANN based models are only as good as the data that was used to calibrate them (Maier and Dandy, 1997). Since 'black-box' models such as ANNs are not believed to have any extrapolation properties, one has to obtain a large body of data for process identification by employing the relevant input variables with a range of fluctuations. A 'grey-box' model may be defined as a suitable combination of 'black-box' and a 'white-box' model, with the expectation of obtaining good interpolation and extrapolation properties (Shioya *et al.*, 1999). An example is shown in Côté *et al.* (1995).

ESs and fuzzy logic rely on rules which have very intuitive basis such as *if<condition> then<outcome>*, although there are significant differences in implementation. Advantages and disadvantages of rule-based systems were already referred to in Section 2.8. Fuzzy rules can be defined just as conventional rules. However, the value portion of the attribute-value pairs are defined by fuzzy sets using fuzzy membership function, which usually has values between 0 and 1 (Andrews, 1992). Fuzzy models are a compromise between the vague statements which humans often use and the strict logic of ESs and quantitative answers provided by other mathematical models. No complex mathematical relationships are required in the construction of fuzzy logic applications. Besides, it is conceptually easy to understand, flexible and tolerant of imprecise data allowing the modelling of complex non-linear functions (Puñal *et al.*, 2000). Fuzzy control theory can easily be extended with additional heuristic rules when for example information on the expected influent or sludge characteristics is to be applied (Kalker *et al.*, 1999). The same author stated that a drawback of fuzzy control is that tuning may be time consuming since there are many tuning parameters. Also, the main difficulty is to define the number of necessary rules which can be wording rules or functional rules (numerical laws) (Jauzein, 1998). Fuzzy logic may also be mixed with conventional control techniques.

The most commonly used tool in WWT is the ES (Andrews, 1992). Since the middle of the 1980s, several demonstration and research projects using ESs for control of WWTPs have been started in different countries such as Canada, USA, Japan, Sweden and Denmark (Harremoes *et al.*, 1993). Known examples of intelligent control of WWTPs are in the city of Vienna, which has been using fuzzy control since 1992. Hoechst has used fuzzy control in the aerobic processing step in WW recycling plants in Germany (Manesis *et al.*, 1998). Müller *et al.* (1997) described a RTC scheme to cope with input disturbances in WWT processes, based on a fuzzy control system. The plant treated baker's yeast and consisted of a buffer tank, a fluidised bed reactor, aerobic tank, settler, nitrification, settler, denitrification and settler. The control system was designed and tested using a pilot plant, to which a toxic disturbance was applied. The influent flow could be straightly directed to the aerobic stage and the final effluent could be used for dilution of the incoming WW. For this purpose the fuzzy process control system was subdivided into a diagnosis and a control unit. The fuzzy diagnosis unit was based on a fuzzy pattern recognition algorithm using biogas H_2 concentration and biogas flowrate from the anaerobic pre-taster as process indicators. Four typical behaviours (normal, overload, inhibition, toxicity) were selected to train the diagnostic, which evaluated the degree of agreement between the incoming data and the predefined behaviours, in fuzzy terms.

2.10.1. AI Applications for Modelling and Control of Anaerobic Treatment Systems

In the literature, several approaches can be found where ES technology was applied for the anaerobic treatment of WWs. The ES developed by Barnett and Andrews (1992) contained knowledge necessary to diagnose and specify control actions properly for correcting hydraulic, organic, toxic, and ammonia upsets in anaerobic digestion. The measurements were: influent VS, influent flow-rate, pH, VS, VFAs, gas flowrates, $\%CH_4$ and rate of CH_4 production. The manipulated variables were the influent flow rate, sludge recycle, dilution, acid or base addition. Moletta *et al.* (1994) developed an ES to control fluidised-bed laboratory and pilot scale reactors during several kinds of organic overloads. They monitored on-line the pH, biogas production and its concentration of H_2 to be used by the ES. The automatic control system calculated the flow rate of the feeding pump in order to adjust continuously the load applied to the reactor. Holst *et al.* (1995) used an algorithm similar to an ES called Méthaveil to monitor and control a full scale anaerobic fluidised bed reactor. Méthaveil used the same three on-line sensors as Moletta *et al.* (1994). The control output was either that the reactor was: stable and could accept a higher feed flow rate; stable but could not accept a higher feed flow rate; or not stable and the feed flow rate had to be reduced. The controller was tested for an organic overload and the H_2 was seen to increase from a steady base line of 65 ppm to 120 ppm, the control action of slowing feed pump speed resulted in H_2 decreasing to a new higher steady state of approximately 100 ppm. The Méthaveil enabled a rapid start-up of the reactor, allowed the immediate detection of overloads and other abrupt changes in the effluent quality and increased the stability of the reactor by directly controlling the feed flow rate. There were four full-scale anaerobic fluidised bed plants using this system, treating a variety of different WWs i.e. starch, paper and perfume (Holst *et al.*, 1997).

Fuzzy control strategies have also been applied to the control of anaerobic processes by several authors, all reporting some success. Polit *et al.* (1995) used a fuzzy logic control system for a fluidised bed reactor. They measured CH_4 flow rate to control the input flow rate. Their results proved that fuzzy control was a good and simple way to improve the working of this type of digesters. Estaben *et al.* (1997) used also a fuzzy logic controller for a fluidised bed reactor treating wine distillery WW. The gas flow rate and the pH were used as input parameters to the controller and the input flow rate was used as the manipulated variable in order to ensure a good COD reduction. The controller was evaluated with success on a quantitative model, and on a real process. Puñal *et al.* (2000) setup a fuzzy system for the diagnosis and supervision of anaerobic digesters with data from Dochain *et al.* (2000). They monitored on-line the biogas flowrate, feed flowrate, CH_4 and CO_2 concentrations in biogas. The system worked properly after tuning, it could predict the future trend in the system and estimated the best set points for pumps, the system could also validate the on-line results and detect the presence of toxic compounds, which could destabilise the operation. In Bernard *et al.* (2000), a mass balance based model representing the dynamic behaviour of an anaerobic digester served as a basis for the design of software sensors for the concentration of COD, alkalinity and VFAs. These predictions were close to the actual off-line measurements. The models were then used to design a model-based adaptive linearising controller and a fuzzy controller whose objective was to regulate the BA under which the process was assumed to remain in stable conditions and avoid VFA accumulation. The fuzzy controller acted on the input flow rate. They stated that both controllers proved successful and the choice between the two controllers would be imposed by the situation.

2.10.2. AI Applications for Modelling and Control of Aerobic Treatment Systems

A discussion of the application of ESs to the activated sludge process has been given by Barnett (1992). An ES for automatically adjusting phase lengths (nitrification-denitrification) and aeration intensity for an activated sludge nutrient removal was examined by Isaacs and Thornberg (1998) using simulations based on the ASM1.

Tsai *et al.* (1993) used fuzzy control of a dynamic activated sludge process for the forecast and control of effluent SS concentration and further predicted the MLSS concentration in the aeration tank. They measured COD, MLSS, SS, turbidity and DO. It revealed that the control strategy not only enabled one to decrease effluent SS concentration, and hence decrease the effluent BOD, but also made it more stable. The varied RAS and influent flow rate were two major operational factors of those affecting effluent SS concentration. A fuzzy logic based control system was developed and tested by Ferrer *et al.* (1998) in the main aerobic reactor of a process pilot plant and it was compared with one- and two-aeration-level on/off controllers. Energy savings of about 40 % over the one-level on/off controller and a more stable closed-loop response was obtained. Kalker *et al.* (1999) developed two types of fuzzy logic controllers for intermittent aeration control: a low-level fuzzy controller for DO control and a high-level controller for N removal. A model was used to subsequently design and optimise the controller and furthermore to compare various control strategies. The results indicated that the fuzzy controller allowed improvements in comparison with a PI controller, namely in terms of energy consumption and at the same time, resulted in a slightly better effluent quality.

2.11. The Use of ANNs for Modelling and Control of Biotreatment Processes

ANNs contain some features that were inspired by biological neural networks (Westland, 1998) and are comprised of many interconnected neurones or processing elements/units. The human brain can learn, recognise, has intuition and can interpret information (Westland, 1998). The structure of the human brain is extremely complex, with approximately 10^{11} neurones and between 10^{14} and 10^{15} synapses (Willis *et al.*, 1991). Whilst the function of single neurones is relatively well understood, their collective role is less clear and a subject of enthusiastic speculations.

A single neurone cannot do very much. However, several neurones can be combined into a layer or multiple layers that have great power. An ANN is a massively parallel system consisting of large numbers of neurones joined together usually into groups called layers (normally the input, hidden and output layer). A typical network consists of a sequence of layers with the connection weights between successive layers (Jain *et al.*, 1996). ANNs can 'learn' (off-line and on-line) from 'training data' and are in reality a form of mathematical function approximators. ANNs have emerged as very powerful tools for designing intelligent control systems or 'neurocontrol'. ANNs have several advantages over expert control methods. It requires no explicit encoding of knowledge, which makes them well suited to applications where knowledge extraction is difficult or in cases where the interrelationships between process parameters are hard to model. ANNs have what is known as distributed associative memory, in which an item of knowledge is distributed across many of the memory units in the network and is shared with other items of knowledge stored in the network. Therefore, ANNs have the ability to learn and build unique structures specific to a particular problem, such as the indications of a specific type of shock to an anaerobic system. They can generalise, intelligent responses to novel stimuli are possible by the combination of knowledge in the network layers. ANNs have fault tolerance, even if a section of the network is destroyed it will not result in a total breakdown. The generalisation capability of ANNs, along with their adaptive, self-organising, and fault-tolerant characteristics, allow them to learn and make reasonable decisions from incomplete or noisy data. Mohan and Keshavan (1998) stated that because of the highly connected structure, ANNs exhibit some desirable features, such as high-speed via parallel computing, resistance to hardware failure, robustness in handling different types of data, graceful degradation, learning and adaptation. Once a network is trained to perform a particular task, new information can easily be incorporated by re-training and therefore, the ANN becomes a dynamic data analysis tool that grows with the data (Collins, 1990). Actually, a regular re-training schedule must be established as a check of the ANN validity (Boger, 1992).

Neural computing has experienced three periods of extensive activities. The first peak in the 1940s was due to the pioneering work by McCulloch and Pitts (1943) in modelling the function of a biological neurone (modelling of the first single artificial neurone) followed by Hebb (1949) who postulated the learning technique that made a profound impact towards the future development of the field. The second phase took place in the 1960s, due to the concept of the 'Perceptron' (predecessor of the modern MLP network) and the learning algorithm of Rosenblatt (1962) who formulated the first weight adjustment mechanism, followed by the work by Minsky and Papert (1969) revealing the limitation of the single layer 'Perceptron' network. The learning scheme of Rosenblatt (1962) could not solve problems, which require the construction of multi-layer perceptron (MLP) networks in order to deal with complex non-linear problems. As a result ANN research lapsed into stagnation for almost two decades. Since the 1980s renewed interest motivated more work into the development of ANN architecture and the associated learning scheme. Among the many who contributed to this resurgence include Hopfield (1984) who introduced the recurrent network (RN) architectures; Rumelhart and McClelland (1986) on the popular backpropagation (BP) learning algorithm for feed-forward (FF) MLP network or more commonly known as the BP network; Kohonen (1989) for work on associative memory for unsupervised learning network for feature mapping. Neural computing is nowadays, one of the fastest growing areas of AI (Meszaros *et al.*, 1997). In the future, the combination of ANNs with ESs, classical algorithms and complementary tools will generate the more interesting data treatment technologies. The fundamental and complementary characteristics of fuzzy and ANNs technologies have led researchers to combine them in 'neuro-fuzzy' or 'fuzzy-neuro' systems, which are suitable for complex and ill-defined applications (Panigrahi, 1998), for control system design (Raychaudhuri *et al.*, 1996). Fundamentals of neural fuzzy modelling for anaerobic WWT systems have been presented comprehensively by Tay and Zhang

(1999). Collins (1990) presented 15 software packages for ANN development from low cost ones to the most complex and expensive ones.

ANNs are able to map the complex non-linear input/output relationships in the data (Baxter *et al.*, 2001). They are better at interpolating than extrapolating (Boger, 1992). ANNs are well suited to situations where sufficient information/data can be gathered for training and where the use of linguistic rules to interpret the outcome of the complex non-linear process is almost impossible. Careful selection of a data range is needed for training the chosen network to avoid erroneous extrapolation (Boger, 1992). The data set should be sufficiently rich to excite different modes of the bioprocess being studied, and a compromise must be made with respect to the number of parameters, the size of the data set, and the accuracy desired (Karim *et al.* (1997). Boger (1992) advised to have an external program serving as a watchdog by comparing the input values against the range of input values in the training data set to give some warning if a significant input is outside the learning data range. The relevant issues to achieve faster convergence during training and a better trained network are: architecture (number of hidden layers and hidden units); network initialisation procedure (initialising weights and biases); sufficient amount of representative data; momentum term; activation function; and stopping rules (preventing over training). The ability of the network to approximate non-linear functions is dependent on the presence of hidden layers with non-linear functions neurones. Some of the frequently used non-linear transfer functions are of the type threshold, sigmoid, hyperbolic tangent and gaussian (Hunt *et al.*, 1992). The most widely applied non-linearity is a sigmoidal function in the interval (0, +1 or -1, +1) (Montague and Morris, 1994). Two hidden layers could be beneficial, but theory indicated that more than two provided only marginal benefit in spite of the major increase in training time (Ungar *et al.*, 1995). The number of nodes in the hidden layer(s) can be as small or large as required, and is related to the complexity of the system being modelled and to the resolution of the data fit required. A bias is included in order to be able to modify the position of the non-linear function (Montague and Morris, 1994) so that the network can represent better the input/output relationships (Demuth and Beale, 1994). It is important to choose the number of hidden neurones carefully since too many hidden neurones will cause the network to memorise the training examples (and result in poor generalisation) or overfitting (Chitra, 1993) while too few hidden neurones will not be able to capture the underlying trend in the data (Côté *et al.*, 1995; Tholudur and Ramirez, 1996). Thus resulting in learning the noise present in the database used in training (Linko *et al.*, 1998). There is no exact rule to specify the optimum number of hidden neurones needed, and as initial connection weights are not known in advance, random numbers are chosen (Boger, 1992). Overfitting may also be caused by an insufficient database (Boger, 1992).

Network Architectures - ANNs can be grouped into two categories based on the network connection architecture, namely feed-forward networks (FFNs) and RNs. In the FFNs neurones are organised into layers where information is passed from the input to the final output layer in a unidirectional manner whereas in RNs, feed-back connections within the network either between layers and/or between neurones can be found (Jain *et al.*, 1996). In general, FFNs are *static*, they are capable of mapping the given set of inputs to the corresponding outputs i.e. the output is independent of the previous input and output of the network. RNs on the other hand are *dynamic*, meaning the output at time instant *t*, is dependent on the previous output or state of the neurones within the network as a result of the feed-back paths. An excellent introductory material on ANNs, particularly on the various ANN architectures, can be found in the publication by Jain *et al.* (1996). An example of a FFN is the MLP (Rumelhart and McClelland, 1986) and of a RN is the Elman network (Elman, 1990).

Learning - In general the learning process in an ANN involves updating of the network weights and/or architecture in order to efficiently perform a particular function. Very often the network must learn from the given examples by iteratively adjusting the connection strength in the network so that its performance is enhanced with training. ANN learning can be broadly classified into *supervised* and *unsupervised.* Supervised learning as the name implies, requires an external reference (teacher) to pair each input vector to the network with a target vector representing the desired output (Hunt *et al.*, 1992). When an input vector is introduced, the network proceeds to calculate the output, and the error (between the target and the output) is often used to modify the weights according to an adopted learning algorithm that tends to minimise the prediction error. The training input vectors are passed sequentially through the network and errors are calculated followed by weight adjustments for each training iteration, until the error for the entire set of training vectors reaches an acceptable level (as specified by the designer) (Demuth and Beale, 1994; Jain *et al.*, 1996). Examples of supervised learning algorithms include the BP algorithm (Rumelhart and

McClelland, 1986) and the LVQ algorithm (Kohonen, 1989). Unsupervised training requires no target vector for the training input vectors presented to the network, hence, no comparisons are done to predetermine the ideal responses. The training set consists solely of input vectors. The training algorithm modifies the network weights to recognise vectors that are consistent. Hence the training algorithm essentially extracts regularities and correlations in the input data patterns and adopts the network's future responses to the recognised patterns accordingly. Upon the completion of network training, it can also be used to recognise unseen data patterns by virtue of the ANNs generalisation ability. Examples of unsupervised learning algorithms include the self organising map (SOM) (Kohonen, 1989) and adaptive resonance theory (Carpenter and Grossberg, 1987).

ANNs have been trained to perform complex functions in various fields of application including pattern recognition, identification, classification, speech, vision and control systems. Demuth and Beale (1994) stated some examples of areas where ANNs have been applied to: aerospace, automotive, banking, defence, electronics, manufacturing, medical, oil and gas exploration, robotics, speech, securities, telecommunications and transportation. In the recent years, ANNs have been applied to address real life problems. In the UK, the Department of Trade and Industry (DTI) has created a web site (DTI NeuroComputing Web, 1998) in their effort to encourage ANN implementation by industry. It covers practical issues concerning the application of ANNs, highlighting many useful aspects such as the feasibility, costs, design, planning and provide pointers as to commercial consulting entities, as well as the range of available software and hardware currently available. However, it is believed that the field is still in its infancy despite a colourful history and the much celebrated success, and much more work will have to be done before this field can realise its full potential (Zurada, 1992).

The application of ANNs in water and WW treatment engineering has drawn much interest to address a variety of issues such as modelling and prediction of a treatment process operation (e.g. Barnett and Andrews, 1992; Boger, 1992), water and WW treatment plant performance evaluation (Pu and Hung, 1995), plant control and optimisation (Ladiges and Monnerich, 1996; Stanley *et al.*, 2000), and plant design (Krovvidy and Wee, 1990). Baxter *et al.* (2002) developed and implemented an ANN model-based advanced process control system for the coagulation process at a pilot-scale water treatment facility in Edmonton

(Alberta, Canada). These authors stated that ANN technology is the most powerful modelling tool currently available to the drinking water treatment industry. However, Syu and Chen (1998) stated that there are still very limited applications of ANNs on WWT. The ES approach is the most prevalent, but difficulties in acquiring and representing knowledge of the complex phenomena in these plants have led to the search for additional approaches. Boger (1992) modelled operational variables at a WWTP in Israel with an ANN system. In this application, it was shown that ANN models' performance was improved by optimising the data input set: from an available set of 106 variables, only 15 to 20 significant inputs were required to identify the optimal ANN for process simulation. Mohan and Keshavan (1998) used a three-layered ANN to model the BOD series of a sewage treatment unit. Time and influent BOD concentration were the inputs while effluent BOD concentration was the output from the network. Input and target vectors were normalised to the range of [0.2, 0.8] before being fed to the network. They found that when the number of hidden neurones was increased the convergence was faster and the minimum sum squared error (SSE) was also significantly lower as the number of local minima in the error surfaces with more hidden layer units is lesser. The training with bias converged faster and resulted in lower values of SSE. They showed that with both long series and short series of data, the ANNs have produced comparable results. They concluded that ANN models are robust and provide good predictions for the performance of the WWTPs. Hamoda *et al.* (1999) developed an one hidden layer BP network to model a municipal WWTP (Kuwait). Results obtained proved that ANNs present a versatile tool in modelling full-scale WWTPs and provided an alternative methodology for predicting the performance of WWTPs. They stated that the best ANN structure did not necessarily mean the most number of hidden layers.

Hunt *et al.* (1992) published a survey on ANNs for control systems. In process control applications ANNs can be incorporated in the controllers in either direct (inverse model) or indirect (model) control methods. Shaw (1990) discussed the use of ANNs for process monitoring and alarming. The review article by Samad (1991) discussed the application of ANNs to various aspects of process control (e.g. modelling and process identification). Montague and Morris (1994) published an excellent review of the ANNs contributions namely modelling, control and pattern recognition in biotechnology. An ANN based strategy outperformed the conventional PID control of a pH neutralisation in a CSTR (Nahas *et al.*, 1992).

An argument for not using ANNs is that training time may be long and that the training methods are not optimised. Training time can be of the order of days for a complex problem, but compared with the time taken to code an ES it is insignificant. Optimised training methods and newer ANN topologies can reduce the training time (Collins, 1990). Although ANNs basically operate as a 'black-box' model, they can be used to determine the strength of the relationship between ANN inputs and outputs (Maier and Dandy, 1997). Therefore, these authors disagreed with the statement that 'ANNs are unsuitable for knowledge acquisition purposes'. Table 2.3 shows some examples of processes modelled and/or controlled using ANNs.

2.11.1. Types of ANNs

According to Hecht-Nielsen (1988), in 1987 there were approximately 50 different types of ANNs being studied and/or used in applications. However, here, only the types used in the developing the ANNBCS are briefly described.

Linear network

A single-layer linear network can perform linear function approximation or pattern association and can be designed directly if all input/target pairs are known and trained with the Widrow-Hoff rule (Widrow and Sterns, 1985) to find a minimum error solution. Linear networks can be trained adaptively on-line allowing the network to track changes in the environment. However, linear networks can solve only linear problems.

BP network

The FF MLP network can be trained with the popular error BP learning rule (Rumelhart and McClelland, 1986). The learning paradigm utilises a gradient descent method, which adjusts the initial weights assigned to the network, by an amount proportional to the partial derivative of the error function with respect to the given weight. More information on this can be found in Demuth and Beale (1994) and Jain *et al.* (1996). The number of elements in the input and output layer are solely dependent of the number of features associated with the input and output vectors, whilst the neurones in the middle or hidden layer(s) are generally subjected to empirical evaluation. The BP learning rule is a typical supervised learning procedure and is as follows (Jain *et al.*, 1996):

1. Initialise the weights of the network at random values between 0 and 1;
2. Present input vectors together with the desired target to the network;
3. The network then proceeds to calculate the output based on the input vectors presented, and then compares this output with the target, to evaluate the error;
4. Adjust the weights of the network using the error BP learning rule to improve the overall network performance so as to achieve the desired target;
5. Repeat steps 2, 3 and 4, and calculate the SSE of the network; If the SSE of the network is within an acceptable range, then terminate the training process, if not, go back to step 2.

According to Hornik *et al.* (1989) multilayer FFNs are a class of universal approximators. Learning rate is a parameter used to influence the size of adjustments made to networks weights during training (Montague and Morris, 1994). Picking the learning rate for a non-linear network is a challenge. A learning rate that is too large leads to unstable learning and if it is too small results in incredibly long training times. Fu and Poch (1995) stated that the higher the learning rate, the bigger the final total error will be. The function *trainbpx* uses techniques called momentum and an adaptive learning rate to increase the speed and reliability of the network. Momentum decreases the sensitivity of the network to small details in the error surface, helping the network to avoid getting stuck in shallow minima, which would prevent the network from finding a lower error solution. The momentum term results in accelerating the weight updating process when the gradient is small (Mohan and Keshaven, 1998). However, Fu and Poch (1995) concluded that with a large momentum coefficient there will be a faster convergent speed during the initial part of training, but finally, divergence will happen. The function *trainlm* uses Levenberg-Marquardt optimisation to make training time shorter (Tholudur and Ramirez, 1996). Levenberg-Marquardt optimisation is a more sophisticated training method, its only limitation is that *trainlm* requires a great deal of memory for large problems. BP network is used in perhaps 80 to 90 % of practical applications (Demuth and Beale, 1994). Figure 2.2 shows a typical network topology.

Table 2.3 - Processes modelled and/or controlled using ANNs

<table>
<tr><th>Pub.</th><th>Process</th><th>Usage</th><th>Input(s)</th><th>Output(s)/Objective(s)</th></tr>
<tr><td rowspan="2">Baba *et al.*, 1990</td><td>Water and sewage treatment plant</td><td>Controller</td><td>Water quality and floc image</td><td>Coagulant injection rate for good water quality</td></tr>
<tr><td colspan="4">**Obs.:** 2 BP algorithms (3 layers, 30-10-1) one for normal conditions and another for abnormal conditions. Good simulation results.</td></tr>
<tr><td rowspan="2">Gagne and Blaise, 1997</td><td>Prediction and classification of trout toxicity</td><td>Model</td><td>Microbiotests</td><td>Predict acute toxicity to trout</td></tr>
<tr><td colspan="4">**Obs.:** Tested 2 types: SOM and BP. SOM identified relevant 5 classes for predicting toxicity. BP analysis yielded two kinds of networks: the 1st one was able to predict the actual toxic concentration with an overall performance of 65%, while the 2nd one, which was designed to differentiate between toxic and non-toxic effluents, exhibited a much better performance (90%).</td></tr>
<tr><td rowspan="2">Linko *et al.*, 1998</td><td>Enzyme production processes</td><td>Model (software sensor)</td><td>On-line measured data</td><td>Prediction of consumed sugar, biomass, and/or enzyme activity</td></tr>
<tr><td colspan="4">**Obs.:** BP algorithm with momentum, 1 hidden layer with 7 neurones. Data was set to 0-1 range. Used hyperbolic tangent transfer function. 2000 epochs for training of the network. The predictions were reliably carried out as far as 10 hours ahead.</td></tr>
<tr><td rowspan="2">Meszaros, *et al.*, 1997</td><td>Continuous flow bioreactor with baker's yeast culture</td><td>Model and Inverse Model (controller)</td><td>DO</td><td>Manipulating air flowrate to maintain desired conc. of biomass and DO</td></tr>
<tr><td colspan="4">**Obs.:** BP algorithm for training. Simulation work. 200 input/output pairs training data. Good results.</td></tr>
<tr><td rowspan="2">Häck and Köhne, 1996</td><td>WWTP (population equivalent: 12,000)</td><td>Models</td><td>e.g. pH value, ORP, conductivity, etc.</td><td>Estimation of process parameters, which are temporarily not available (e.g. ammonium conc.)</td></tr>
<tr><td colspan="4">**Obs.:** BP algorithm was used with sigmoid transfer function for the hidden neurones and linear for the output layer. Estimations were reliable and could be used for process control.</td></tr>
<tr><td rowspan="2">Gagnon *et al.*, 1997</td><td>Water treatment plant</td><td>Models</td><td>e.g. temp., pH, turbidity</td><td>Predict coagulant dosage</td></tr>
<tr><td colspan="4">**Obs.:** 3-layer BP algorithm. Bias in the input and hidden layer. 4 neurones in the hidden layer by trial and error. Data included 340 000 elements (i.e. learning, testing and validation data).</td></tr>
<tr><td rowspan="2">Acuna *et al.*, 1998</td><td>Fermentation process</td><td>Models</td><td>Lactic acid or glucose conc., pH and temperature</td><td>Estimate biomass conc. in lactic acid fermentations</td></tr>
<tr><td colspan="4">**Obs.:** 4 static and 1 dynamic model (one step ahead prediction). 3-layer FFNs with a sigmoidal non-linear activation function (for hidden and output neurones). Input data normalised (0–1). The dynamic model, performed just as well as the static models but offered more stable responses.</td></tr>
<tr><td rowspan="2">Basheer *et al.*, 1996</td><td>Adsorption column in a water treatment process</td><td>Model</td><td>e.g. influent conc., GAC depth, etc.</td><td>Predict the breakthrough time of adsorption column</td></tr>
<tr><td colspan="4">**Obs.:** 3 layer BP network, momentum (0.45), learning rate (0.9). The number of hidden neurones was determined by trial and error. Number of hidden neurones from 1 to 7. Best results were achieved with 2 hidden neurones, the ANN could explain about 80% of the variability in the data.</td></tr>
<tr><td rowspan="2">Syu and Chen, 1998</td><td>Chemical oxidation – Fenton's method and coagulation</td><td>On-line trained controller</td><td>Influent COD, H_2O_2 flowrate, effluent COD and the set point of COD</td><td>Control chemical addition (H_2O_2 and ferrous chloride) to reach the required COD <100 ppm</td></tr>
<tr><td colspan="4">**Obs.:** BP algorithm with momentum term. Time-delayed type with a structure of 7-4-1 (Bias in the 1st and 2nd layers). 15 sets of training data (moving window technique). Good results were obtained.</td></tr>
<tr><td rowspan="2">Chong, 1999</td><td>Stoker Fired Boiler Plant</td><td>3 ARX MISO Models</td><td>Rotary valve, grate speed, air flows, O_2 in flue gas</td><td>Predictions of O_2, NO_x and CO in the flue gas</td></tr>
<tr><td colspan="4">**Obs.:** FFN MLP type with an ARX structure. The inputs varied with the model from 14 to a maximum of 18. Bias for the hidden and output layers. 10 hidden neurones. Some of the models were pruned. The resultant models were able to represent the dynamics of the process and delivered accurate one-step ahead predictions (30 seconds) over a wide range of unseen data.</td></tr>
</table>

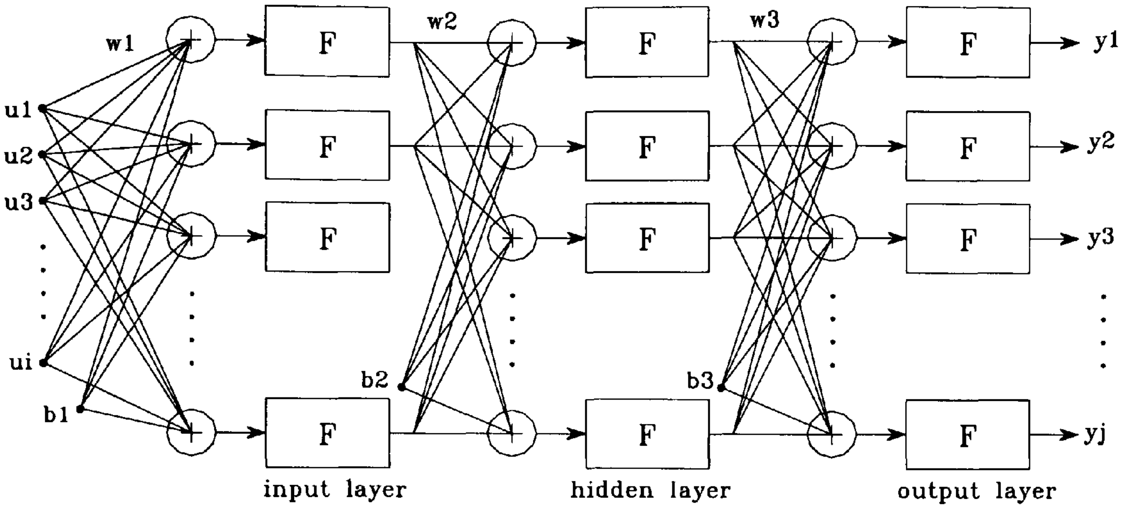


Figure 2.2 - Diagram of a 3-layer FF MLP Network (from Demuth and Beale, 1994)

Where u and y are the input and output vectors of the network architecture, F is the transfer function of the neurones, w and b are the weight and bias matrix for the three layers (denoted by 1, 2 and 3).

Radial Basis Function Network (RBFN)

Local networks, such as RBFN, are particularly suitable for applications where on-line control and optimisation is the main goal (Eikens and Karim, 1994), however, they are poor at extrapolating (Karim *et al.*, 1997). In the RBFN, it is necessary to locate centres among the input/output vectors such that the SSE of the distance from the centre to the training data set is minimised (Karim *et al.*, 1997). RBFN is quite popular and as the MPL is also an universal approximator (Hunt *et al.*, 1992). The activation function in each unit of a RBFN uses a distance measure (Euclidian distance) as an argument, instead of the inner product of the input vector and the weight vector of that node for the MLP (Haykin, 1994). The RBFN is a supervised training algorithm and has two layers, namely the hidden layer (with a non-linear activation function with a parameter termed centre), and the linear output layer. The function used to design the RBFN creates one hidden neurone at a time and these are added to the network until the SSE falls beneath an error goal or the maximum number of neurones has been reached (Demuth and Beale, 1994).

RBFNs may require more neurones than BP networks, but often they can be designed in a fraction of the time. They work best when many training vectors are available. Biases in this case are used to adjust the sensitivity of the neurone. The only design decision for RBFN (besides picking an error goal) is finding a good value for the spread constant. This constant determines how wide the radial basis function (RBF) is and which is normally of gaussian form (Montague and Morris, 1994). It is important that the RBF of the hidden layer overlap so as to allow good generalisation. However, they should not be so spread out such that the radial basis neurones return outputs near 1 for all the input vectors used in design. Ideally, the spread constant should be much larger than the minimum distance and much smaller than the maximum distance between input vectors (Demuth and Beale, 1994). Since RBFN have temporal information built into their formulation, they generally possess better 'generalisation' capabilities for time variant processes (Karim and Rivera, 1992). Thompson and Kramer (1994) modelled chemical processes using prior knowledge and a RBFN.

Elman Network

In RNs, the outputs of some neurones are fed back to the same neurones or to neurones in preceding layers, thus allowing signals to propagate in opposite directions. RNs are able to learn both the non-linear characteristics and the long-term dynamics of the plant although the measured data vector only includes a short time horizon in each step (Harremoes *et al.*, 1993). These networks are preferred both in identification and adaptive control of dynamic non-linear systems (Raychaudhuri *et al.*, 1996; Zhao *et al.*, 1999). In contrast to the standard FFN, a RN can be much smaller in size and use fewer parameters (weights). Only a single vector of process variables at time t is used to predict the process variables at time $t + 1$. This type of ANN can be further classified into partially or fully recurrent. In partially RNs, the main network structure is FF whereas in fully RNs there can be arbitrary FF and feedback connection. The feedback connections are formed through a set of 'context' units and are not trainable. Examples of RNs include the Hopfield network (Hopfield, 1984) and the Elman network (Elman, 1990).

An Elman network is a partially RN which consists of a two-layer structure (Elman, 1990) (Figure 2.3). It differs from conventional two-layer FFNs in that in addition to an ordinary hidden layer, there is another special hidden layer usually called the 'context' or state layer. This layer receives feedback signals from the ordinary hidden layer and the outputs of the neurones in the 'context' layer are fed forward to the ordinary hidden layer. The 'context' units are used only to memorise previous activations of the hidden units and can be considered to function as one-step time delays (Pham and Liu, 1995). The hidden (recurrent) layer contains tan-sigmoid transfer function neurones and linear neurones in its output layer and with this combination it can approximate any function (with a finite number of discontinuities) with arbitrary accuracy, as long as there are enough hidden neurones (Demuth and Beale, 1994).

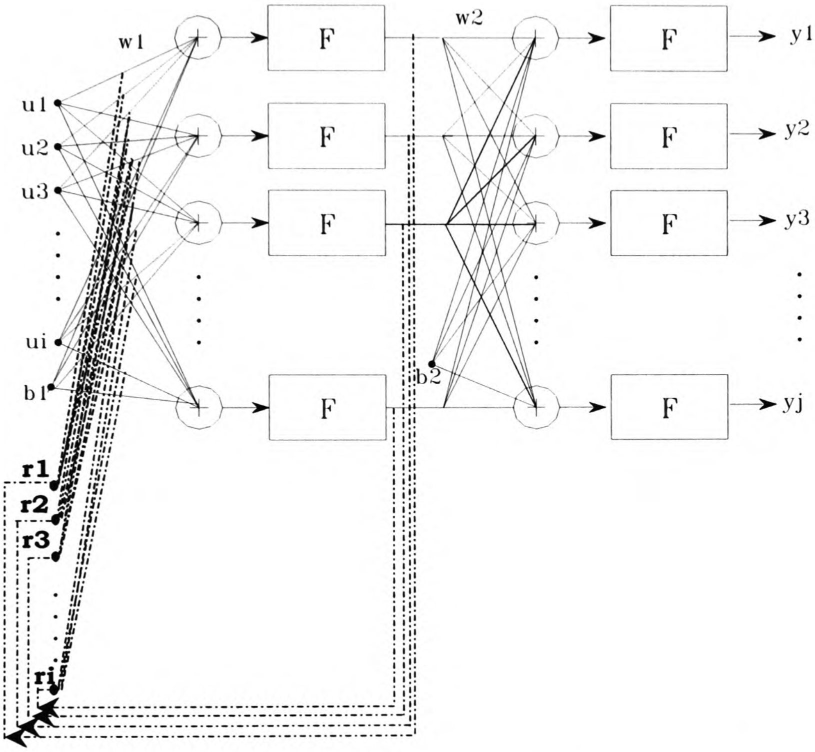


Figure 2.3 - Elman network (from Demuth and Beale, 1994)

Where the r (1 to i) are the 'context' units.

Self-Organising Map (SOM)

Unlike the MLP (in which multiple output neurones can be activated simultaneously), 'competitive' neurones compete with each other for activation. As a result, only a single output neurone in the competitive layer is active at any particular instant – 'winner-take-all' learning (Zurada, 1992). Competitive learning is often employed to cluster input data, where similar patterns are collectively grouped together based on data correlation and represented by a single winning neurone. During this process, only the weight vectors associated with the winning neurone are updated. Such learning is unsupervised, and the learning procedure is following summarised (Jain *et al.*, 1996):

1. Initialise weights to small random values and set the initial learning rate;
2. Present input vectors and evaluate the network outputs;
3. Select the neurones, which weight best match the input vectors;
4. Update the weights of the winning neurone;
5. Decrease the learning rate by a fractional amount;
6. Repeat steps 2 to 5 until the change in weight values is less than a specified threshold or a maximum number of iterations is reached.

The competitive neurones are ordered physically in one or more dimensions (usually 2 dimensional grid will be more than enough). Each neurone has neighbours. One of the limitations of competitive networks is that some neurones may not always get allocated i.e. some neurone weight vectors may start out far from any input vectors and never win the competition, no matter how long the training is continued. The result is that their weights do not get to learn and they never win. These unfortunate neurones, referred to as 'dead neurones', never perform a useful function. To stop this from happening, biases are used to give neurones, which are only winning the competition rarely an advantage over neurones, which are winning often. The biases force each neurone to classify roughly the same % of input vectors. Kohonen (1989) presented a competitive network, which produced what he called the SOM. It learns to recognise groups of similar input vectors in such a way that neurones next to each other in the network learn to respond to similar vectors. The SOM has been used in areas such as pattern recognition and robotics (Kohonen *et al.*, 1996), image processing (Sabourine and Mitiche, 1993) and economy issues (Martin-del-Brio and Serrano-Sinca, 1993). Because SOM can output values of 0, 0.5 and 1, the instar rule must be used instead of the Kohonen rule, which assumes only 1/0 values. Neurones next to each other in the network learn to respond to similar vectors. The layer of neurones can be imagined to be a rubber net, which is stretched over the regions in the input space where input vectors occur. As the vector moves from the original winning neurone's classification region, its value drops to 0.5, and one of the neighbouring neurone's outputs increases to 1 (Demuth and Beale, 1994).

Learning Vector Quantization (LVQ)

The most popular form of competitive learning is LVQ employed for data compression, speech and image processing (Jain *et al.*, 1996). The structure of the LVQ network bears close resemblance to the standard MLP network and consists of two layers. The first layer is a competitive layer, which learns to classify input vectors as described above where training is performed in an unsupervised mode. The second layer transforms the competitive layer's classes into target classifications defined by the user in binary form 0/1 (Demuth and Beale, 1994). Therefore, the LVQ is a supervised learning network and its structure is shown in Figure 2.4. A rule of thumb is to use more competitive neurones than the possible input patterns to enable the competitive layer to create sub-classes for the desired target through the use of the linear layer. Cooper *et al.* (1992) compared a LVQ network with a BP network as a pattern analysis tool and found that both networks were equally capable with the LVQ's ease of training and implicit ability to assess the accuracy of the pattern match as deciding factors in network selection.

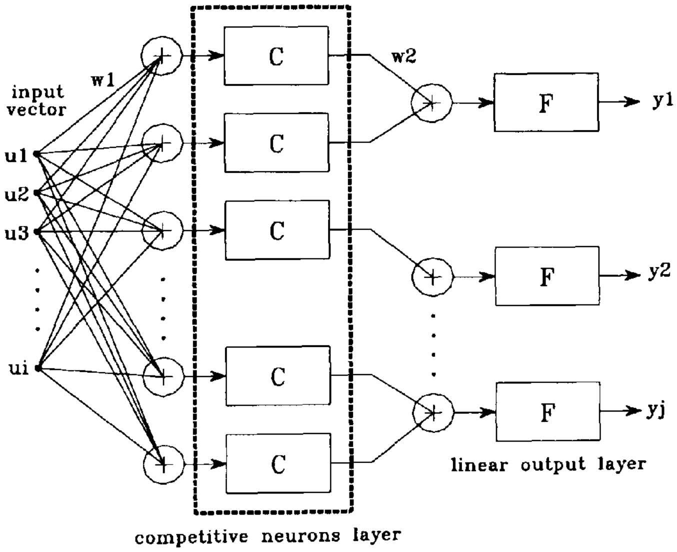


Figure 2.4 - LVQ Network Structure (from Demuth and Beale, 1994)

Where C denotes the competitive neurones.

2.11.2. ANNs for Modelling and Control of Anaerobic Treatment Systems

The large volume of literature directed to the control of anaerobic reactors evidences the difficulty in establishing mathematical models based on the underlying biochemical processes in an anaerobic reactor i.e. with non-linear and time varying characteritics. There is some evidence in the literature that ANNs would be ideal for use in WWT systems due to their ability to learn and generalise. The use of ANNs appears to be a great value within the area of process identification and control (Emmanouilides and Petrou, 1997). Table 2.4 presents a few examples of studies where ANN alone or in conjunction with other techniques have been applied for the modelling and/or control of anaerobic treatment systems.

Premier *et al.* (1999) compared the ability of 'black-box' models of ARX structure to represent a fluidised bed reactor. The models were a linear single-input single-output (SISO) model, a linear MIMO model and a non-linear ANN model (MLP architecture with a single hidden layer of non-linear activation function neurones and a linear output layer - Nørgaard, 1995). The performance of the models were compared using correlation analysis of the residuals (one-step ahead prediction errors – 48 h) and it was found that the SISO model was the least able to predict the changes in the reactor parameters (BA, gas production rate and %CO_2). They also reported that the MIMO and ANN models both performed reasonably well.

Table 2.4 – Applications of ANNs alone or in conjunction with other techniques for modelling and/or control of anaerobic treatment systems

<table>
<tr><th>Pub.</th><th>Process</th><th>Usage</th><th>Input(s)</th><th>Output(s)/Objective(s)</th></tr>
<tr><td rowspan="2">Guwy <i>et al.</i>, 1997b</td><td>Laboratory scale fluidised bed reactor operated on a simulated baker's yeast WW</td><td>Controller</td><td>On-line BA</td><td>Control of $NaHCO_3$ dosing pump for digester buffering during organic overloads</td></tr>
<tr><td colspan="4">Obs: The on-off controller with a set point at the steady-state level (2700 mg $CaCO_3$ l^{-1}) maintained BA conc., but resulted in levels above the upper set point. The ANN controller (BP algorithm with a structure of 8-8-1 – Wilcox <i>et al.</i> 1995) was configured to take consecutive values from a moving window containing the last 8 BA data points trained on BA data from an anaerobic filter operating on ice-cream processing WW (BA ~ 1400 mg $CaCO_3$ l^{-1}). No re-training was performed. Despite the different steady-state BA levels and reactor type, the controller was capable of maintaining stable BA levels during overload without the overshoot observed in the operation of the on-off controller. Control of BA during organic overloads did not prevent changes in gaseous CO_2, and H_2 and gas flow rate.</td></tr>
<tr><td rowspan="2">Fell and Wheatley, 1995</td><td>Anaerobic digester for coffee processing effluent</td><td>Controller</td><td>CH_4, COD and volume of biogas production</td><td>Control influent flowrate to adjust organic and toxic loading for constant effluent COD and gas production with a constant quality</td></tr>
<tr><td colspan="4">Obs: 3-layer FFN (BP algorithm). The controller performed as well as an adaptive control system, whilst required less expert input, accepted more parameters and had a faster response. Little data was required for training.</td></tr>
<tr><td rowspan="2">Tay and Zhang, 2000</td><td>3 Laboratory scale high-rate digesters (including an UASB reactor)</td><td>Model</td><td><i>Liq. Phase</i>: pH, total and specific VFAs, BA, COD (or TOC), COD (TOC or VSS) reduction, ORP; <i>gas phase:</i> gas flowrate, CH_4, CO_2, H_2, CO</td><td>Predict anaerobic systems response 1 h in advance</td></tr>
<tr><td colspan="4">Obs: In all three cases, the model learned well and exhibited good and fast predictions when the reactors were subjected to hydraulic and organic overloads and variations in alkalinity loading rates. The model was expected to have a great application potential in RTC.</td></tr>
<tr><td rowspan="2">Emmanouilides and Petrou 1997</td><td>Simulated anaerobic digester using a mathematical model</td><td>Model followed by a controller</td><td>Data revealing the operation of the digester</td><td>Manipulate the influent dilution rate to control the total substrate conc. and the CH_4 production rate</td></tr>
<tr><td colspan="4">Obs: Model: On-line trained to provide one-step ahead predictions (6 h) of the plant response. 48,000 initial training pairs, normalised (0.1-0.9 for outputs and –1-1 for inputs). BP algorithm allowed adjustments of the learning rate and momentum in order to improve convergence. By trial and error a network with 2 hidden layers with 6 hidden neurones each appeared to be a good compromise between predictive accuracy and network size. A moving-time-zone fixed-length training data set was employed for on-line neural model training. A maximum allowable number of 200 iterations was imposed for training due to time constraints. Controller: Utilised model predictions to minimise a cost function. On-line trained using BP, chemotaxis and a random search algorithm, with a maximum number of 500 iterations. The last two techniques appeared to be superior then the BP both in terms of speed of convergence and computational simplicity.</td></tr>
<tr><td rowspan="2">Steyer <i>et al.</i>, 1997</td><td>Anaerobic digestion fluidised bed reactor for treatment of wine distillery</td><td>Hybrid Models</td><td>pH, temperature, recirculation flow rate, input flow rate and gas flow rate</td><td>Detection and analysis of problems (e.g. foam forming, sudden changes in the influent, or bed temp. changes)</td></tr>
<tr><td colspan="4">Obs: The process variables were pre-processed using fuzzy logic to build a vector of features i.e. process states. The ANN (BP algorithm with 2 hidden layers with 4 and 2 neurones, respectively) was used to classify the process states and to identify the faulty or dangerous ones. Trained with the Levenberg-Marquardt method. The hybrid approach was seen to be a complement to control systems.</td></tr>
</table>

2.11.3. ANNs for Modelling and Control of Aerobic Treatment Systems

Control of the activated sludge process has historically been more art than science. Several process parameters are routinely calculated at many plants (e.g. organic loading and sludge age). However, the relationship between these parameters and process performance in full-scale plants is not well understood and many factors that affect the process are not even measured. Although a number of deterministic models exist to describe the activated sludge process, their usefulness is primarily for research purposes or for design - not for control (Vaccari and Christodoulatos, 1992). This because the models are capable of predicting removal of substrate in the feed, but much of the effluent contamination is from biomass that escapes the sedimentation stage as SS and no accurate deterministic models exist for that as it depends on the physical processes in the sedimentation stage as well as on the biological properties of the biomass (Vaccari and Christodoulatos, 1992). A lot of effort has been devoted to the modelling of the activated sludge process since the early 1970s (Lessard and Beck, 1991). However, even though many models have been proposed to simulate the dynamic behaviour of both the biological reactor and the secondary settler, very few studies looked at the interactions between these two units (Dupont and Henze, 1992). Moreover, very few models have been validated with real field data (Côté *et al.*, 1995). Deterministic models require a large number of analytical parameters and are, at large, linear space parameters and rates are invariant over long periods of time (Hamoda *et al.*, 1999).

Watanabe *et al.* (1993) proposed an intelligent operation support system for bulking prediction and control of the activated sludge process. It integrated a physical model, image processing, knowledge engineering including fuzzy theory and an ANN. A successful application utilising the combination of an ANN and the ASM1 was reported by Zhao *et al.* (1997). Cohen *et al.* (1997) applied a neuro-fuzzy process model for on-line control of a sequencing batch reactor. Output data generated by the model was used to assist control its cycle duration, sludge wasting, and temporary storage of excessive load in a lagoon. A simplified hybrid neural net approach was applied by Miller *et al.* (1997) for the modelling and subsequent analysis of a WWTP in Mizushima, Japan, with success (combination of an ANN model, principal component analysis and simple physical or semi empirical relationships supported by the available data). The objective was to reduce the occurrences of overflow in the clarifier caused by filamentous bulking. Table 2.5 summarise some studies where ANNs alone or in conjunction with other techniques have been applied for modelling and/or control of aerobic treatment systems.

Table 2.5 - Applications of ANNs alone or in conjunction with other techniques to model and/or control aerobic treatment systems

Pub.	Process	Usage	Input(s)	Output(s)/Objective(s)
Zhu *et al.* 1998	Aerated stabilisation basins for WWT	Prediction model	10 variables were used	Prediction of effluent BOD
	Obs.: 3-layer partially RN. The model was updated based on a moving window technique, which made an improvement in the prediction accuracy. More accurate when compared with the standard MLP model.			
Tyagi *et al.*, 1993	Activated sludge process	2 Models for control (M1 + M2)	**M1**: Substrate concentrations and flow rate (simulated data). **M2**: recycle ratio and clarifier overflow rate	**M1**: Predict recycle ratio and WAS flow rate.; **M2**: Predict MLSS and recycle SS
	Obs.: **M1**: Standard 3-layer FFN (BP algorithm, 2 hidden neurones with sigmoidal function). **M2**: Same structure but 4 hidden neurones. ANN models performed well in terms of accuracy and prediction capability, and better than the traditional regression method.			
Fu and Poch, 1995	Activated sludge process	Model		Estimate the effluent COD
	Obs.: 4-layer FFN trained using BP algorithm (5-15-8-1), with generalised sigmoid transfer function for all neurones. Several simulations were investigated in order to select suitable learning rates and momentum coefficients. The performance of the one-step prediction (2 h) was the best.			
Du *et al.*, 1996	Activated sludge process	2 MISO Models for analysis	Conc. of active heterotrophic biomass, active autotrophic biomass, readily biodegradable substrate, NH_3 nitrogen and DO in bulk liquid phase.	Relate the diffusional effects in terms of effectiveness factors, for the processes of carbon oxidation and nitrification in relation to the operating parameters
	Obs.: A FFN was developed by employing the BP learning algorithm 3-layer network (18 hidden neurones), sigmoidal function was applied in all layers' neurones. Normalised data (0-1). 1000 training sets of data. 10 sets of validation data and the prediction accuracy was found generally to be over 90%. The ANN greatly improved the predictive performance in relation to a multiple linear regression model.			
Capodaglio *et al.*, 1991	Activated sludge processes	Model	BOD:N; N:P; DO; Temp.; and F:M ratio	Prediction of SVI value
	Obs.: BP algorithm, 25 inputs and 1 output. Each input pattern fed to the system consisted of 5 values (due to the sludge age of the system) representing the previous days 1-5 for each of the input parameters previously described. The output consisted of the one-day ahead prediction of SVI value. 90 % of learning accuracy was verified.			
Too and Chen, 1997	Activated sludge process	Controller	Difference between substrate conc. in the effluent and its set point (error); difference between the current and previous errors	Regulate the reflux ratio of an activated sludge aerator
	Obs.: Trained on-line with a moving-window learning rule (10 samples). 3-layer topology (the best was 2-5-1). Learning rate of 0.01 was the best by trial and error. Tangential activation function. The controller was able to bring the system back to the set point regardless if the system encountered a disturbance due to a sudden change of the feed concentration.			

Table 2.5 - Applications of ANNs alone or in conjunction with other techniques to model and/or control aerobic treatment systems (Cont.)

Pub.	Process	Usage	Input(s)	Output(s)/Objective(s)
Côté *et al.*, 1995	Activated sludge process	5 MISO models (M1-M5)	Common inputs: RAS and overflow consolidation tank. In addition: M1) SS (*t*); M2) SS(*t*) and COD(*t*); M3 & M4) $NH_4(t\text{-}3)$ and COD(*t*-3); M5) COD(*t*) and COD(*t*-1)	Predict at (*t*): M1) Effluent SS; M2) Effluent COD; M3) Effluent ammonia; M4) VSS in the RAS; M5) DO. To improve accuracy of a mechanistic model
	Obs.: 3-layer FFN, sigmoid transfer function was used. The coupling of a mechanistic model with the ANN error predictor yielded significant improvement in the simulation of all variables for which a linear function of the influent flow was clearly inadequate.			
Chang *et al.*, 1998	Activated sludge process	Neurogenetic model		Predict effluent SS from an enhanced biological phosphorus removal system
	Obs.: Genetic search through ANN architecture space (input variables and network architectures) to select the optimal-performing ANNs and error BP learning in individual networks to evaluate the selected architectures. The neurogenetic model predicted accurately the effluent SS, according to experimental work.			
Wen and Vassiliadis, 1998	Activated sludge process (primary settler, aeration tank and clarifier)	Modelling + control with an hybrid AI system (ES + 2 ANNs)	See obs.	Control the RAS to remove pollutants in the water for discharge, to reduce energy consumption, and to improve the efficiency of the treatment
	Obs.: The hybrid AI system converted the data collected from the simulation (effluent's BOD, and SS as well as microorganisms) using a mathematical model The ES monitored the BOD and MLSS values in the aeration tank, and changed the RAS until the level of the BOD or MLSS was acceptable. ANN1 predicted the future condition of the aeration tank, and ANN2 arrived at a suitable RAS. The inputs of ANN1 were the current condition of the aeration tank, the inflow and the recycle. ANN2 inputs were the desired value for the BOD or the MLSS and the present and past inflow conditions. The hybrid system proved to be a powerful tool for the control of WWT processes, which were poorly understood or difficult to model with conventional control methods.			

2.12. Use of ANNs for Process Fault Detection and Tolerance

Fault diagnosis problems have been studied very actively during recent years. The literature on failure detection and diagnosis is extensive and various approaches have been proposed. These include simple limit checking, parameter estimation, analytical redundancy, and AI techniques (Liu, 1999). In many applications sensors are reliable but in WWT sensor failure can be a regular occurrence, which makes continuous long-term measurement rather difficult and so the control system must be robust enough to continue operating in such circumstances. ANNs have been successfully used for sensor data analysis (e.g. Piovoso and Owens, 1991) and fault detection (Iordache *et al.*, 1991; Ungar *et al.*, 1990). On-line fault detection and diagnosis are particularly desirable. Fault detection via a state space approach is difficult because the technique assumes that the process model is known quite well. ANNs possess an inherent non-linear structure suitable for mapping complex characteristics, learning, and optimisation, which have recently been shown to have considerable potential for fault detection or diagnosis of the process control systems (Liu, 1999). One added advantage of ANNs is that sometimes classification can be accomplished more rapidly than classification using statistical packages (Hoskins *et al.*, 1991). Therefore, these authors concluded that, given a valid database, fault detection and diagnosis is a promising area for the application of ANNs. Ungar *et al.* (1990) described adaptive ANNs for fault diagnosis and process control. Connection strengths representing correlations between inputs (alarms and sensor measurements) and outputs (faults, future sensor measurements or control actions) were learnt using a BP algorithm. Venkatasubramanian (1990) observed that the recall and single-fault generalisation performances of the ANNs (BP algorithm) were very good, when they were trained with data covering as much of the fault space as possible and more hidden neurones were needed for two-fault than one-fault generalisation performance. Bulsari *et al.* (1991) investigated the use of a state vector estimator based on delayed measurements of the control and output vectors and FFNs (Levenberg-Marquardt method) to detect sensor faults in biochemical processes (a bias was added to the neurones). Sensor fault detection was achieved by examination of the model residuals. Liu (1999) published a fault diagnosis strategy for a glutamic acid fermentation based on an extended Kalman filter and an ANN classifier (BP algorithm). The author demonstrated the suitability of the strategy (i.e. accuracy and speed during real-time implementation). However, the author found considerable improvements using an unsupervised trained ANN classifier.

2.13. Important Points Stated in the Literature

1. Textile industry utilises high quantities of water and produce highly polluting effluents - ***Highly stated.***
2. Cotton fibre died with reactive dyes - ***Highly used.***
3. Colour removal from textile WWs - ***Difficult task.***
4. Sequential anaerobic-aerobic treatment of textile WWs – ***Effective.***
5. Biotreatment processes have non-linear dynamics and time varying characteristics - ***Highly stated.***
6. Lack of reliable sensors and fast remedial actions for biotreatment processes - ***Present reality.***

7. The importance given by researchers to certain monitoring parameters for biotreatment process operation, modelling and control - ***Highly variable*** *(depends on the effluent and type of reactor used and the modelling and control algorithms utilised).*

8. 'Conventional' modelling and control of biotreatment processes – ***Difficult task.***

9. ANNs used for modelling and control of biotreatment processes - ***Highly recommended.***

10. ANN applications for sensor failure detection and data analysis - ***Used with success.***

3. APPARATUS AND PROCEDURES

This Chapter describes the anaerobic and aerobic stages and also the auxiliary equipment used in this work, the methodology adopted for off-line analyses and on-line instrumentation. It includes a discussion of the equipment setup and operation for best performance, and experimental design to test the monitoring and control strategies.

3.1. Laboratory Biological Treatment Stages and Operation

Data from a previous project (Guwy *et al.*, 1997a) was used to train and test the use of different types of ANNs for a control scheme. The reactor setup, on-line instrumentation and influent to the laboratory WW treatment system have also been previously described in the above work. Therefore, this will not be analysed here, instead a small summary of the experimental design and monitoring system will be presented in Section 3.6.1. The next two Sections will describe the two biological stages, the respective auxiliary equipment and the STE utilised.

3.1.1. Anaerobic and Aerobic Stages

An UASB reactor, an aerobic tank and an aerobic settling vessel were used to carry out the experiments described in this thesis. All vessels, made up of perspex, were tested for water leaks. The UASB reactor was checked also for gas leaks by filling it with water (i.e. up to the working volume) and sealing all the ports except two of them in the lid. Nitrogen gas was pumped into the top of the reactor through one of the gas sampling ports at the top of the reactor at a low pressure to produce a steady stream of bubbles through the Drexel bottle (this attached to the main gas port). A soap solution was placed around all ports and joints so that any escape of gas resulted in the production of bubbles. All peristaltic pumps used in this series of experiments were from Watson Marlow (Cornwall, UK) being of the type 505U, 503S and 303A (all controllable pumps were of the 505U type). Silicone rubber tubing was used for the transfer of liquids, except in the pump heads. However, for Experimental Phases 3, 4 and 5 the silicone rubber tubing was replaced by polyurethane tubing for the flow to the BA, TOC, TOD and colour analysers. Marprene double MC manifold tubing was used with Watson Marlow 505U and 503S 8-roller multi-channel head pumps. This tubing was used for pumping the STE concentrate, HCl and the OECD waste, for dilution of the biogas for the H_2 monitor and dilution of the sample to the colour analyser, and also for all the pumped substances controlled by the ANNBCS. Watson Marlow marprene tubing was used with all other pump heads. The influent to the UASB reactor and the aerobic stage is defined in Sections 3.1.2 and 3.6. The liquids were pumped at appropriate rates into a common feed line and mixing occurred in-line. Water for dilution of the STE was stored in a 200 l covered tank. T-joints were built into the two main lines (i.e. for the UASB reactor and to the aerobic stage) from where samples of influent could be extracted for off and on-line analyses.

Anaerobic granules were obtained from BPB Paperboard Davidson Mill, Aberdeen. This is a paper pulp processing plant, and therefore the granules were not adapted to degradation of dyes. Activated sludge was collected from Coslech, a local sewage treatment plant. This plant also treated industrial waste from a local L'Oreal factory and therefore the sludge tended to foam greatly after collection. This was counteracted by the use of antifoam. The reactor and vessels used here are discussed in more detail below.

UASB reactor

An UASB reactor of 30 l working volume was used to treat the STE based on the following advantages. Lettinga *et al.* (1980) initiated the development of the first full-scale installation of an UASB reactor. It represents 67 % of the total number of digesters worldwide (Barber and Stuckey, 1999), which suggests its significant commercial advantages (Laguna *et al.*, 1999). There were seven UASB reactors in UK industrial sites (e.g. Davidsons Papermill, Aberdeen; Coca Cola, Wakefield; Everest Potato Foods, Kidderminster) (Wheatley *et al.*, 1997). A distinctive feature of successful UASB reactor operations is the very high loading rate achieved by the systems (Morgan *et al.*, 1990). No artificial mixing is required as biogas circulation and the up flow velocity of feed are sufficient (Hickey *et al.*, 1991). UASB reactors are generally used for WWs that have a low SS concentration (Angenent and Dague, 1995). Granular sludge has a number of advantages over flocculent biomass: it is better retained within the reactor due to its superior settleability (Archer, 1983); it has higher specific methanogenic activity (Speece, 1996), which is maintained during less favourable conditions due to the higher internal pH of the granules (Angenent and Dague, 1995). Dense granules in the UASB reactors, with their high settling velocity, avoid the costly packing/carrier, which is otherwise necessary in other configurations to provide similar conditions (Laguna *et al.*, 1999). Speece (1996) also noted that the UASB reactor shows a strong capability to absorb shock loadings. However, there are also a few disadvantages, the bed can be disrupted if the flow in is too fast or if the gas production is too vigorous (Hawkes and Hawkes, 1994) and at low organic loading rates the contents of the UASB reactors are not adequately mixed (Speece, 1996). At extremely high loading rates a significant fraction of biomass will become dispersed in the liquid above the biomass bed because of high turbulence (caused by high gas evolution in the bed), which causes biomass aggregates to float, due to adherent or entrapped gas bubbles (Speece, 1996). Arcand *et al.* (1994) used scanning electron microscopy (SEM) for granules examination and observed a clear predominance of fermentative bacteria in the external layer of the granules, acetoclastic activities were evenly distributed along the granule depth, the core was almost exclusively composed of methanogens. The same researchers used an intragranular kinetic model and predicted pH values, 1 mm inside the granule, to be over one pH unit more alkaline than in the bulk liquid, which gives a competitive advantage for methanogens to proliferate in the granule core. Based on atomic spectrophotometry, Wu (1991) found that the major cations in granules were the Na, K, Ca, Mg, Fe, P and S. In UASB reactors changes of pH, nutrient balance or the presence of toxic compounds in the substrate could damage the granules structure and consequently the process efficiency (Gonzalez *et al.*, 1998).

Being a high-rate reactor it made possible to draw suitable quantities of samples for off and on-line analyses and also be able to feed the aerobic stage and cope with the sampling. A 1 day HRT was chosen to operate the UASB reactor throughout the Experiments. A schematic of the UASB reactor is shown in Figure 3.1. The UASB reactor's built-in water jacket was attached to a Grant FH15 thermostatically controlled flow heater (Grant Instruments, Cambridge, UK), which pumped heated water through the tubing in order to maintain an internal temperature of 35 °C. Water coming from the reactor was displaced into a ballast bottle and was released above the surface of the water in the bottle. Water was removed from underneath the surface to replace it. This arrangement eliminated air bubbles, which would cause the flow heater to fail if they gathered, and compensated for the evaporation of water over time. Feed was introduced via two ports each leading to a cross-shaped outlet at the base of the reactor. This ensured more even distribution of the STE over the base of the reactor and meant that there was less likelihood of a serious blockage occurring at this point. The base and upper unit containing the 3-phase separator were removable and were sealed using o-rings and silicone grease. The separator consisted of 2 'V' shaped perspex pieces, which extended across approximately two-thirds of the width of the reactor. The lid of the UASB reactor housed an on-line pH meter. A temperature probe was installed through one of the side top ports of the UASB reactor. Various on-line monitors (as presented in Sections 3.3 and 3.6) were connected to the other ports. Samples for off-line analysis were taken of the STE from a T-joint before it entered the UASB reactor and from the recycle port.

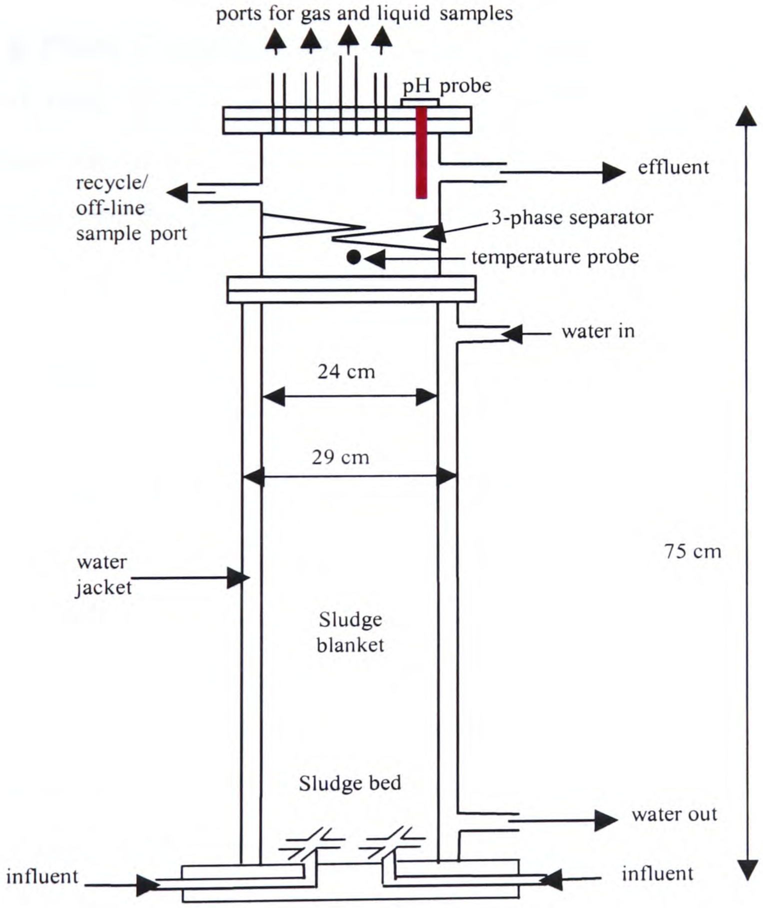


Figure 3.1– UASB reactor

Aerobic tank

The effluent from the UASB reactor was collected in an Erlenmeyer flask, which acted as a mixing vessel. A tube, reaching the bottom of the mixing vessel, was attached to a pump. This removed any liquid collecting in the vessel and displaced it to the aerobic vessel. When the aerobic stage was not in use, anaerobic effluent was diverted to drain by switching off the transfer pump thus causing it to overflow through the arm of the Erlenmeyer flask which was attached to a drainage system. During Experimental Phase 5 the STE was fed straight into the aerobic tank without previous treatment by the UASB reactor.

The aerobic tank was a cylindrical vessel (41 cm in height and 30 cm in width) resting on a square base of 36 cm length. It had a water jacket, which was not used during this project, as there were no extremes of temperature in the laboratory. The liquid level in the aerobic tank was controlled by altering the depth of a tube within the reactor, which was attached to a Watson Marlow pump. A vertical depth controller (RS Components, UK) was also used to maintain a depth of 20 l through control of the aerobic tank influent pump. The HRT was 16 h except during Phase 5 where it was 17 hours. The lid of the tank had 5 ports which served for input of feed, OECD waste and HCl, off-line sampling, pH probe, DO probe, output of sludge and return from the settler and from Filter 3 in the case of Experimental Phase 5. At the bottom of the tank there was a port where two silicone tubes were inserted connecting the two Capex L2C air compressors (Charles Austen Pumps, Lancs., UK) with 7 l min^{-1} of capacity (one constantly aerating and another on stand-by) to two air stones glued at the bottom of the tank.

Local controls - On-off set-point controllers were built in LabVIEW™, which sampled the pH and DO levels in the aerobic tank and controlled the addition of 1 M HCl and the stand-by air compressor. The pH controller was operational during Experimental Phases 2, 3 and 5 and the DO controller only during Experimental Phases 3 and 5. During some of the Experiments of Phase 5 pH and DO were controlled by the ANNBCS. Both actuators worked with a voltage input of 0 – 5 V. The set point levels were for addition of acid at pH above 7.2 and to pump extra air at a DO level below 3 mg l^{-1}. pH control was necessary as the pH within the activated sludge tank would rise to about 8 – 9 pH units and at these levels most of the protozoa die (Harold Buckland, Yorkshire Water, *pers. comm.).* This rise in pH was due to the large quantity of NaOH used to hydrolyse the starch (Section 3.1.2). The anaerobic digestion process was maintained at a pH of 7 due to the production of VFAs, however, under aerobic conditions the VFAs volatilised or were metabolised, resulting in a rise in pH.

Aerobic settling vessel

The aerobic settler dimensions were 36 cm height and 20 cm wide in the top of the funnel. The settler working volume was 3.75 l (3 h HRT except for Phase 5 where it was 3.2 h). A slow stirrer (1 revolution per minute - RPM) was fitted in the settler to assist on the settling of solids. Liquid was pumped from the aerobic tank to the settler through the influent port at the top of the vessel. Effluent was displaced through the effluent port, which went to drain. During Experimental Phase 2 the solids were recycled every four hours with the aid of a RS 139-710 7 day timer (RS, Northants, UK) linked to a pump for controlling the RAS. In Experimental Phases 3 and 5 solids were recycled continually. Sludge could be disposed of to waste using the effluent port of the vessel rather than recycled from the bottom of the vessel when required.

3.1.2. Influent to the anaerobic and aerobic stages

In this project a mixed cotton production effluent was simulated as cotton dyeing effluent and contained already the starch to provide the carbon required for reduction of the azo bonds in the UASB reactor. The work presented was focused on reactive dyes as they present the greatest environmental problem for three reasons, discussed previously in Section 2.1: they represent an increasing market share because they are used to dye cotton; their low fixation rate and therefore higher dye loss in the effluent; and conventional WWTPs, which rely on sorption and aerobic biodegradation, have a low removal efficiency for reactive dyes, which leads to coloured waterways, and public complaints.

Due to the variability in textile WW composition, no artificial waste can be truly representative, either of a particular type of waste or even of a particular factory. O'Neill *et al.* (1999a) cited that many researchers have used STEs in the investigation of treatment technologies. This is useful as it enables research to be carried out in the absence of a local source of effluent and simulated effluents have constant composition and therefore enable the effects of treatment to be more readily understood. These should resemble real wastes as closely as possible, however it is difficult to replicate the ADMI values, absorbance and spectra of real effluents. The concentrations of dye used in simulated effluents examined in literature varied from 0.01 g l^{-1} to 7 g l^{-1} (O'Neill *et al.*, 1999a). The same authors stated that dye concentrations of 0.01 g l^{-1} up to 0.25 g l^{-1} have been cited as being present in dyehouse effluent, depending on the dyes and processes used while Steenken-Richter and Kermer (1992) reported that up to 0.8 g l^{-1} of hydrolysed dye could remain in the bath after completion of the reactive dyeing process.

The composition of the STE used in this project was based on some of the principal components of real effluent and their contribution to effluent COD and BOD. Components such as surfactants, oil and grease contained in real wastes were not included. Therefore, the STE contained only the most common pollutants, such as dye, size and salt. Reactive dyes do not adhere to glass or perspex, making them ideal for laboratory use. Only one reactive dye was chosen, except in Experiments 3.8 and 3.9, so that the processes occurring within the reactors would not be complicated by the different characteristics and the wide range of breakdown products that would be produced by the use of a mixture of dyes. The potential anaerobic breakdown products of PROCION Red H-E7B the dye used were identified elsewhere (Carliell *et al.,* 1995). This dye has a double azo bond and it has a low toxicity with a 36 h lethal concentration (50 %) to rainbow trout exceeding 100 mg l^{-1}. It is said to be unlikely to inhibit aerobic bacteria and the manufacturer reported no evidence of inhibition to anaerobic treatment at 25 g l^{-1} (BASF, Manchester, UK). Approximately 75 % of sizes used in textile industry are starch-based (Weber and Ströhle, 1997). The starch most commonly used in sizing are corn, maize and potato starches (Allied Colloids, UK, *pers. comm.*). Tissalys 150 (Roquette UK, Turnbridge Wells, UK) is a common potato starch widely used in the cotton industry.

A colleague in the laboratory prepared the recipe of the STE, except for Phase 4, and also of the OECD waste. The STE differed during the experiments, due to the concentrations of acetic acid (0.53 – 5.03 g l^{-1}), starch (0.95 - 3.8 g l^{-1}) and dye (0.075 – 10 g l^{-1}). These concentrations will be found in Section 3.6. The starch used was Tissalys 150 (Roquette, Kent, UK) and the dye used was a reactive azo dye PROCION Red H-E7B (CI Reactive Red 141) (BASF, Manchester, UK). Three other dyes were also used in Experiments 3.8 and 3.9

(Section 3.6.3). Both starch and dye in the STE were hydrolysed in order to convert them into the form in which they are normally found in a 'typical' cotton effluent.

Two methods of starch hydrolysis were adopted. The first method was based on information obtained from Instituto Superior Técnico (IST, Lisbon, Portugal), who based it on desizing methods obtained from Hoechst (1991). The starch was hydrolysed by mixing a stock solution containing 100 g l^{-1} of starch and 40 g l^{-1} of NaOH and leaving at room temperature overnight. The second method of starch hydrolysis was performed by adding an enzyme, Amylase 10L (Biocatalysts Ltd., Pontypridd, UK) which was used as recommended by the supplier. A stock of 100 g l^{-1} starch was mixed with 0.2 ml l^{-1} of amylase and was maintained at 80 °C for 1 hour. A few drops of HCl were then added to the solution to bring the pH below 3 to stop further reaction. The pH of the samples was then adjusted to 5.9 using NaOH. The dye was hydrolysed using a method recommended by IST who derived it from information obtained from Dystar (Portugal). A 50 g l^{-1} solution of dye was hydrolysed by addition of NaOH to pH 12 and maintained at approximately 80 °C for 1.5 hours.

Speece (1996) reported that the N concentration should be maintained between 40-70 mg l^{-1} to prevent reduction in biomass activity. Alphenaar *et al.* (1993) found that P deficiency reduces methanogenic activity in UASB reactors to 50 %, however, this reduction was found to be reversible by dosage of phosphate. Overdosage of phosphate was found by the researchers to be unprofitable. It is normally added to the WW to reach concentrations of 2 - 50 mg l^{-1}. Forster (1991) cited a recommended COD:N:P of <350:5:1 for good operation of anaerobic systems. Iron, cobalt, nickel, and sulphide have been shown to be obligatory trace elements for methanogens to convert acetate to CH_4 (Speece *et al.*, 1986). Molybdenum, tungsten, and selenium have also been reported as required trace metals (cited by Speece, 1983). Also, metals such as Zn, Cu, Cd, Cr(vi) and Cr(iii) are required in biological processes in minute quantities (Schroder and de Haast, 1988). Archer (1983) stated that sodium, potassium and magnesium ions are required by methanogenic bacteria, but very high levels of the cations inhibit methanogenic fermentation (Kugelman and Chin, 1971). Therefore, the feed also contained 0.15 g l^{-1} NaCl, 0.23 g l^{-1} NH_4Cl, trace elements (H_2SO_4, EDTA.Na, $FeSO_4.7H_2O$, $ZnSO_4.7H_2O$, $MnCl_2.4H_2O$, $CuSO_4.5H_2O$, $Co(NO_3)_2.6H_2O$, $NaB_4O_7.10H_2O$ and $NiCl_2.6H_2O$ – refer to O'Neill *et al.* (1999b) for quantities) and nutrients ($(NH_4)_2SO_4$, 0.28 g l^{-1}; $Na_3PO_4.12H_2O$, 0.123 g l^{-1}; $Na_2HPO_4.12H_2O$, 0.096 g l^{-1}).

The STE was made in the form of concentrate and was stored in a refrigerator at 6 °C. It was diluted with water containing $NaHCO_3$, which was added in order to maintain the buffering capacity of the UASB reactor. An addition of 2.5 g $NaHCO_3$ l^{-1} was performed in the STE to the UASB reactor, except during Experiment 3.5 and Experiment 4.3 (Sections 3.6.3, 3.6.4 and 4.3.1), as the natural BA of the STE was 95 mg $CaCO_3$ l^{-1} (standard deviation - *sd* = 5, n = 5) indicating that little natural buffering capacity was present. During Experimental Phase 5 only 0.5 g $NaHCO_3$ l^{-1} was added to the STE, which was fed straight to the aerobic stage. OECD waste was fed to the aerobic stage during Experiments 3.7, 3.8 and 3.9 and throughout Experimental Phase 5. This contained: 4.8 g l^{-1} of peptone, 3.3 g l^{-1} meat extract, 0.9 g l^{-1} urea, 0.21 g l^{-1} sodium chloride, 0.12 g l^{-1} calcium chloride dihydrate, 0.06 g l^{-1} magnesium sulphate heptahydrate, and 0.84 g l^{-1} potassium hydrogen phosphate (OECD, 1981). The OECD waste simulated the situation where sewage was fed to the activated sludge stage in an attempt to mimic a textile effluent treatment plant where almost one third of the COD to the aerobic stage comes from domestic sewage and also to help increase the MLSS. The OECD waste was made up as concentrate and stored refrigerated for a maximum of 7 days (6 °C) and was mixed with the UASB reactor effluent before entering the aerobic system.

The STE contained sulphur and sodium, both of which can cause toxicity in anaerobic systems. Archer (1983) referred that production of H_2S reduces CH_4 yields but does not affect COD reduction too much. Sulphide concentrations of 1 – 25 mg l^{-1} have been cited as optimal for methanogens metabolism (Parkin et al., 1990). Concentrations of 2 - 4 g l^{-1} sulphate have been found to be inhibitory to anaerobic processes (O'Flaherty and Colleran, 1999) and 0.1 - 0.8 mg l^{-1} dissolved sulphide has been reported to be toxic to methanogenesis. Toxic H_2S concentrations are in the range of 40 - 430 mg l^{-1} as S (Parkin *et al.*, 1990) with 1 % H_2S corresponding to 26 mg l^{-1} H_2S or 52 mg l^{-1} total sulphide in the liquid phase (pH 6.9, 35°C) (Speece, 1996). The total sulphur concentration in the STE (i.e. from nutrients and dye) was 0.08 – 0.18 g l^{-1}. Some toxicity from the sulphur concentration may have occurred with the highest concentration of dye (i.e. Experiments 3.2, 3.4, 3.9, 4.2 and 4.4). Concentrations of 3.5 - 5.5 g l^{-1} Na have been cited as causing moderate inhibition of methanogenesis with strong inhibition occurring at 8 g l^{-1} (Carliell *et al.*, 1996). Methanogens require small concentrations of sodium with a reported optimum of 0.23 - 0.35 g l^{-1} for organisms grown in a low salinity medium and higher for organisms grown in high salinity medium (Feijoo *et al.*, 1995). Concentrations of 3 - 16 g l^{-1} caused 50 % inhibition of methanisation of VFA mixtures in 3 anaerobic sludges in the absence of nutrients or other salts (Feijoo *et al.*, 1995). No sludge adaptation to the sodium inhibition was observed upon 12 weeks exposure (Rinzema *et al.*, 1988). The highest sodium addition was 2.4 g l^{-1} below the value reported for moderate toxicity.

3.2. Off-line analyses

Off-line samples were collected and preserved according to the guidelines specified in the Standard Methods (APHA, 1989). Analyses were performed immediately except for the measurement of colour and VFAs where sometimes the samples were stored at 4 °C in a domestic refrigerator for short periods or frozen at –20 °C in case of prepared samples for VFA analysis.

3.2.1. pH analysis

The pH was determined by electrometric measurement as described in the Standard Methods (APHA, 1989). A Mettler Toledo 340 pH meter (Switzerland) with a glass electrode was used. The pH meter was calibrated daily before use with standard buffer solutions of pH 4.0 and pH 9.2 (Fisons Ltd., Loughborough, UK). A 25 ml sample was agitated by the use of a magnetic stirrer to establish equilibrium between sample and electrodes. The analysis was performed within one minute so that minimum interference occurred from the loss of dissolved CO_2.

3.2.2. Bicarbonate alkalinity (titration to a pH of 5.75)

In this work BA was measured by titration with standard 0.05 M HCl to pH 5.75 as proposed by Jenkins *et al.* (1983). Titration to this end point reflected mainly alkalinity due to the presence of bicarbonate. At pH 5.75, 80 % of the bicarbonate will have been titrated and less than 20 % of the VFAs will have contributed to the alkalinity measured by this point

(Jenkins *et al.*, 1983). Even if the VFAs have a concentration of 500 mg $CaCO_3$ l^{-1}, and the total BA is 2000 mg l^{-1}, less than 5 % error is introduced (Jenkins *et al.*, 1983). Therefore, even at high concentrations of VFAs an accurate estimate of BA can be obtained. The equation used to calculate BA includes a compensation factor to account for the contribution of VFAs to the measurement. Titration of 25 ml aliquots was carried out at room temperature using the same pH meter and calibrated in the same way as in Section 3.2.1 after the initial pH had been noted. Samples were stirred with a magnetic stirrer at 75 RPM while additions were made to ensure good mixing. This procedure was done as quickly as possible in order to minimise the loss of the dissolved CO_2. The 'true bicarbonate alkalinity' (TBA) was then calculated using the following equation:

$$TBA_{5.75}\,(\text{mg } CaCO_3\ l^{-1}) = 1.25 \times ALK_{5.75}$$

$$\left(\text{being, } ALK_{5.75} = \frac{A \times M \times 50000}{\text{ml sample}}\right)$$

Where A = ml of standard acid used; M = molarity of the acid (i.e. 0.05 M); 1.25 = factor given by Jenkins *et al.* (1983) to compensate for alkalinity due to the VFAs; 50000 = to express the answer in terms of mg $CaCO_3$ l^{-1}.

3.2.3. Off-line colour analysis

The spectra of samples were determined off-line using an ATI Unicam UV1-UV/Visible Spectrophotometer (ATI Unicam Ltd., Cambridge, UK) with an optical glass visible cell of 10 mm path length. Samples for true colour measurement were centrifuged at approximately (3000 RPM) for 5 minutes to ensure all particulate material had settled. The linear operating range was 0 - 2.9 absorbance units, therefore some samples had to be diluted with deionised (DI) water. The baseline was zeroed using DI water in both the reference and the sample cell. Samples were scanned in the visible spectrum using a 2 nm bandwidth using a tungsten halogen lamp for the measurements. The true colour of the samples was determined by calculating the average of three measured optical densities (ODs) at 436, 525 and 620 nm on the centrifuged sample at a pH of 7.1 - 7.3 in a similar way to BS 6068 (1995). The spectrum was scanned for the sample and the absorbance at the required wavelengths read by means of tracking. At an absorbance of 1 the manufacturer reported an error of 0.5 %.

3.2.4. Gas chromatography

Gas chromatography was carried out to determine VFA concentrations in the reactors' liquid content and the percentage of CH_4 and CO_2 in gaseous samples. In a GC, samples were injected as either gases or liquids that are consequently vapourised. The gases then move through the column at different rates and were detected as they emerge (APHA, 1989).

VFA analysis

VFAs were determined by a GC fitted with a FID after extracting the VFA with diethyl ether from acidified samples as described by Peck *et al.* (1986). VFA samples were measured on a Varian Star 3400 CX analyser with a 6 feet x 4 mm packed glass column of 15 % SP1220/1 % H_3PO_4 on 100/120 Chromosorb W/AW (Supelco, Poole, UK) as the support phase. The analyser's FID was connected to a GC Star Workstation as described by Varian Associates Inc. (1993). The system setup and method were those recommended by the manufacturers. The sample for measurement was taken from the recycle port of the UASB reactor when the recycle pump had stopped for at least 10 minutes. Three 5 ml samples of effluent and standard were placed in 13 x 120 mm glass tubes with screw-on solvent-resistant lids (Fisons Ltd., Loughborough, UK). The tubes were then placed in a fume cupboard and 0.75 ml of orthophosphoric acid (BDH Ltd., Poole, UK) was added. This was followed by an addition of 5 ml of a diethyl ether (Fisher Scientific Ltd., Loughborough, UK) solution containing 0.1 ml l^{-1} of 4-methyl-n-valeric acid as an internal standard. The lids were placed on the tubes and then inverted them 10 times to mix the solutions. They were then left for three minutes before being centrifuged at 4000 RPM for a few seconds so that two liquid layers were formed within the tube. The top layer of sample was removed with a pipette and placed in a small glass screw-top vial (Alltech Associates, Lancs., UK). The vials lid had a hole in the centre and was freshly lined with red Teflon/silicone liners (BDH Ltd., Poole, UK) for each analysis with the white side uppermost. This seal was necessary for pressurised filling of the injection system. The standards and samples were run in the manner recommended by the manufacturers, the nitrogen carrier gas was maintained at a flow rate of 30 ml min^{-1}, the air and hydrogen gases at a rate of 300 ml min^{-1} and 25 ml min^{-1}, respectively to the FID. Samples were analysed by an auto-sampler attached to a Varian Star 3400CX analyser. Using the peak area the Workstation converted the results into concentrations of VFAs. After use, the vials were cleaned, by heating them in a furnace at 500±50 °C for about 30 minutes to remove all VFAs. The calibration was verified using a standard every run and re-calibration was carried out as necessary. VFAs can adsorb to the column and therefore after injection of samples at least one wash containing 10 % formic acid in diethyl ether was injected to clean the column. The standard error for this system has been cited as less than 2 % for each VFA (Peck *et al.*, 1986).

Biogas composition analysis (CO_2 and CH_4)

Gas chromatography was also carried out to determine the quantity of CO_2 and CH_4 in the biogas produced by the UASB reactor. A Varian Star 3400 CX GC (Varian Ltd., Walton-upon-Thames, UK) was used with a 2 feet stainless steel column packed with Porapak Q80-100 (Supelco Ltd., Poole, UK) and fitted with a TCD. This system can detect a minimum gas concentration of 5 mg l^{-1}. The Varian Star was linked to a PC, which was used to control the analysis (Varian Associates Inc., 1993). Chromatography grade helium (MG Gas Products, Cardiff) at a flowrate of 30 ml min^{-1} was used as the carrier gas. A single point calibration was carried out prior to use, using a standard gas with a CO_2:CH_4 ratio of 40:60 (BOC Gases, Guildford, UK). A three-way valve syringe was used with 10 ml of standard and flushed twice before the manual injection to ensure a representative sample. If the calibration was satisfactory i.e. $sd < 5$ %, gas samples from the UASB reactor were then analysed. The sample port for gas samples on the UASB reactor was located on the main gas line leading from the UASB reactor and was located prior to the Drexel bottle. The workstation calculated the percentage of CO_2 and CH_4 in the sample from the peak area.

3.2.5. Determination of biogas H_2S concentration

The concentration of H_2S in the biogas was determined by collection of approximately 300 ml of gas in a gas bag and using 0.1 – 0.4 % H_2S Kitagawa precision gas detector tubes (Alltech Associates, Lancs., UK) in conjunction with a Matheson Toxic Gas Detector Model 8014KA (Alltech Associates, Lancs., UK).

3.2.6. COD determination

For this work, the closed reflux titrimetric method was chosen for the COD analysis and adopted according to Standard Methods (HMSO, 1986). Prior to analysis, each sample was left to settle for 1 h and then was diluted with DI water as this method can only accurately measure up to 250 mg COD l^{-1}. 2.5 ml of the diluted sample was pipetted into a Pyrex culture tube for Teflon lined screw caps (BDH Ltd., Poole, UK), followed by the addition of 5 ml of Ficodox Plus standard COD reagent (Fisher Scientific Ltd., Loughborough, UK). This reagent contained silver nitrate, chromium III potassium sulphate solution and concentrated sulphuric acid. The tube was then sealed and inverted several times to mix completely, before it was placed into a Driblock DB-4 heating block (Techne Ltd., Cambridge, UK) preheated to 150±3 °C. After 2 hours the tubes were removed and cooled to room temperature. The content of the tube was emptied into a 250 ml conical flask and it was filled and emptied three times with DI water, placing the water into the flask. Two drops of ferroin indicator solution (1, 10-phenanthroline ferrous sulphate (0.025 M)) were added and titrated against 0.0125 M FAS solution to the endpoint where the colour changes from blue green to orange. This operation was performed whilst constantly mixing the contents of the flask. The blank that contained 5 ml of the COD reagent and 2.5 ml of DI water was refluxed and titrated in the same manner. Each sample was analysed in triplicate. From the titration, the COD was calculated using the following equation:

$$\text{COD mg l}^{-1} = \left(\frac{(ml\ FAS\ (blank\ titre) - ml\ FAS\ (sample\ titre)) \times FAS\ molarity \times 8000}{2.5\ ml\ of\ sample} \right) \times dilution\ factor$$

$$\text{Where FAS molarity} = \frac{\text{ml dichromate solution titrated} \times 0.25}{\text{ml FAS used in titration}}$$

Equipment was kept solely for COD measurement to avoid contamination problems. The FAS solution was standardised by titration in triplicate against a solution containing ferroin indicator, 2.5 ml of 0.0418 M (0.25 N) potassium dichromate, 25 ml of DI water and 7.5 ml of concentrated H_2SO_4. Standard deviations of up to 5.6 % have been cited for the closed reflux method (APHA, 1995) although HMSO (1986) cited standard deviations of up to

9.01 % for industrial effluent samples. Guwy (1995) found precision between duplicate samples to be ±100 mg COD l^{-1} for effluent from an anaerobic digesters operating on ice-cream waste. For example, the *sd* was found to be 58 mg COD l^{-1} (n = 12) in triplicate samples of the UASB reactor taken from Experiment 3.1. The mean effluent COD during Experiment 3.1 after 3 HRT was 810 mg l^{-1} (*sd* = 96, n = 4). Therefore, a 7.2 % mean error was found.

3.2.7. Total solids (TS) and Volatile Solids (VS) for the UASB reactor

The methods used were the ones recommended by the APHA (1995) for measuring TS and VS of the UASB reactor biomass and a colleague at the laboratory performed them. TS were determined by drying, to constant weight, a known volume of sample at 105 ± 2 °C. The precision of TS measurements has been cited as ±5 % (APHA, 1989). The level of VS was determined by incinerating at 500±50 °C to constant weight a sample which had undergone TS analysis (APHA, 1989). An error of 6.5 % has been found for VS (APHA, 1995). TS and VS measurements were made in triplicate. For these procedures the following equipment was used: volumetric cylinders, 100 ml glass beakers, 'Eurotherm' electric furnace (Carbolite Ltd., Sheffield), desiccator, 4 decimal point balance, Gallenkamp Hotbox convection over (Grant Instruments Ltd., Cambridge).

3.2.8. Total Suspended Solids (TSS) and Volatile Suspended Solids (VSS) for the aerated stage

APHA (1995) recommended the methods used for measuring TSS and VSS of aerobic suspended biomass. The TSS measurements for aerobic biomass are referred to as MLSS. TSS is the sample component, which cannot pass through a Whatman G/FC filter paper in this case with a 70 μm thickness and then dried at 105 ± 2 °C. Standard deviations of 0.76 – 33 % have been found in samples containing 15 – 1707 mg l^{-1} TSS, increasing with decreasing sample size, and the *sd* of 7.2 % has been found for VSS (APHA, 1995). The sample which had undergone TSS analysis was incinerated at 500 ± 50 °C to constant weight (APHA, 1995) to measure VSS. Both measurements were made in triplicate. The VSS measurement was used to determine how much of the solids were bacterial, as inorganic solids are not volatile at 550 °C. The *sd* for MLSS and VSS between 12 replicates during Experiment 5.1 was 0.073 g l^{-1} and 0.074 g l^{-1}, respectively. This corresponded to a mean error of 3 % for MLSS and 3.4 % for VSS. For these procedures all the equipment presented in Section 3.2.7 plus the following was used: Whatman G/FC filter paper, Hoffman filter unit to hold the paper filter, Buchner flask and a water vacuum pump (Charles Austen Pumps, Lancs., UK).

3.2.9. Biomass Catalase Activity

Measurements of catalase activity as a measure of active aerobic biomass were made intermittently using an assay method (Guwy *et al.*, 1998). These authors concluded that the technique had potential to measure active aerobic biomass in activated sludge plants on-line without prior treatment over at least the range of MLSS tested (0.3 – 4.5 g TSS l^{-1}). The authors tested it on dilute samples and obtained a linear result according to the dilution factor. The measurement could not be performed continuously as there was not enough biomass to be taken out from the aerobic stage. The apparatus consisted of a perspex reaction chamber with a liquid working volume of 60 ml, mixed by a magnetic stirrer bar. The monitor used a solution of 0.485 M H_2O_2 prepared from a 30 % solution (Fisher Scientific Ltd., Loughborough, UK) in 0.1 M sodium dihydrogen orthophosphate buffer at pH 7.0 as reagent. Both solutions were prepared daily with ultra pure DI water and were stored in a dark bottle. The stirred activated sludge samples and the peroxide reagent were introduced into the reaction chamber using a Watson Marlow 505 U peristaltic pump with an 8-roller multi-channel pump head each at a rate of 10 ml min^{-1}. Each run used up about 150 ml of sample and the same quantity of the reagent mixture. The activated sludge samples from the aerobic tank were run through the prototype monitor as soon as they were collected. Dilution of the sample, although possible, was never required. When the sample was expected to foam antifoam was added before the run. During each run the sample was pumped out of the volumetric flask where it was being continuously stirred to the reaction chamber where was also stirred in conjunction with the reagent. The temperature of the cabinet containing the reaction chamber was set to ± 37 °C. The reaction chamber had a gas outlet, which was connected to a LFM 300 gas meter (G.H. Zeal, London, UK). The gas meter was connected to a portable computer where date/time and volume of O_2 produced were logged for about a 40 minute period. A measurement was recorded every 15 seconds. The volume of oxygen evolved was averaged from the steady state period over 15 minutes. During the run there was a need for constant supervision as the tubes for pumping the sample and the reagent were prone to blockage. A constant drip from the chamber had to exist throughout the experimental run. Figure 3.2 shows a photograph and a schematic of the prototype biomass activity monitor. During the experimental measurements the gas meter shown in the photograph was replaced by the commercial low flow gas meter mentioned above and due to its slightly larger dimensions was located outside the cabinet.

The specific catalase activity (SCA) of the samples was related to the VSS of samples taken at the same time. It is known that, one unit of catalase activity corresponds to the breakdown of 1 μmol of H_2O_2 per minute under specified conditions, which would produce a gas flow of 11.2 μl of O_2 per minute at STP (Guwy *et al.*, 1998). The following equations were used to calculate the SCA related to g of VSS.

$$\text{Catalase activity (catalase units l}^{-1}\text{ sample)} = \frac{1000 \times \text{ml min}^{-1}\ \text{O}_2\text{ evolved}}{11.2} \times \frac{1000}{\text{ml sample}}$$

$$\text{Therefore: SCA (catalase units g}^{-1}\text{ VSS)} = \frac{\text{Catalase activity (catalase units l}^{-1}\text{ sample)}}{\text{g VSS l}^{-1}\text{sample}}$$

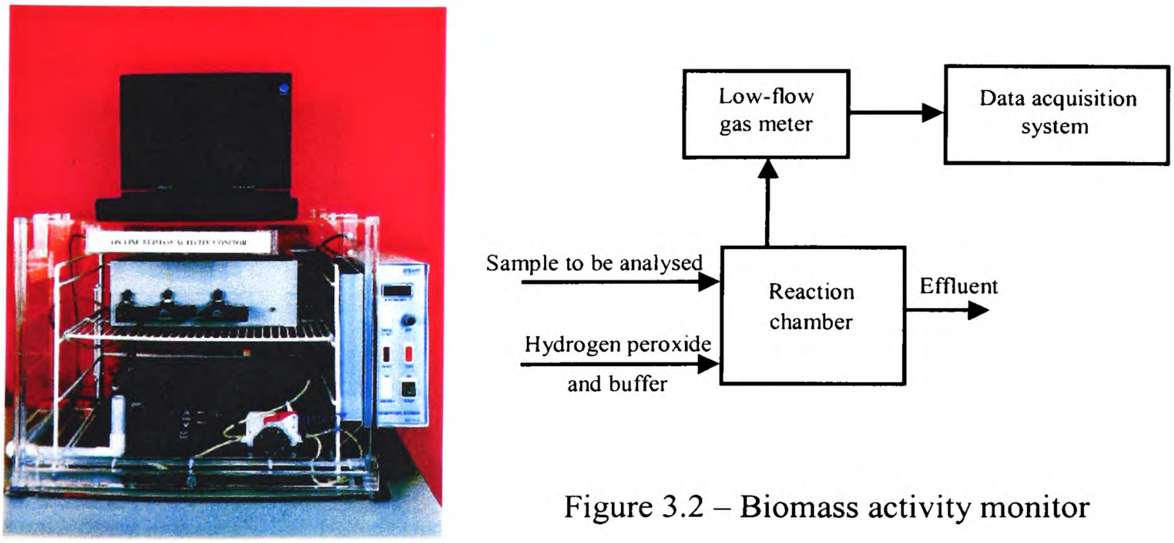


Figure 3.2 – Biomass activity monitor

3.3. On-line Instruments

On-line instruments were used for the monitoring and development of the control scheme for the treatment process and to test the application of the TOD and TOC monitors and the UV/Visible Spectrophotometer on-line with this type of textile effluent. This section will briefly describe the operation, calibration, requirements, settings and data acquisition utilised by the on-line monitoring instruments.

3.3.1. Biogas Related Measurements

The operation of on-line monitors such as low flow gas meters and H_2 and CO_2 monitors will be presented in the following three sections.

Gas meters

Two low flow gas meters were used to measure the gas flowrate produced by the UASB reactor. One being a prototype and the other a commercial instrument LFM 300 (G.H. Zeal, London, UK) (Guwy *et al.*, 1995) (Figure A.9, Appendix A). The first one monitored the gas flowrate for Experimental Phase 2 and the second for Experimental Phases 3 and 4. These gas meters were designed to measure low flow rates (<5 ml min^{-1}) and operate continuously (Guwy *et al.*, 1995). They were not affected by irregular gas production, and did not cause sudden large changes in pressure, which could have displaced liquid from the reactors. The maximum flowrate for the prototype instrument was variable as it had 4 different size ballast chambers to choose from. For the commercial meter the maximum flowrate was 22.2 ml min^{-1}.

Data acquisition and calibration - Both instruments output a voltage of 0 – 10 V from the gas meter to the data logger built in a LabVIEW™ virtual instrument (VI). The prototype gas meter was calibrated using a SAGA 400 bubble flow meter (Ion Science Ltd., UK) at different flow rates. The gain and bias were therefore calculated from the relationship between the bubble meter readings and the on-line readings. The bubble flow meter was attached to a supply of nitrogen gas at a specific flowrate. 10 readings were taken to get a reliable mean. The nitrogen gas was then attached to the gas meter with gain set to 1 and bias set to 0 on the VI. Once the readings obtained in LabVIEW™ were steady the values were noted. This procedure was repeated with other flowrates. The mean of each point was then plotted against the reading required and the equation resulting from a linear regression analysis was then used to introduce the new values of gain and bias in the VI. The manufacturer calibrated the commercial gas meter yearly and the only calibration needed between the instrument and the data logging system was due to the small variance caused by electrical noise. Therefore the gain and the bias in LabVIEW™ were adjusted using the relationship between the gas meter displayed readings and the ones in the data logging system.

Hydrogen analyser

The GMI exhaled hydrogen monitor (GMI Ltd., Renfrew, Scotland) was only setup to work on-line on the 5th run of Experiment 3.1 with its linear range up to 900 ppm (no measurements were performed for Experiment 3.2) (Figure A.3, Appendix A). Flow to the H_2 meter (normally of 2.2 ml min^{-1}) was performed by a peristaltic pump (503S Watson Marlow Ltd., Cornwall, UK) attached to the H_2 monitor and was not recycled. To compensate, the gas flow measurements using the gas meter were recorded with the adjustment of this flowrate. Stripping of the H_2S was accomplished with a perspex tube filled with glass beads and a 560 ml saturated solution of copper sulphate (Fisher Scientific Ltd., Loughborough, UK) and resulted in a 15 minutes lag. After the 1st run of Experiment 3.4 (Section 3.6.3) there was a 2-fold dilution of the biogas as it overloaded. This dilution was achieved with air pumped through a 505U Watson Marlow pump making a total flow to the analyser of 4.4 ml min^{-1}. Collins and Paskins (1987) reported monitor cross-sensitivity to oxygen producing a low reading of approximately 1 ppm per 1 % of oxygen present, in this work this did not occur. Also, Kidby and Nedwell (1991) showed this error to be approximately half that reported by Collins and Paskins (1987), and stated that it may be a function of each polarographic cell.

Calibration and data acquisition - For calibration purposes a CH_4:CO_2 (60:40) standard mixture (BOC Gases, Guilford, UK) and a 96 ppm hydrogen in air standard (GMI Ltd., Renfrew, Scotland) were pumped continuously at 2.2 ml min^{-1} to the scrubber and then to the H_2 monitor. Collins and Paskins (1987) showed that the instrument gave no response to the CH_4 and CO_2 mixture and therefore was suitable for use in anaerobic digestion. The H_2

monitor had a 0 - 100 mA output range. The signal was sampled after passed the current through 3 resistors and then been amplified 10 times (Section 3.5.1 and Figures A.1 and A.4 of Appendix A), which resulted in a voltage signal of 0 - 5 V being sent to the central logging system.

Carbon dioxide analyser

A SB-100 carbon dioxide analyser (ADC Ltd., Hodderson, UK) was used to monitor the percentage CO_2 in the biogas produced by the UASB reactor (Figure A.3, Appendix A). The instrument's response time was dependent on the gas flow through the infrared cell therefore, it was important to maintain a constant gas flow. The biogas was continually recycled in a closed loop through the analyser, which allowed in-situ measurements to be made. The biogas was recycled at a rate of 19 ml min^{-1} from the gas space by a 503S peristaltic pump (Watson Marlow Ltd., Cornwall, UK). Marprene tubing was used throughout the cycle to avoid any permeation of CO_2 to the atmosphere. The analyser output was 0 - 5 V (Figure A.5, Appendix A) and was calibrated monthly using GC grade nitrogen gas (Messer UK Ltd., Reigate, UK) and a standard mixture of CH_4:CO_2 (60:40) (BOC Gases, Guilford, UK). The accuracy of the instrument was checked regularly with the standard mixture and also daily with off-line GC gas analysis (see Section 3.2.4).

3.3.2. On-line pH and DO determination

Both pH and DO measurements were logged via a WP4007 (Solomat Ltd., Herts, UK) and therefore they are both covered in this Section.

pH probe - The pH level of a liquid is defined as the negative logarithm of hydrogen ion activity, which can be detected by using a glass combination electrode (i.e. with a reference electrode). The glass electrode acts as a transducer, converting chemical energy into an electrical signal (measured in mV). The glass combination electrodes used to measure pH 'in situ' in both the UASB reactor and also the aerobic tank were of the type Ingold Xerolyt HA405-DXK-S8 (lengths: 120 mm (UASB reactor) and 225 mm (aerobic tank)) (Mettler-Toledo, Ltd., Halstead, UK). They were used in conjunction with a WP4007 instrument (Solomat Ltd., Herts, UK) (Figure A.9, Appendix A). This instrument was setup for channels and calibration points using the CS6 Software using a serial connection (RS232) (Section 3.5.1). The results were logged in the central data logging system using the 0 - 10 V signal from the MX8000 output multiplexer connected to the WP4007. From this multiplexer 3 outputs were used (i.e. 2 pH probes and 1 DO probe). Calibration of the pH probes was performed every week using the 'single mode' using two freshly prepared standard buffer solutions i.e. pH 7 and 4 (Fisons Ltd., Loughborough, UK). When calibrating the high reference point for pH, the result of the calibration was displayed as a percentage. If the % was within 85 - 100 % the pH probe was in good condition and was calibrated. The probe was replaced if it was less than 85 %. After calibration the WP4007 was set to 'scan mode'.

DO probe – A Capsule 8012170 (ABB Kent-Taylor, Ltd., St. Neots, UK) was used to measure the DO in the aerobic tank. This DO probe with automatic temperature compensation was also integrated with the WP4007 and MX8000 instruments described above. The operating range was 0 - 9.06 mg l^{-1}. The DO probe was also calibrated in 'single mode'. The calibration was performed using an electrolyte solution, supplied by Solomat Ltd., for zero calibration. For the saturation calibration point the probe was immersed in a container with 2 l of water with an air stone connected to an air compressor similar to the ones used in the aerobic tank (7 l min^{-1}). The compressor was left to aerate the water for 2 minutes and then during the calibration the sensor was allowed to stabilise for 3 minutes. If the sensor responded slowly during calibration it was assumed that the membrane was clogged, necessitating cleaning with DI water. Every week there was inspection for any tears, air bubbles or white residue on the cathode. The capsule was replaced as necessary. The oxygen content was set to be displayed in mg l^{-1}. The data acquisition was performed similarly as for the pH probes and after calibration the WP4007 was also set to 'scan mode'.

3.3.3. Intermittent BA analyser

The intermittent BA analyser prototype worked on a similar principle to the continuous BA monitor presented in Guwy *et al.* (1994) and was initially built under another project and it was first tested here (Figure 3.3). This novel monitor presented considerable advantages mainly when operating on a laboratory scale digester. These were: a small sample was required (approximately 50 ml), short analysis time (minimum of 10 minutes, the maximum would depend on how many washes and purges were required), it can be set to sample as often as needed given the extra flexibility for sampling on small throughput reactors. One to two analyses were performed per hour as described in Section 3.6. The monitor measured CO_2 liberated from bicarbonate acidification of the sample to pH 4 or below. This pH was selected to ensure that all bicarbonate was converted to CO_2. The sample chamber volume was 130 ml. Each sample was automatically saturated with CO_2, and approximately 0.5 ml of 2.5 M sulphuric acid was added per measurement. For each mole of bicarbonate decomposed, one mole of CO_2 is formed. Therefore, the CO_2 concentration in the sample after acidification was proportional to the concentration of bicarbonate originally present in the sample. Rinsing between samples with tap water was also automated. Gas production pressure from the sample was measured from 0 bar to a maximum of 1 bar using a very sensitive gauge pressure sensor (manufactured by SenSym SCX15DN and supplied by Farnell Components, UK) which provided a voltage output of 0.087 mV per mbar. The monitor analysed BA in the range of 100 to 2500 mg $CaCO_3$ l^{-1}. The system if stopped was warmed up for 90 minutes prior to analysis. Refer to Section 3.4 for the description of the filtration system for the BA analyser.

The effluent of the UASB reactor was pumped out at 160 ml min^{-1} by a 503S Watson Marlow pump (Cornwall, UK) through a 5 mm tube connected to a course filter in Experimental Phase 2 placed above the plates of the gas disengagement zone. However, this funnel/filter arrangement kept blocking and falling from the tube. Consequently, for Experimental Phases 3 and 4, the sampling was performed with the same tube but placed just underneath the perspex plates i.e. granules clear zone. During the 3 Phases, the effluent was recycled from Filter 2 back to the side-port located above the plates. This need for recycle was to decrease the time that the samples needed to reach the on-line instruments and at the same time not wasting a lot of sample. The large flowrate used was so that not so many granules would be able to deposit in Filter 2 (Figure 3.5).

Calibration - The BA analyser was calibrated using three standard solutions of 500, 1000 and 2000 mg $CaCO_3l^{-1}$ (using sodium hydrogen carbonate (Fisher Scientific Ltd., Loughborough, UK)) every week. Calibration of the monitor showed linearity within 94.6 % (Figure 3.4).

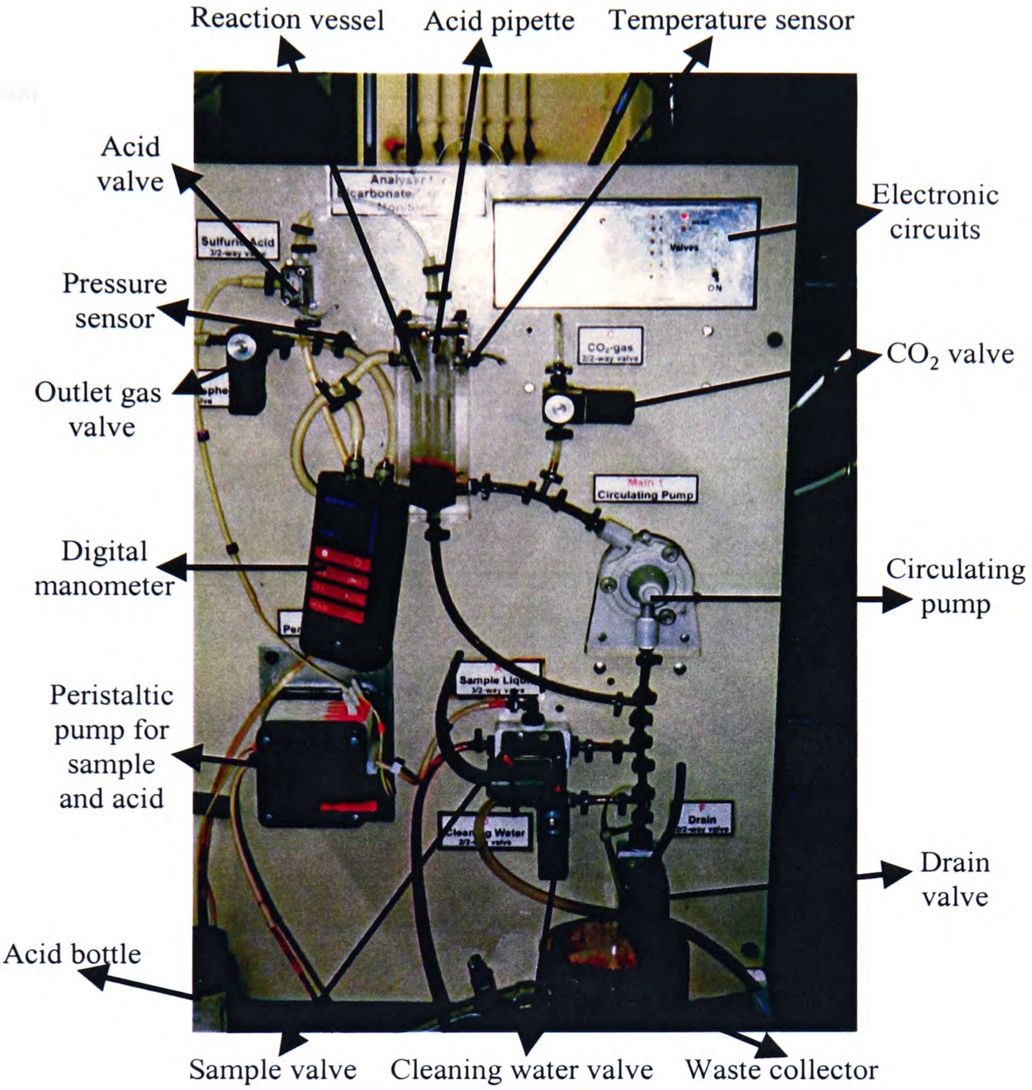


Figure 3.3 – Front of the intermittent BA analyser

Data acquisition - A dedicated computer (Figure A.8, Appendix A) fitted with a 12 bit data acquisition board (RTI 815, Analogue Devices Inc., Norwood, MA) was used for local data acquisition and display and under this project also to transmit the data to the central logging/control computer (Section B.1, Appendix B).

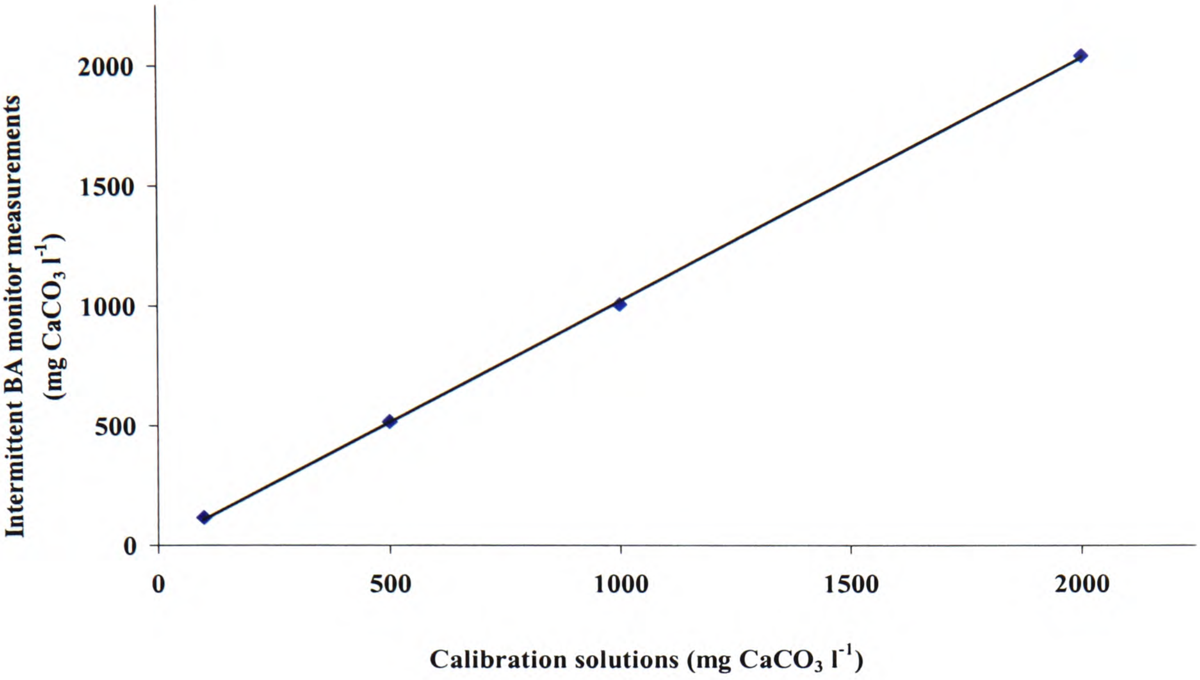


Figure 3.4 – Calibration graph for the intermittent BA monitor

3.3.4. Temperature probe

A temperature probe was assembled using an inexpensive calibrated thermistor IC LM35 DZ (RS Components Ltd. Corby, UK) (Figure A.13, Appendix A) a three-terminal integrated circuit temperature sensor, which gave a linear voltage output of 10 mV per degree centigrade. A signal was sampled with the NB-MIO-16 in the central logging/control computer (Section 3.5.1). The operating range of the sensor was 0 - 100 °C and it was calibrated against a digital thermometer (Minitherm HI 8757, Hanna Instruments). The temperature probe was sealed within a polyurethane tube with permanent glue and inserted into the UASB reactor liquid through the side port (Figure 3.1). Due to fouling in the UASB reactor the temperature sensor was re-calibrated monthly against the digital thermometer, which was inserted through one of the gas ports in the lid of the reactor.

3.3.5. Organic Strength Monitors

Two commercial organic strength monitors were used to monitor the health and efficiency of the two biological treatment stages. The TOD analyser measured the oxygen consumed during catalytic combustion of a sample, whereas the TOC analyser measured the difference between the TC and the IC.

Total Oxygen Demand (TOD) Analyser

The commercial Ionics Model 7800 E Total Oxygen Demand Analyser (Ionics UK Ltd., Manchester, UK) was designed to continually measure TOD in a sample of water. TOD is the amount of oxygen consumed during the catalytic combustion of a sample. The momentary depletion of oxygen in the carrier gas was detected by a zirconium oxide fuel cell and the voltage was logged (Section 3.5.1). The analyser was configured to have an operating range of 0 to 3333 mg l^{-1} by changing the length of the silicone rubber tubing through which the oxygen from the air permeated. Operation, calibration and maintenance proceedings were accomplished accordingly to the manufacturer's manual. The TOD analyser was tested during Experimental Phase 2 and used continuously during Experimental Phase 3. The sample stream was filtered continuously (Section 3.4) whilst being pumped at 10 ml min^{-1} with 20 μl being drawn off for measurement. Samples were taken approximately every 2.5 minutes. After every measurement, there was a rinse with DI water and after Experiment 3.4 this rinse was performed with a solution of 5 % nitric acid (Fisher Scientific Ltd., Loughborough, UK) and DI water.

Calibration and data acquisition - The TOD was calibrated using a 3333 mg l^{-1} solution prepared with 2.8363 g of potassium hydrogen phthalate per 1000 ml of DI water. This TOD measurement corresponded to 10 V output using a voltmeter and by adjusting the voltage pot. Posterior, a display pot adjustment was performed so that the corresponding value of 3333 mg l^{-1} was obtained. After the monitor was tested with a 1500 mg l^{-1} TOD solution. A linear voltage output was sent to the central logging/control computer where there was also a calibration of the voltages received to the corresponding TOD values (Section 3.5.1). Calibration of the monitor was performed every 1 to 2 days.

Maintenance - The injector was taken apart and cleaned on a regular basis and every 1 to 4 days the injection tube was replaced. The platinum balls inside the catalyst chamber were cleaned with DI water once a month and once they were boiled in concentrated HCl for 5 minutes and then washed with DI water and thoroughly dried. The chamber was repacked and a fibre frax material was put on top of the catalysts to ensure that the sample was filtered slowly onto the catalysts.

Related analysis – since keeping the monitor working for at least 1 week was a problem due to blockages of the injection tube, analysis of its contents was performed. Two injection tubes were autoclaved and cross-sectioned. Two samples were then gold coated. One of a tube section, another of just the residue inside. They were both analysed by X-ray microanalysis with a Stereoscan 240 electron microscope (Cambridge Systems, Cambridge, UK) fitted with a Link System X-ray analyser (Link Systems, High Wycombe, UK).

Total Organic Carbon (TOC) Analyser

The TOC analyser used was a DC-190 Rosemount Dohrmann (Sartec Ltd., Borough Green, UK) (Figure A.8, Appendix A). Its principle of operation was based on the high temperature catalytic combustion method followed by NDIR detection of CO_2. Samples were automatically injected into the analyser and an open glass flowcell was used for continuous measurements. This instrument measured TOC by determining TC and IC independently, injecting the samples in two columns, with TOC being obtained from the difference between TC and IC. TC was analysed by injecting a sample of the reactor's waste stream into a blended air gas stream, which passed through a combustion tube set at a temperature of 680 °C packed with platinum catalyst. The organic carbon was oxidised to CO_2 and transported by the carrier gas to a NDIR detector, where it was quantitatively measured. IC was analysed similarly by injecting a sample from the reactor into a separate reaction chamber at 150 °C, packed with phosphoric acid-coated quartz beads. The organic carbon was prevented from oxidising under acidic conditions. Only the IC was converted to CO_2, which was also measured by the same detector. The IC and TC sample acquisition of 50 μl was performed by an automatic injection system, which was then, introduced into the TOC analyser. A complete TOC analysis took approximately 6 - 8 minutes from which 3 to 4 minutes was the time taken from the output of the TC result and the starting of the IC measurement. The operating range of the analyser was of 0.2 – 50,000 (ppmC). The TOC monitor was only operated for Experimental Phases 4 and 5 (Sections 3.6.4 and 3.6.5). In Experimental Phase 4 the sample was from the UASB reactor effluent and during Phase 5 the sample was taken from the middle of the settler to reduce blockage of the filters.

Requirements and settings - The sample was filtered (Section 3.4) and was not recycled to the UASB reactor during Phase 4 (Filter 3 and TOC flowcell were open to the atmosphere) however, it was recycled back to the aerobic tank during Phase 5. A 60 μm mesh filter from Sericol - Industrial Fabrics (Kent, UK) was used with Filter 3.

Calibration - The instrument was calibrated weekly or at the start of a new run, whichever came sooner, for TC and IC. TC stock solution 2.126 g of potassium hydrogen phthalate (Fisher Scientific Ltd., Loughborough, UK) was made up to 1000 ml with DI water. The final concentration was 1000 mg C l^{-1}. Phosphoric acid was added to lower the pH to 3 or less. For the IC stock solution 6.99 g of $NaHCO_3$ (Fisher Scientific Ltd., Loughborough, UK) was made up to 1000 ml with DI water, giving a concentration of 1000 mg C l^{-1}. Each standard was injected 3 times by the auto-sampler. When the *sd* was greater than 5 % for the TC measurement the chamber was cleaned with HCl and for the IC measurement the chamber was cleaned with two automated injections of phosphoric acid (20 % phosphoric acid solution) which was prepared monthly, or even with the replacement of the contents of the chamber with DI water primed with phosphoric acid.

Data acquisition - The TOC analyser had software for data acquisition (DC190 Terminal Software – Dohrmann Data Talk II (Sartec Ltd., Borough Green, UK)) installed on a PC (Figure A.10, Appendix A), which transferred data and time via the serial communication port (Section 3.5.1). The data was also sampled every 0.5 seconds using a built LabVIEW™ VI and a NB-MIO-16X data acquisition card on another computer by using the waveform voltage output from the analyser's NDIR detector. The VI integrated the area under the 2 peaks (i.e. TC and IC) to calculate the final value of TOC (i.e. TC – IC) (Figure A.10, Appendix A) (Figure 4.15). The TOC analyser had 1 light-emitting diode - LED for each of the two chambers, which radiated in the visible region every time each chamber was in operation. Two optical schmitt trigger detectors (IS 436) (RS Components, UK) (Figure A.12, Appendix A) were set above the LEDs and each signal 0 - 5 V was taken by the PC that indicated the start and the end of each measured peak. The three input signals were filtered using a hardware low-pass filter (i.e. circuit built using a 1 μF capacitor and a 82 kΩ resistor from RS Components, UK) (Figure 3.6). After the integration of the peaks the same board output the voltage signal of TC and IC to the central logging computer running another LabVIEW™ program and only then the TOC was calculated.

3.3.6. On-line Colour Analysis

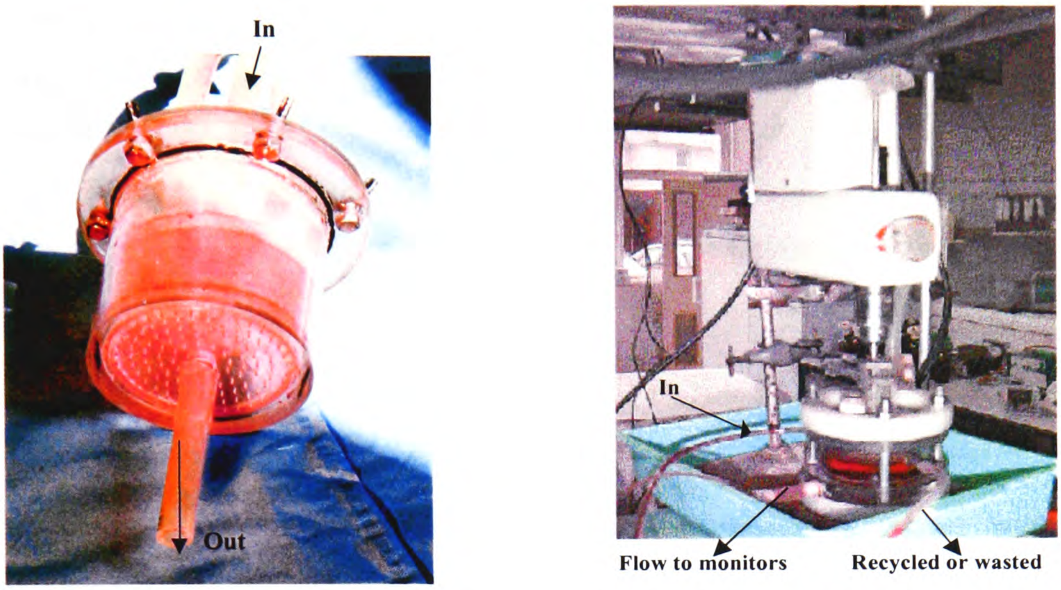
An UV/Visible Spectrophotometer using a standard 10 mm quartz flowcell both from Unicam Ltd. (Cambridge, UK) were used for on-line colour measurements (Figure A.11, Appendix A). It was set to measure on-line OD of samples at the wavelengths of 436, 525 and 620 nm (as recommended in BS 6068) within a range of 0 - 2.9 absorbance units. Calibration was performed as in Section 3.2.3 using the flowcell instead of the visible cell. The sample was filtered prior to measurement to avoid interference by non-dissociated matter (as in Sections 3.4, 3.6.3 and 3.6.5). The filtration systems used were thought not to cause oxidation reactions, as there was no contact with air except minimal contact in Filter 3. The sample flowrate to the flow cell was set to 1.8 and 1.5 ml min^{-1}, for Experimental Phase 3 and 4, and Phase 5, respectively. For efficient operation of the UV/Visible spectrophotometer, the samples were diluted from 3 – 7.5 times depending on the influent dye concentration using DI water (to be within the linear range of the instrument).

Extra requirements – The flowcell was getting dirty sometimes after only 3 hours of operation and had to be cleaned using acid ethyl-acetate (AnalaR), which disrupted the measurements. Residue inside the flowcell was collected and stained using 1 % methylene blue for 30 seconds and then was observed using a microscope. As filamentous bacteria were observed, the use of a biocide was appropriate. The dosing of this biocide was noticed to be even more fundamental after dilution as for two trials the growth of bacteria was even more effective suggesting that the UASB effluent was quite toxic but after diluted with water bacteria could grow quite effectively. The choice of biocide depended on its physical-chemical characteristics. The biocide was first tested and characteristics were found which proved to be suitable for the task. A broad spectrum biocide (Panabath M, BDH Chemical Ltd., Poole, UK) was added at 0.25 ml l^{-1} of DI water used for dilution preventing the growth of biofilm. After diluted it presented no absorption for the 3 measured wavelengths, it did not alter the pH of the sample and it did not help degradation of the coloured sample.

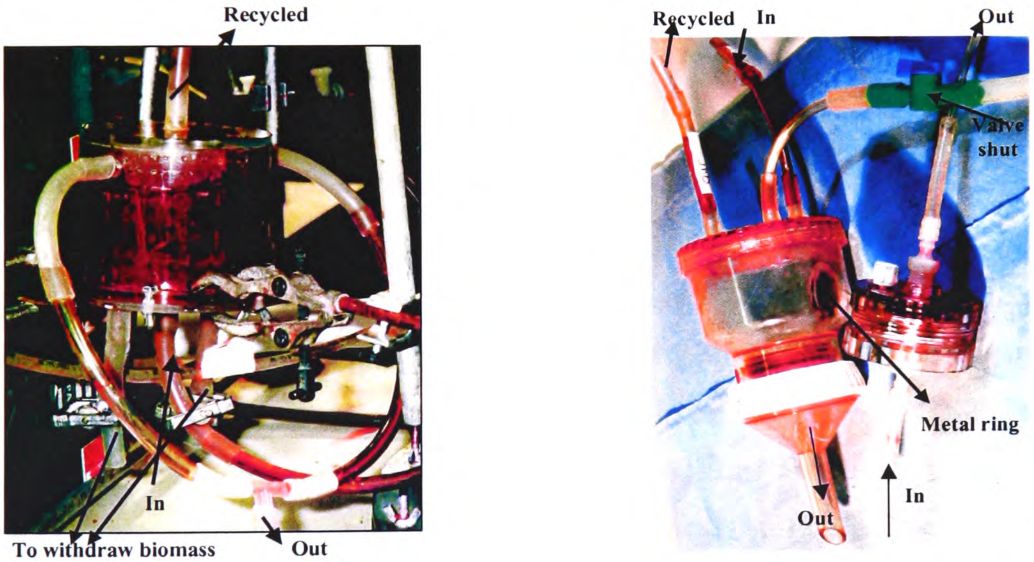
Data acquisition - A PC program (ASDS – Unicam Ltd., Cambridge, UK) already existed for dual communication with the analyser. However, the data was required in the central logging computer where the serial links were different than a standard RS-232. Therefore, a QuickBasic program was built for communicating in terminal mode with the analyser so that the data acquired by the PC via the serial port could be manipulated and sent to the central logging system as voltage signals (Section B.2 – Appendix B). Therefore, a digital to analogue (D/A) card was assembled using a quad-8-bit converter (RS Components, UK) in Interface Box 4 (Figure 3.6, Figures A.6 and A.7 of Appendix A). The inputs to this card were values sent out using the PC parallel port and the converter output 4 voltage signals, which corresponded to absorbance at the 3 wavelengths and a value for the average. However, only the ODs at 525 nm and the average of the three were sent to the central computer due to restrictions in the NB-MIO-16 card.

3.4. Filtration systems for on-line instruments

On-line instruments such as the BA, TOD, TOC and colour monitors needed filtered samples so as to minimise blockages. In case of the colour analyser, it was also to approximate the reading to the true colour measurements performed off-line as in Section 3.2.3 and not to interfere with the measurements of the TOD and TOC analysers. The setup of each filter together with the on-line instruments is shown in Section 3.6 for each of the Experimental Phases. Two different sizes Saatifil Polyester meshes (Industrial Fabrics – Sericol Ltd., Broadstairs, UK) were used by the five different filters. An in-line filter (Filter 1) and a cross-filter (Filter 2) were designed and built using perspex for this particular project. A self-cleaned cross-filter (Filter 3) built under another project and two reusable plastic filters holders (BDH Ltd., Poole, UK) one with funnel (Filter 4) and an in-line filter (Filter 5) were also used. Photographs of the filters are presented in Figure 3.5. The liquid volumes for Filters 1, 2 and 3 were 261, 650 and 83 ml, respectively. For all the filters the 185 μm mesh was utilised except for Filter 3. This last one being a self-cleaned cross-filter (cleaned by a rotating brush) had the capability of withstanding without frequently blocking a tighter mesh (60 μm).



Filter 1 Filter 3



Filter 2 Filters 4 and 5

Figure 3.5 – Photograph of filters

In case of Filters 2, 3 and 4, a recycle was included to promote the monitoring of fresh samples. The tubes going from the stages to the filters and to the analysers were all polyurethane tubing to avoid growth of bacteria on the walls. Silicone rubber tubing promoted the growth and deposit of the bacteria on the walls possibly due to its roughness.

All the filters, had to be taken apart and cleaned regularly as granules, suspended biomass and also bacteria which grown in the meshes clogged them. For all the filters except Filter 3 the operation time was of 1 – 2 days before cleaning, whilst Filter 3 stud 4 days operation without being manually cleaned. These polyester meshes were cleaned with tap water and reused again. Before assembly the filters had to be filled with tap water for pumping purposes and also for Filter 2 and 4 no air was to be pumped to the UASB reactor by the recycle line. Filter 4, as it can be seen in Figure 3.5, had a metal ring to sit on top of the mesh so that this did not lift up by the vortex force created at the top part of filter.

3.5. Interfacing Hardware and Software for Monitoring and Control

This Section describes the contents of the interface boxes, the central logging/control computer in terms of hardware and software and also the software for data transfer between LabVIEW™ and MATLAB®. Finally, it presents a description of actuators used for controlling the biological treatment process on-line and respective calibration.

3.5.1. Interface Boxes and Central Logging/control Computer

The flow of the on-line instrument signals to the central logging/control computer and the respective feedback control signals to the actuators is depicted in Figure 3.6. All the signals from the on-line sensors described above were joined in the Interface Box 1 and sent through to Interface Box 2 (Figures A.1 and A.2, Appendix A). Interface Box 1 had an electronic circuit for conversion from current to voltage and also a 10-fold amplification of the signal from the H_2 analyser (Figure A.4, Appendix A). It had also a circuit built for stability of the CO_2 analyser signal (Figure A.5, Appendix A). Interface Box 2 included two CB50 (National Instruments, Newbury, UK). One of the CB50 was used to connect the signals reaching Interface box 2 to the NB-MIO-16H card (National Instruments, Newbury, UK) (16 inputs and 2 outputs analogue channels). In this CB50 hardware standard low-pass filters (82 kΩ resistor and 1μF capacitor) for all the monitored signals were built to attenuate the electrical noise. The other CB50 was used to ungroup the output signals from the NB-AO-6 card (National Instruments, Newbury, UK) (6 output analogue channels), which were then sent back to the Interface Box 1. Interface Boxes 3 and 4 were directly related with the TOC and colour analysers (Sections 3.3.5 and 3.3.6). The central computer incorporated the two interface cards (NB-MIO-16H and NB-AO-6). A VI developed in LabVIEW™ was configured to scan the on-line sensors every 2 minutes, with the data and time being stored in a data file. Data updates for the colour and TOD analyser occurred only every 4 minutes, for the TOC analyser every 6 – 8 minutes and for the BA monitor every 1 hour except during Experiment 4.2 where the measurements were performed every 30 minutes. This VI also included the on-off pH and DO set-point controllers for the aerobic stage.

Two other VIs included output control signals from the ANNBCS in addition to the acquisition of data. One of these VIs was for the control of the UASB reactor during Experiment 4.2 and the other for the control of the aerobic stage during Experiment 5.3. A screen capture of part of the first VI is shown in Figure 5.8.

Within the central computer there were two other packages, a data storage software which served to save sensorial and remedial action information and also used to transfer data from LabVIEW™ to MATLAB® and back, as described in Section 3.5.2. The MATLAB® programming package and its associated Neural Network Toolbox®, both supplied by Cambridge Control Solutions (Cambridge, UK) were used to build the ANNBCS developed in this work. More information on this will be provided in Sections 3.5.2.

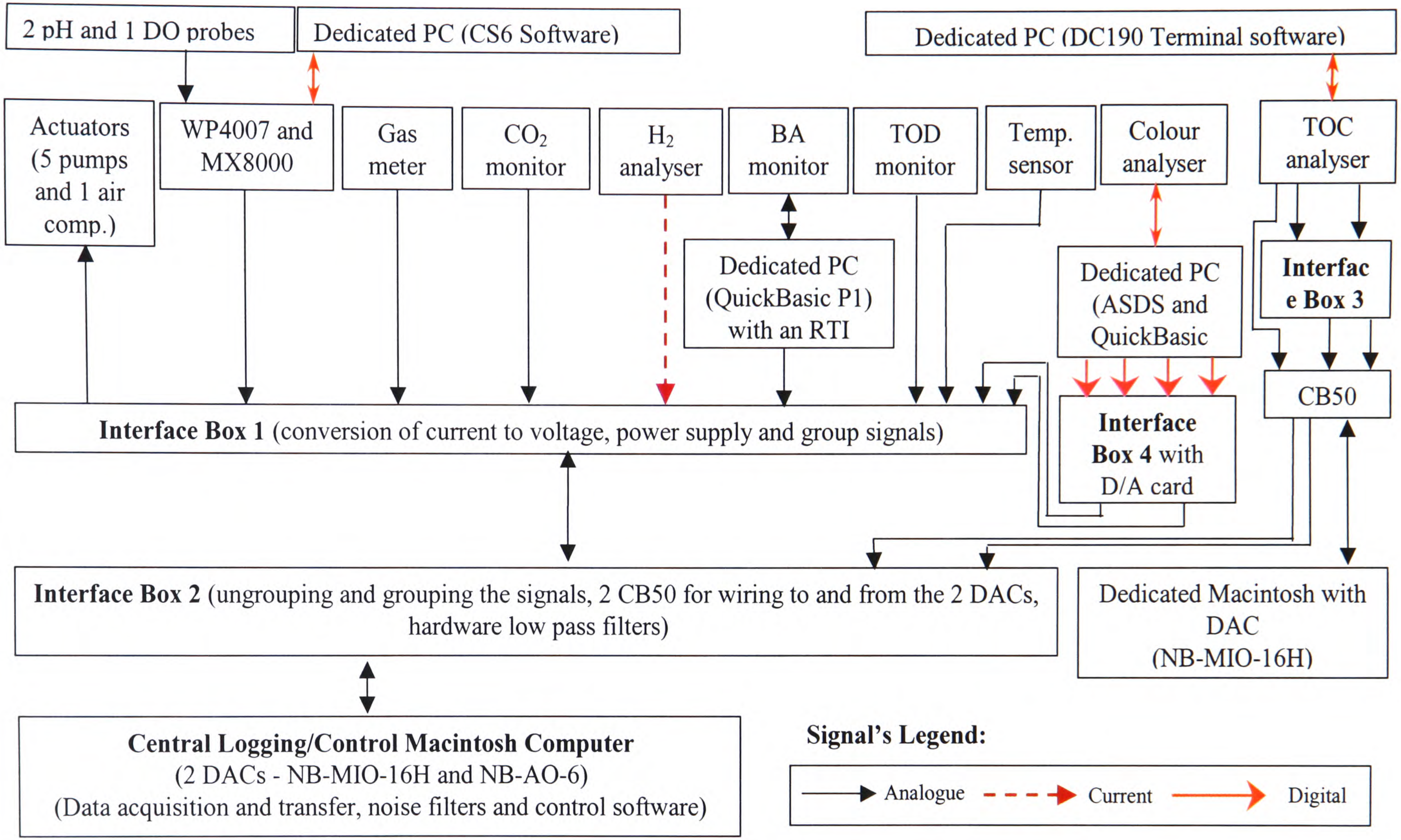


Figure 3.6 – Summary of the hardware and software used for monitoring and control

3.5.2. Data Transfer Software for Control

Figure 3.7 shows the data flow in the central computer during experiments controlled by the ANNBCSs. From the sequence it can be seen that sensorial information from the plant was logged in the central computer using LabVIEW™ **(1)**. The VI in LabVIEW™ transferred sensory data, date and time to a file **(2)**. MATLAB® in **(3)** retrieved this information, processed it and returned the necessary remedial actions to another data file **(4)** which was subsequently read by LabVIEW™ **(5)** at the frequency of 2 minutes where it was converted to an analogue signal that was used to manipulate plant actuators **(6)**. An example of the software for data transfer and control written in MATLAB® is presented in Section B.3 of Appendix B.

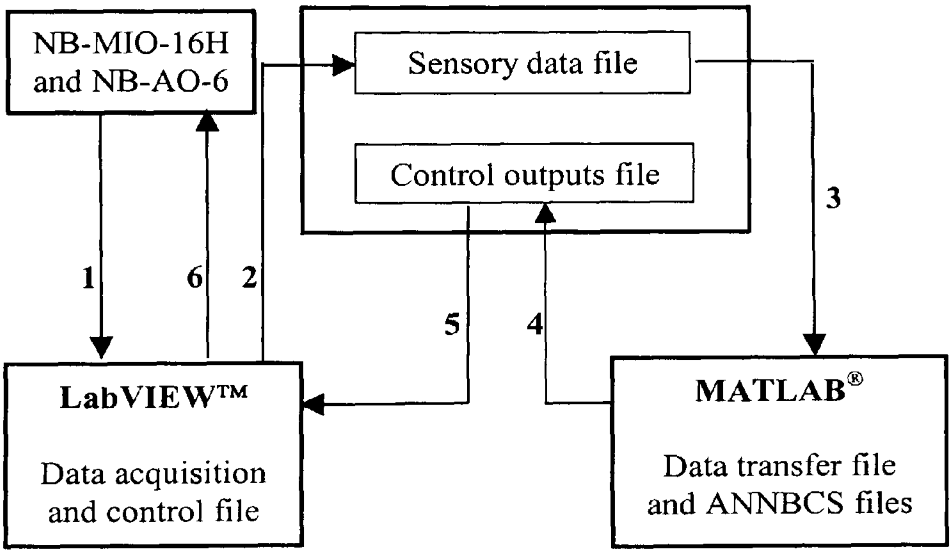


Figure 3.7 – Software within the central logging/control computer for use with the ANNBCSs

3.5.3. Actuators Controlled Via the Central Logging/control Computer

Two types of actuator were controlled via the central computer. These were five peristaltic pumps (model 505U, Watson Marlow Ltd., Cornwall, UK) and one air compressor. The five peristaltic pumps controlled the pH in the aerobic vessel in an on-off mode at constant speed (Experiments in Phase 2 and 3 and Experiment 5.1 and 5.2), and with variable speed the pH of the aerobic tank and the extra starch delivery to the aerobic tank (Experiment 5.3), the delivery of dye and the adjustment of BA both for the UASB reactor (Experiment 4.2). For the last four pumps the calibration was made in 'voltage mode' accordingly to the Watson Marlow manual for variable speed output.

An on-off switch was adapted to the back of one air compressor and was connected to the central computer. The air compressor was controlled in an on-off manner as described in Section 3.1.1 in Experimental Phase 3 and in Experiments 5.1 and 5.2. It was controlled by the ANNBCS during Experiment 5.3.

3.6. Experimental Design, Monitoring and Control systems

This Section describes the experimental design, and briefly the monitoring and control systems adopted during the five Experimental Phases.

3.6.1. Experimental Phase 1 – Monitoring of a Fluidised Bed Reactor (Previous Project)

The reactor setup, on-line instrumentation and influent to the WWT system have been previously described (Guwy *et al.,* 1997a). A laboratory scale fluidised bed anaerobic digester, consisting of two linked reactors giving a total working volume of 11 litres. It was operated for 8 months at an 8 - 9 h HRT on a simulated baker's yeast WW, with a sintered glass carrier (Siran®). During operation the average volumetric loading rate (B_v) was 18.87 kg COD m^{-3} day^{-1}, giving a 75 % removal of soluble COD. Percentage CO_2, biogas H_2 concentration, biogas flowrate, BA and pH were measured on-line. The organic loading rate could be varied automatically, as concentrated feed and dilution water were supplied via two separate pumps. The relationship between the feed delivering into the reactor with the pump input voltage was linear. As the rate of flow of the dilution water was considerably larger than the rate of flow of the concentrated feed, the HRT in the reactor changed only slightly during changes in the organic load. Figure 3.8 shows a typical pattern of the concentrated feed into the reactor, which consisted of the three types of input signals. The flow pattern was also obtained via the use of a Pseudo Random Binary Signal (PRBS) generator to alter the input voltage to the pump.

The data used to train the ANNs was acquired from operating the anaerobic digester for three different conditions (Table 3.1). As for the textile effluent treatment project sensory information from the plant was logged on a computer with a NB-MIO-16H interface card in conjunction with LabVIEW™, both supplied by National Instruments Corporation Ltd. (Newbury, UK). The VI was configured to scan the on-line sensors every 2 minutes with the data, date and time being logged to data files for off-line processing. The data acquired was used to select the most appropriate ANN to control the biological process (Section 5.1).

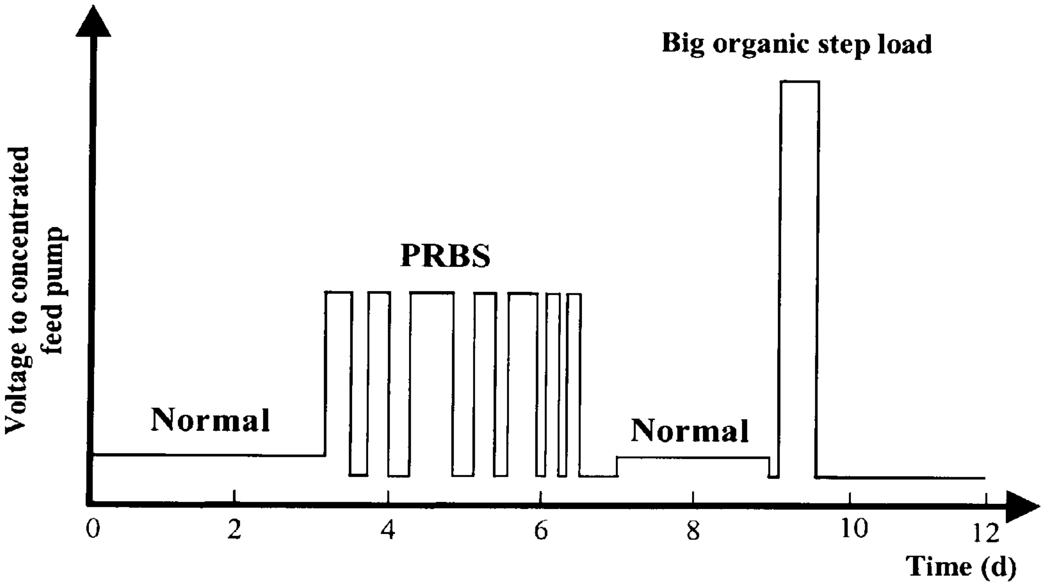


Figure 3.8 – The three feeding signals to the reactor (not to scale)

Table 3.1 - Organic Loading Rate for the three different operating conditions (Experimental Phase 1)

Designation	Organic Loading Rate (kg COD m^{-3} d^{-1})
Normal	18.87
PRBS	17.89 - 38.33
Big organic overload	16.41 - 70.03

3.6.2. Experimental Phase 2 – Monitoring of the Combined Anaerobic and Aerobic Treatment (A)

The UASB reactor, aerobic tank and settling vessel used here are described in Section 3.1.1. The setup, auxiliary equipment and on-line monitors used during this Phase are illustrated in Figure 3.9. The gas meter (Guwy *et al.,* 1994) was the prototype presented in Section 3.3.1. The CO_2 analyser, pH and DO probes, intermittent BA monitor and the temperature probe were described in Section 3.3. The sample to the BA monitor was filtered using Filter 2 (Section 3.4). The TOD analyser was operated sporadically with samples from the side port of the UASB reactor. The sample to the TOD analyser was manually filtered using the 185 μm mesh (Section 3.3.5). Off-line analyses were performed for VFAs, pH, BA, CH_4, CO_2, TS and VS for the UASB reactor biomass, MLSS and VSS for the aerobic tank, and colour and COD of the STE, UASB reactor effluent and settler effluent (Section 3.2).

The UASB reactor was seeded with 10 l of granules from the BPB Paperboard Davidson (Aberdeen, UK) (TS of 60 g l^{-1} (*sd* = 2.28, n = 6) and a VS of 49 g l^{-1} (*sd* = 1.85, n = 6)), whilst the aerobic tank was filled with 10 l activated sludge from a Welsh Water Sewage Treatment Works, UK. The STE described in Section 3.1.2 was fed to the UASB reactor and its effluent was fed to the aerobic tank. Aerobic tank effluent was passed to the settling vessel prior to discharge. The reactor was operated for 28 days after a 7 day start-up period with a similar STE described in Section 3.1.2, except that the NaCl content was 1.5 g l^{-1} instead of 0.15 g l^{-1} at a 2.5 d HRT with a starch concentration of 1.9 g l^{-1} and azo dye 1.5 g l^{-1}. Afterwards the reactor was operated for 26 days at 1.7 d HRT with a reduced salt and dye content (both were 0.15 g l^{-1}). Due to the long HRT a recycle flow from the topside port back to the influent line was used to assist mixing within the reactor. Some results obtained with these working conditions were presented by O'Neill *et al.* (1999b). For Experimental Phase 2 reported here the UASB reactor HRT was reduced to 1 d and recycle was resumed. The influent used was that described in Section 3.1.2. The recipe was changed to promote better colour removal; a larger throughput was needed for sampling and the feeding of the aerobic stage, and also to reduce the toxicity potential from the salt content. During Experimental Phase 2 the aerobic stage was operated at a 19 h HRT and was restarted with fresh sludge. The pH was controlled and the sludge was recycled every 4 hours (Section 3.1.1).

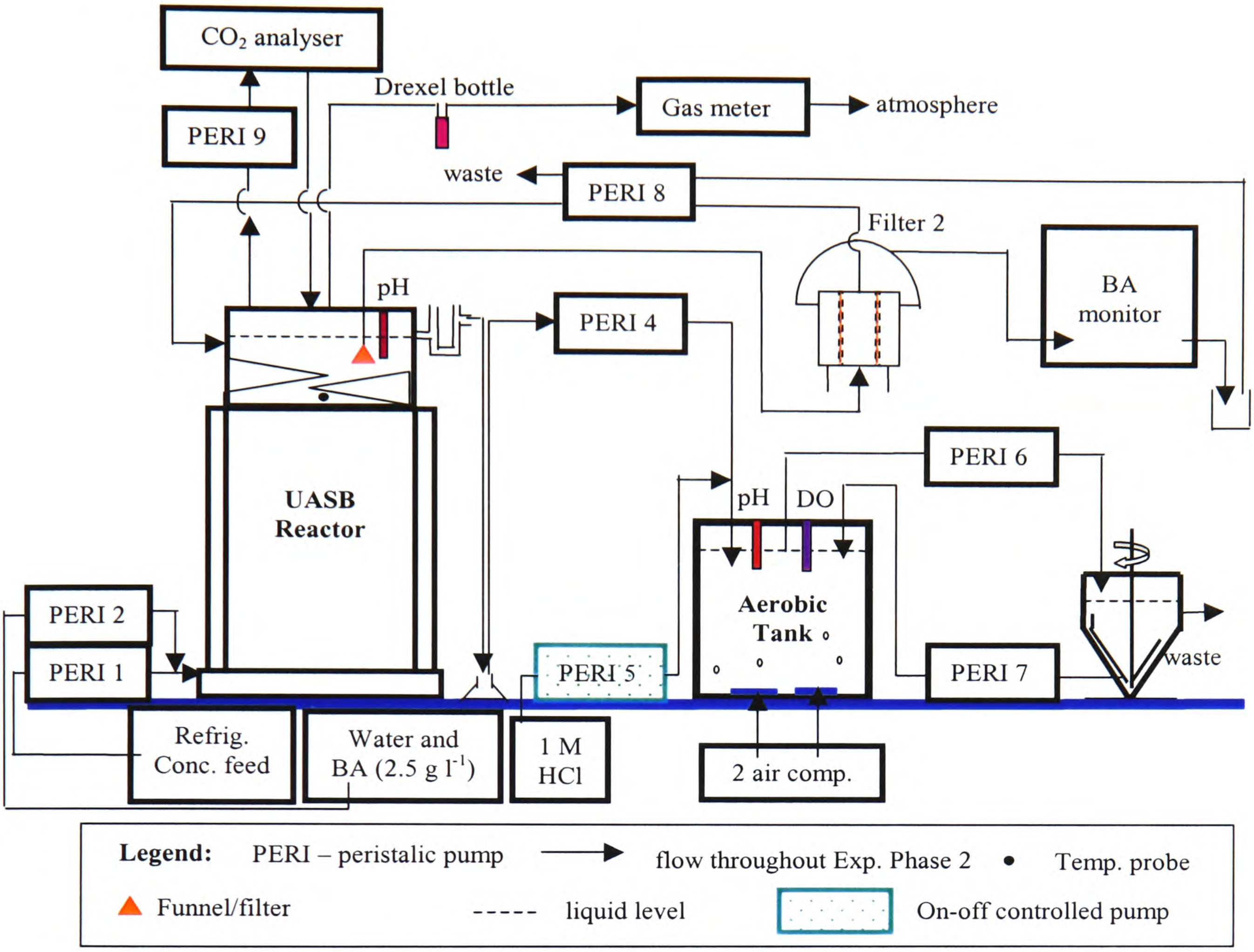


Figure 3.9 - Schematic of the rig, location of the on-line instruments and local control of aerobic tank pH (Experimental Phase 2)

A series of four systematic experiments were conducted which consisted of varying both the dye and starch concentrations in a series of step changes during a four-week period. The objective was to investigate the effect of some feed variations in the dye and starch levels (Table 3.2) on the anaerobic process and to determine an optimal set of operating conditions about which to control the process. All experiments were conducted at a 1 day HRT for the UASB reactor, for 7 days. The data acquired from this Phase was used in Section 5.2 in order to develop a Control scheme.

Table 3.2 - Concentrations of dye and starch (Experimental Phase 2)

Experiments	Dye (g l^{-1})	Starch (g l^{-1})
2.1	0.075	1.9
2.2	0.075	0.95
2.3	0.15	1.9
2.4	0.15	0.95

3.6.3. Experimental Phase 3 – Monitoring of the Combined Anaerobic and Aerobic Treatment (B)

Stronger organic and colour loads were designed for Experimental Phase 3 as compared to Phase 2. This because the B_v for the experiments of Phase 2 was below the recommended level by An *et al.* (1996) of 5 g COD l^{-1} d^{-1}. Also the maximum B_v of Phase 2 did not stress the UASB reactor and the aerobic biomass did not grow, even after pH control, possibly due to lack of organic load.

Experimental Phase 3 followed Phase 2 after a non-operating period of 52 days. The granules at the end of Phase 2 had a 43.8 % and 46.7 % loss of TS and VS, respectively. The same biological treatment stages were used with the same HRT as in Phase 2. The aerobic stage was re-started at the beginning of this Phase. Settled sludge was recycled continuously from the settler to the aerobic tank. Both pH and DO were controlled in an on-off manner (Section 3.1.1). The rig was instrumented as shown in Figure 3.10. The on-line monitors of the UASB reactor were the low flow gas meter (LFM 300, G.H Zeal, London, UK), CO_2 analyser, exhaled H_2 monitor, pH and temperature probes, intermittent BA monitor, TOD analyser, and UV/Visible spectrophotometer. There were 'in situ' on-line pH and DO probes in the aerobic tank.

The filtration used for the BA, TOD and colour monitors are shown in Figure 3.10 and more information can be found in Sections 3.3.3, 3.3.5, 3.3.6 and 3.4. On-line data was supplemented with off-line results. These were pH, BA, gas composition (CH_4 and CO_2), VFAs, COD, and true colour measurements, TS and VS concentration and MLSS and VSS for the aerobic stage and occasional readings of H_2S in the biogas of the UASB gas space were carried out. The two injection tubes for the TOD analyser (i.e. section and residue) analysed using X-ray micro analysis were used during this Experimental Phase. Staining analysis of the residue inside the UV/Visible Spectrophotometer flowcell was also performed during this Phase.

In order to choose the best monitoring parameters and also to train the control scheme distinct changes in treatment efficiency and effluent quality (i.e. effluent organic and colour residuals) had to be accomplished. Therefore, an experimental programme was designed where starch and dye concentrations in the STE were varied. The concentrations chosen were based on previous results and literature. Also the maximum concentration of starch was determined in order to avoid sodium toxicity in the UASB reactor since the starch was hydrolysed, using NaOH. The sodium concentration in the STE was added to by the sodium hydrogen carbonate used as a buffer and slightly by components such as: salt, nutrients and the salt content within the dye.

Throughout the experiments, the rig was operated at an overall system HRT of 1.8 days. The dye and starch concentrations used in this Phase can be seen in Table 3.3. The rig was operated on a feed composition of 0.15 g l^{-1} dye and 1.9 g l^{-1} starch for 22 days (defined as 'Initial Experiment' in Table 3.3) to obtain steady state values with good effluent quality. To collect data under conditions giving varying treatment efficiency in order to train the control system, a programme of varying starch/dye contents in the STE was then followed, by varying the dye and starch concentrations in 7 day periods in three further experiments (Experiments 3.2 – 3.4 in Table 3.3) each followed by a return for 7 days to the initial experimental conditions (Experiment 3.1). Each Experiment 3.2 – 3.4 was repeated. Thus Experiment 3.1 spanned seven 1 week periods. In Experiments 3.3 and 3.4 the starch was doubled to 3.8 g l^{-1} ('high starch'), and in Experiments 3.2 and 3.4 the dye was increased 5-fold to 0.75 g l^{-1} ('high dye'). Also no addition of BA for a period of 25 h (Experiment 3.5) and step ups by a factor of 10 of the value of acetic acid in the feed during 4.5 h (Experiment 3.6) were accomplished using the dye and starch concentrations as in Experiment 3.1. Afterwards the Experiment 3.7 of 16 days duration was performed using 0.45 g l^{-1} ('medium dye') and 2.9 g l^{-1} ('medium starch'). During Experiment 3.7 a concentrated supplement of OECD simulated sewage waste (OECD, 1981) was fed to the activated sludge stage at a rate of 1.4 litres per day. During Experiments 3.8 and 3.9, two colour impulses were performed.

In the two colour impulses three other reactive dyes were used: PROCION Blue P-GR, PROCION Yellow P-3R and PROCION Orange PX-2R. In Experiment 3.8, the extra dye concentration was 0.3 g l^{-1} for a period of 5 hours and 30 minutes while in Experiment 3.9 an extra concentration of 0.6 g l^{-1} was used for a period of 5 hours. Including the background dye concentration of 0.45 g l^{-1} (PROCION Red H-E7B) a maximum dye concentration of 0.75 g l^{-1} and 1.05 g l^{-1} were achieved for Experiments 3.8 and 3.9, respectively.

Table 3.3 – Influent for the biotreatment stages (Experimental Phase 3)

Exp.(s)	**Dye (g l^{-1}) (PROCION Red)**	**Starch (g l^{-1})**	**Others**
Initial	0.15	1.9	-
3.1	0.15	1.9	-
3.2	0.75	1.9	-
3.3	0.15	3.8	-
3.4	0.75	3.8	-
3.5	0.15	1.9	No addition of BA
3.6	0.15	1.9	Addition of extra acetic acid
3.7	0.45	2.9	Addition of OECD waste
3.8	0.45	2.9	Addition of 3 extra dyes (0.3 g l^{-1}) and OECD waste
3.9	0.45	2.9	Addition of 3 extra dyes (0.6 g l^{-1}) and OECD waste

Experimental Phase 3 ran for 8 months with the following sequence of experiments: Initial, 3.1, 3.2, 3.1, 3.3, 3.1, 3.2, 3.1, 3.3, 3.1, 3.4, 3.1, 3.4, 3.1, 3.5, 3.1, 3.6, 3.1, 3.7, 3.8, 3.7, 3.9, and 3.7. All the experiments had a duration of 7 days except Experiments 3.5 – 3.9. The first run of Experiment 3.7 had a duration of 2.5 weeks, the second of 1.5 weeks and the third of 5 days. During this Phase the aerobic sludge was collected 3 times. However it was given 1 week to adapt at Experiment 3.1 operating conditions. The UASB reactor was not fed (but was heated) for 4 weeks after Experiment 3.6. The highest B_v achieved was during Experiment 3.4 i.e. 3.9 g COD l^{-1} reactor d^{-1}. It was below the recommended value by An *et al.* (1996) however, as described in Chapter 4 some of the experiments in Phase 3 did stress the UASB reactor.

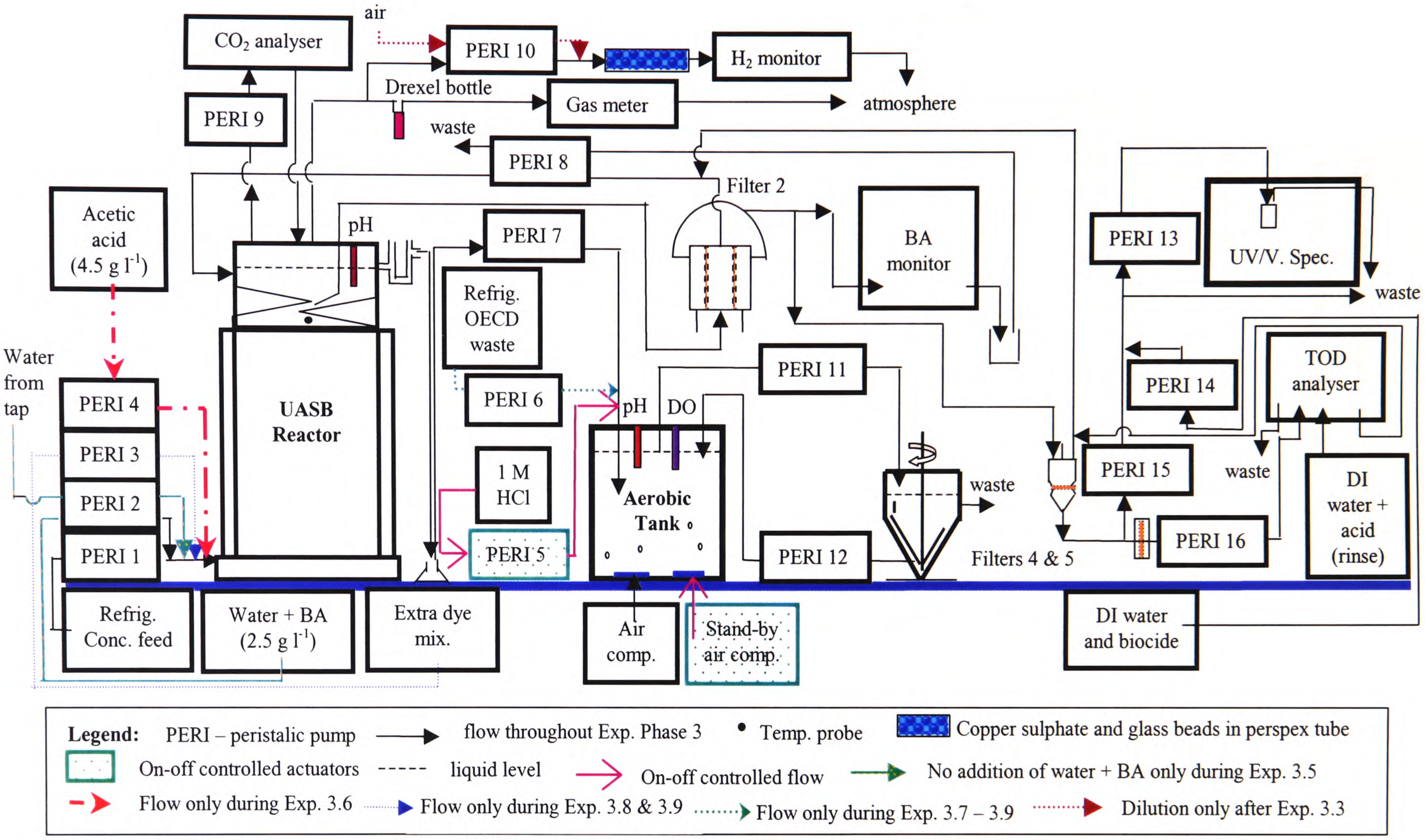


Figure 3.10 - Schematic of the rig, location of the on-line instruments and actuators (Experimental Phase 3)

3.6.4. Experimental Phase 4 – On-line Monitoring and Control of the Anaerobic Stage Using ANNs

After Experimental Phase 3, the UASB reactor was suffering from problems, which are discussed in Section 4.3.1 and therefore no starch step loads could be performed. Experimental Phase 4 followed a period of attempts to recover its health of 63 days (also summarised in Section 4.3.1) followed by another 16 days period of which the reactor was left heated but not fed. There were 6 litres of granules at the beginning of Phase 4 and TS = 13.1 g l^{-1} and VS = 10.6 g l^{-1}. A simple set of experiments was designed for Phase 4 in order to test the ANNBCS performance on the UASB reactor. The ANNBCS was required to control an extra addition of dye (PROCION Red H-E7B) and the addition of BA for buffering of the reactor. There was no acquisition of data for an uncontrolled experiment, as the reactor would not stand a BA starvation. The starch, dye and BA concentrations are defined in Table 3.4. Experiment 4.1 was only monitored and not controlled by the ANNBCS. Both experiments were repeated. Experiment 4.2 followed Experiment 4.1 and this sequence was repeated. Experiment 4.1 had duration of 37 days and its second run the duration of 12 days. During Experiment 4.1, 2.5 g of BA was dissolved in the water, which was used for dilution of the concentrated STE. During both runs of Experiment 4.2, the extra dye and the BA solution were available for the 7 hours before the extra dye and the BA pumps were manually turned off. At the end of this 7 hour period the water was pumped from the tank with a BA concentration of 2.5 g l^{-1}. Both, the extra dye and BA were stored in a concentrated form in bottles at concentrations of 9.925 g l^{-1} and 60 g l^{-1}, respectively. The STE for Experiment 4.1 was the same as for Experiment 2.2 (i.e. the lowest concentrations of dye and starch used during the experimental work). The UASB reactor, on-line monitors and actuators for this Phase are shown in Figure 3.11.

Table 3.4 – Influent starch, dye and BA concentrations (Experimental Phase 4)

Experiments	Starch (g l^{-1})	Dye (g l^{-1})	Extra dye (g l^{-1})	BA (g l^{-1})
4.1	0.95	0.075	-	2.5
4.2	0.95	0.075	Controlled[1]	Controlled[2]

[1]Maximum flowrate = 9.925 g l^{-1}; [2]Maximum flowrate = 60 g l^{-1}

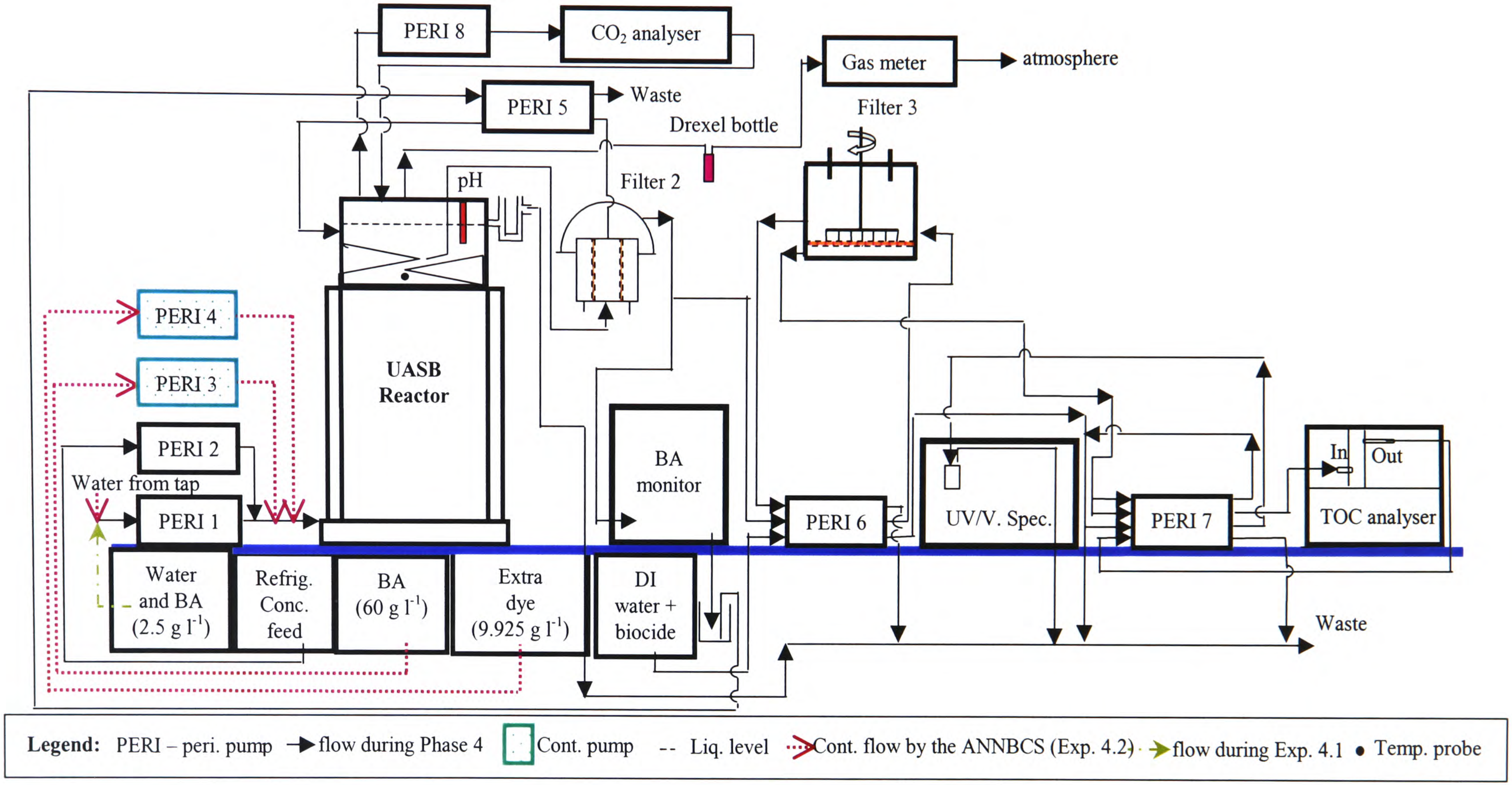


Figure 3.11 – Schematic of the rig, location of the on-line instruments, filters and actuators (Experimental Phase 4)

The UASB reactor was monitored by the LFM 300 gas meter, CO_2 analyser, pH and its effluent by the UV/Visible Spectrophotometer and TOC analyser. There were also off-line analyses: pH, BA, VFAs, gas composition, and the true colour and COD of the influent and effluent. The BA analyser, during Experiment 4.2 was set to sample every 30 minutes rather than every 1 h as the predictions for BA adjustment made by the ANNBCS were dependant on the analyser's measurements and also the pH. There were no measurements of H_2 in the biogas as the levels of gas production were considerably lower and 2.2 ml min^{-1} would have to be wasted. The overall lag time for the monitoring system was 16 minutes.

3.6.5. Experimental Phase 5 – On-line Monitoring and Control of the Aerobic Stage Using ANNs

The experiments of Phase 5 followed also the Experimental Phase 3 and were performed in parallel with the recovery period of the UASB reactor. The experiments here consisted of simulating an anaerobic stage failure and therefore raw industrial waste would have to be diverted straight to the aerobic stage. Phase 5 comprised 3 experiments of which Experiment 5.1 was performed 4 times and Experiment 5.3 twice. The sequence for the experiments was: new sludge, 5.1 (12 d), 5.2 (5 h), 5.1 (10 d), 5.3 (4 h), 5.1 (3 d), break for 16 days, new sludge, 5.1 (7 d), 5.3 (5 h), and 5.1 (13 d). The starch concentrations used during each experiment can be seen in Table 3.5. Experiments 5.1 and 5.2 were used to train and test the ANNBCS off-line. The ANNBCS controlled the extra starch intake, pH and DO of Experiment 5.3.

Table 3.5 – Influent starch concentrations (Experimental Phase 5)

Experiments	**Starch (g l^{-1})**	**Extra starch (g l^{-1})**
5.1	0.95	-
5.2	0.95	4.5
5.3	0.95[1]	Controlled[2]

[1] Hydrolysed with NaOH; [2] Maximum concentration 4.5 g l^{-1}

As the UASB reactor during the recovery period was being fed with starch hydrolysed with amylase (Section 3.1.2) to avoid sodium toxicity, the experiments in this Phase were also performed with amylase hydrolysed starch, except for one case shown in Table 3.5 due to difficulties in the hydrolysis process. In this case the starch was hydrolysed with NaOH (Section 3.1.2). The dye concentration was maintained at 0.075 g l^{-1} throughout the experiments, and there was also a constant addition of OECD waste (as in Experiments 3.7 – 3.9). The BA was added at 0.5 g l^{-1} and all the other substances were added in the same concentrations as those outlined in Section 3.1.2. There was on-off control of pH and DO (as in Section 3.1.1) for Experiments 5.1 and 5.2. For Experiment 5.3 these were controlled by the ANNBCS using the same actuators. There was continuous recycling of the solids so that they could be maintained in the aerobic vessel.

The aerobic stage, on-line monitors and actuators are shown in Figure 3.12. The aerobic tank was monitored 'in situ' by 1 pH and 1 DO probes. The TOC monitor measured the effluent of the settler and the UV/Visible Spectrophotometer measured the colour changes of the STE. The colour of the STE was monitored because the aerobic stage alone did not decolourise the waste and therefore, it was better to use the monitor at the influent so that the effect of the sample pH could be studied. The sample to this monitor was filtered, diluted and included biocide (Section 3.3.6). The flow to the colour analyser was 1.5 ml min^{-1}, lower than before, so that it would not significantly affect the HRT of the system. In this Phase the aerobic stage HRT was 20.2 h instead of the 19 h of Phases 2 and 3. Samples for the TOC had to be taken from the settler due to the density of biomass in the aerobic tank, which would demand cleaning of Filters 2 and 3, at least every 2 hours disrupting continuous measurements.

There were also off-line analyses, these were: influent and effluent COD and true colour, pH, MLSS, VSS and biomass activity (Section 3.2). The first three in order to make a comparison with similar on-line data and the others to complement knowledge about the system. The last three parameters were selected mainly to study how useful a measure of biomass activity would be for control of the RAS.

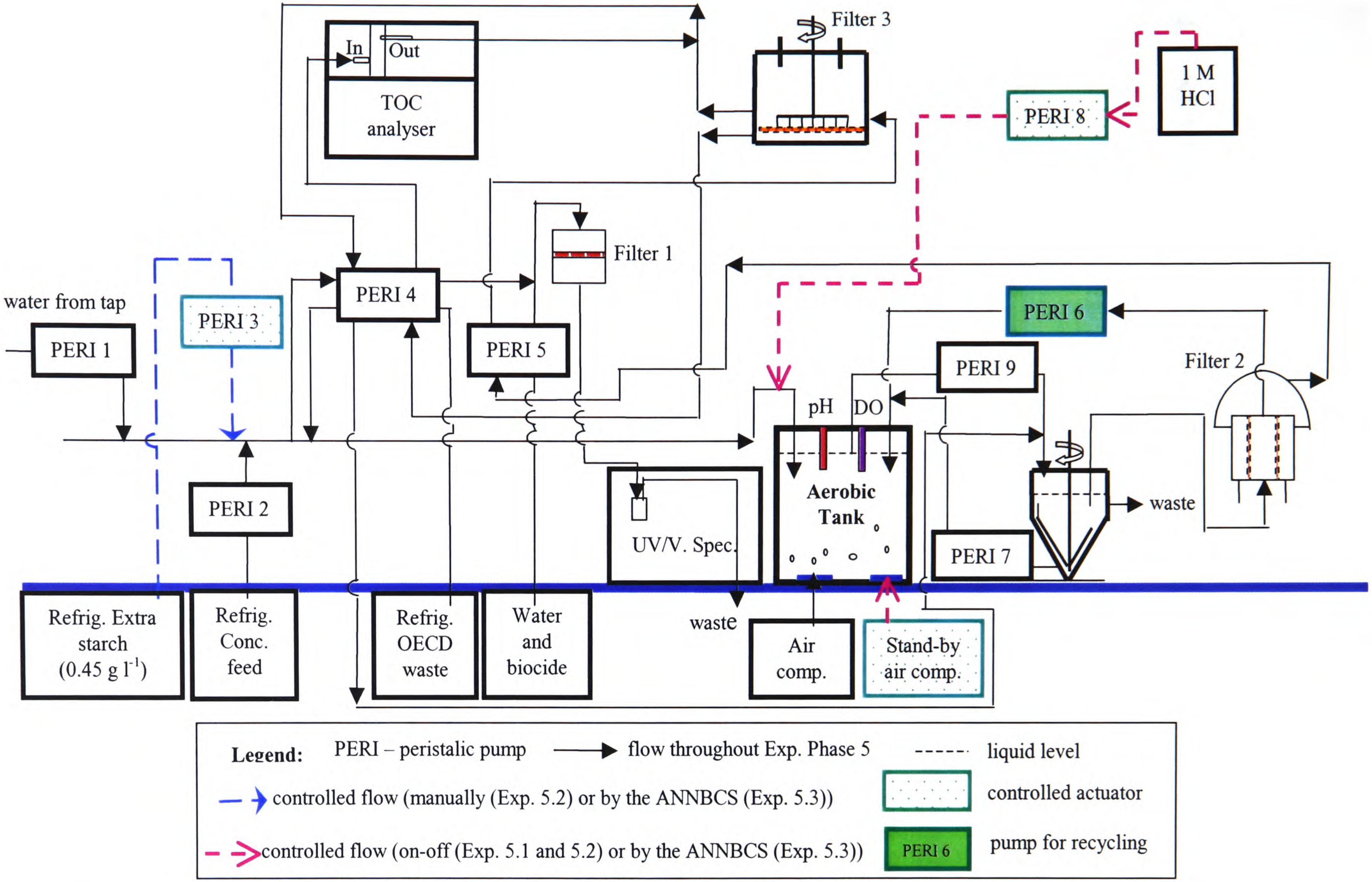


Figure 3.12 - Schematic of the rig, and location of the on-line instruments, filters and actuators (Experimental Phase 5)

4. SELECTION OF CONTROL PARAMETERS AND REMEDIAL ACTIONS

This Chapter examines the monitored parameters in the laboratory biological WWT system treating STE for use in a control scheme. It also compares the on-line measurements with off-line analyses where appropriate. The plant was operated according to Experimental Phases 2 to 5 (Chapter 3). The B_v and the COD and colour removed from the combined anaerobic-aerobic system is presented in Appendix C for Experimental Phases 2 and 3 (except experiments 3.6-3.9 because they were not 'steady-state' experiments). The results presented here were attained mainly from Experimental Phase 3 and the Experiments 5.1 and 5.2. The other experiments are dealt within Chapter 5 since the results were very much related to the on-line testing of the ANNBCSs performance. On-line parameters such as gas flowrate, $\%CO_2$, biogas H_2 levels, BA, pH, and effluent TOD, TOC, and colour, were assessed on-line for the UASB reactor, together with some measurements of SCA and on-line measurements of pH, DO, effluent TOC and influent and effluent colour for the aerobic stage. The ability of these parameters to indicate process instability and efficiency, the reliability/maintenance of the instrumentation and delay in response were evaluated. The intention was to determine the most useful measurements and also useful remedial actions for process control.

4.1. Results from Experimental Phases 1 and 2

Results from Experimental Phase 1 have been reported elsewhere (Guwy *et al.*, 1997a). The data was collected under another project and was only used here for the purpose of selecting the type of the ANN appropriate to use in a control scheme for the textile WWT rig. The rig used by Guwy *et al.* (1997a) was an anaerobic treatment system with equivalent parameters being monitored to those used in this work. Therefore, it was thought while data was being collected for the textile laboratory scale plant, the fluidised bed reactor data could be used to start defining a suitable control scheme for biological treatment processes. The results gathered from Experimental Phase 2 will be described in Section 5.2 as they were used to train and test four different ANNBCSs in order to select the most appropriate. There will also be a comparison in Section 4.3.2 of the results acquired during Experiments 2.2 and 4.1, as the influent to the UASB reactor was the same.

4.2. Results from Experimental Phase 3

Figure 4.1 shows a photograph of the laboratory rig for Phase 3 with some of the instrumentation. During Experimental Phase 3 on-off control of pH and DO was performed on the aerobic stage (Section 3.1.1). The section of the LabVIEW™ VI diagram for on-off control is shown in Figure 4.2. Figure 4.3 shows the panel where graphs of the signals from the on-line monitors were displayed. The responses of the UASB reactor biogas flowrate, biogas CO_2 and H_2, pH and BA, its effluent TOD and colour and also pH and DO for the aerobic tank are discussed below. There is also a brief discussion of the influence of the temperature of the UASB reactor on its metabolism and hence biogas production and its $p\mathrm{H}_2$.

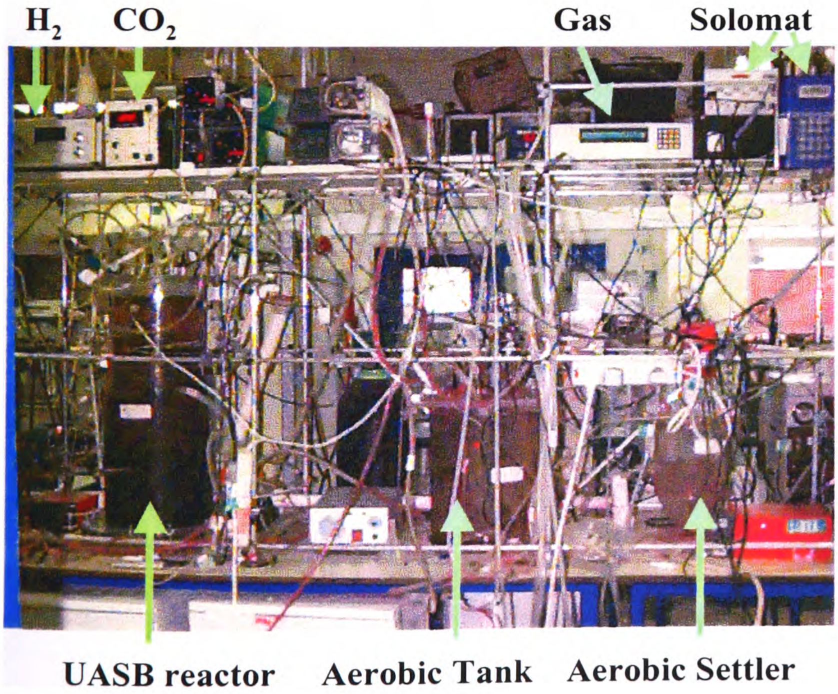


Figure 4.1 – Rig setup for Experimental Phase 3

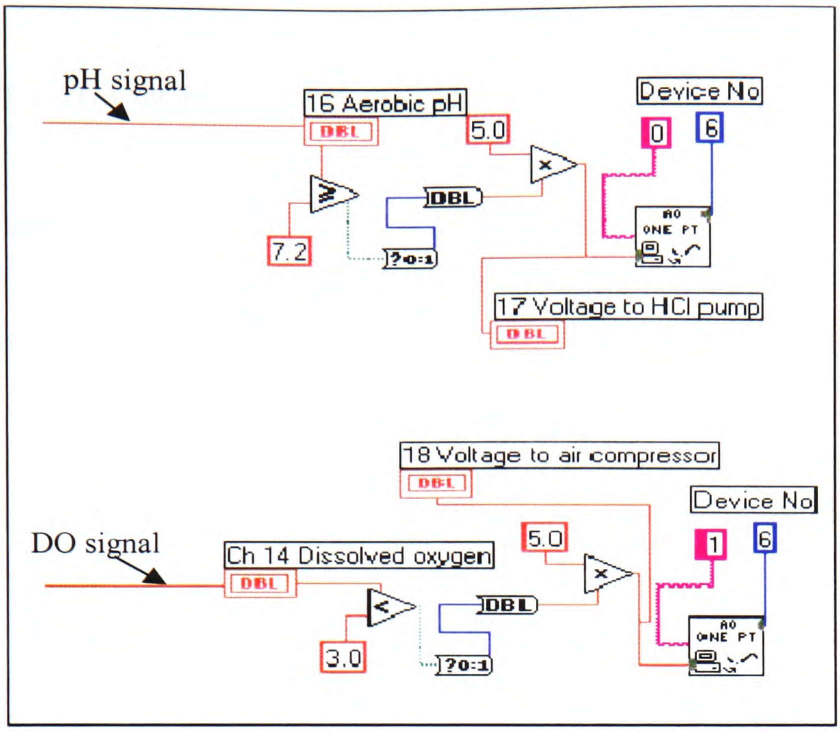


Figure 4.2 – Section of LabVIEW™ VI diagram for on-off control of pH and DO in the aerobic tank

Figure 4.3 – Photograph of the central computer screen showing a section of the LabVIEW™ VI Panel

4.2.1. UASB Reactor Effluent TOD

During operation on low starch, low dye (Experiment 3.1), the mean TOD of the UASB reactor effluent was 1049 mg l^{-1}. With an increase in starch concentration from low to high with a low dye concentration, the TOD of the UASB reactor effluent increased to a maximum of 2250 mg l^{-1}, which was then maintained (Figure 4.4). At medium starch, medium dye the TOD achieved was 1800 mg l^{-1}. The UASB reactor did not totally degrade the additional starch. Although most BOD was removed by the aerobic stage and effluent quality maintained, too high a starch level in the feed to the aerobic reactor could give rise to sludge bulking problems. The comparison of TOD and off-line COD measurements revealed a TOD:COD ratio of 1.4 (*sd* = 0.23 and n = 25) for the UASB reactor effluent similar to that found in other work (e.g. Ionics, 1993).

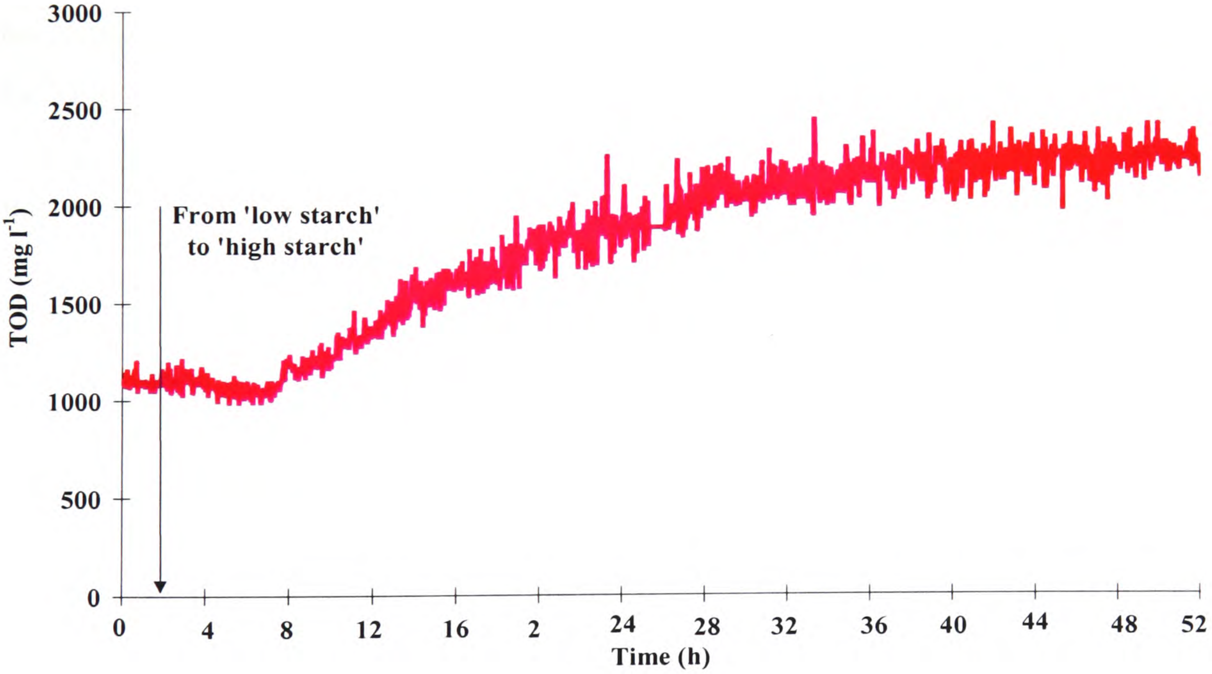
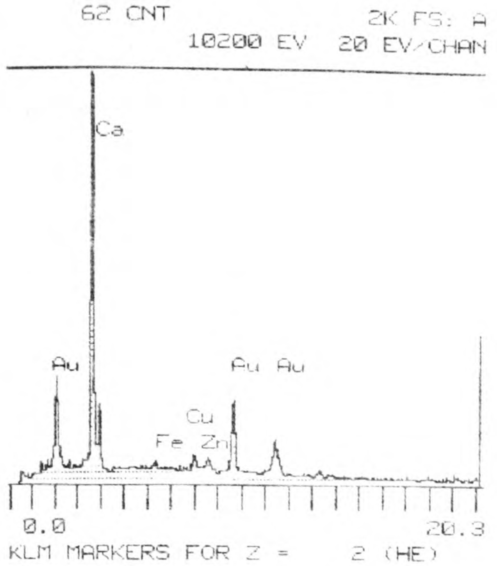


Figure 4.4 - Effect on UASB reactor effluent TOD of step-up from low to high starch at low dye

However, the TOD analyser did not operate reliably on the STE used here, due to constant blockages of the injection tube. The maximum time period between replacements was 90 hours, just over 3.5 days, however the normal time period was only 2 days. The injection tube was frequently replaced and the analyser calibrated and the different STE recipes did not appear to affect the formation of blockages. Figure 4.5 shows a X-ray analysis of a blocked injection tube (Section 3.3.5) and also a microphotograph of the same specimen. As it can be seen from the spectrum there were traces of Ca, Fe, Cu and Zn, with the Ca mineral content dominant. The probable origin of the Fe, Cu and Zn would be the trace element solution, which was added at 1 ml l^{-1} of the STE, which contained $FeSO_4.7H_2O$ (5 g l^{-1}), $ZnSO_4.7H_2O$ (0.011 g l^{-1}) and $CuSO_4.5H_2O$ (0.392 g l^{-1}). There were three possible origins of Ca: the water in the STE, the rinse water for the TOD monitor or most probably due to degradation of the UASB reactor black granules as the colour of the residue was black which resembled broken granules. Dubourguier *et al.* (1988) used X-ray analysis for observation of mineral precipitates of Ca and minor amounts of P or Fe and S. Thaveesri *et al.* (1995) found that measurements of co-enzyme 420 revealed that black granules consisted mainly of acetoclastic methanogens and they were particularly rich in Ca, Mn, and Zn minerals.

A few suggestions were made related to the problem encountered with the injection system of the TOD analyser apart from the high mineral content of the WW. These were: the use of 0.2 mm WW suspended solids, the tiny hole diameter of the tube (0.5 mm diameter) in conjunction with the use of a metallic ferrule around the injection tube which, at high temperature may have expanded and made the hole even smaller. The injection tubes were not able to be unblocked as their internal diameter was very small and with the high temperature from the oven the residue glued to the internal walls. Every change of the injection tube disrupted TOD monitoring for approximately 4 hours (i.e. cooling of the ovens, replacement of injection tube, warm-up of the ovens and calibration).

As it would be very difficult to reduce the calcium ions in this system, to diminish the blocking frequency a rinse cycle with a 5 % nitric acid solution made up with DI water and also a wider injection tube from the manufacturer were tried but to no avail. Even if the injection system was reliable the interference of ammonia, nitrates, chlorides and sulphates (Ionics, 1993) within the STE with the analyser measurements would have to be analysed thoroughly. Perhaps another organic strength analyser for example a TOC analyser could be used instead.



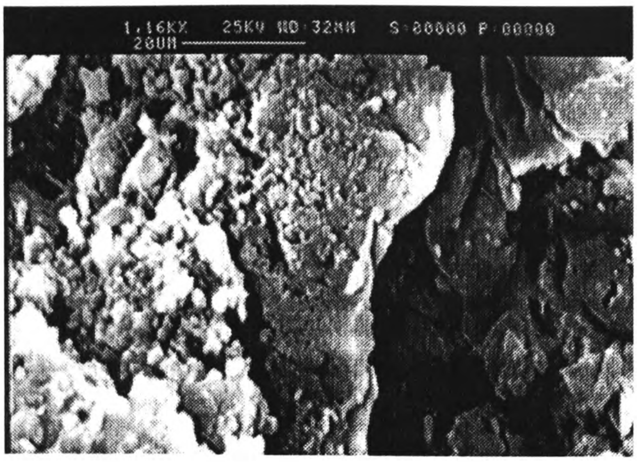


Figure 4.5 – Spectrum (left) and microphotography (right) of the TOD injection tube residue

4.2.2. Biogas flowrate, pCO_2 and pH_2

Both increases and decreases in starch concentration were always reflected in gas production and %CO_2 values. There was no noticeable effect on any of the biogas related measurements when the dye concentration was increased 5-fold in the feed. On changing from low to high starch concentrations at low dye concentration, the gas production increased from an average of 11.2 to an average of 16.5 ml min^{-1} with a maximum of 21 ml min^{-1} achieved 3 h after the step change in load. The % CO_2 increased from 26 to 32.5 % in 3 h. After a change from low starch, low dye to high starch, high dye, gas production increased from 15.2 ml min^{-1} to over 22 ml min^{-1}, 7 hours after the step change. This was similar to that achieved at high starch, low dye, suggesting the dye was not mineralised anaerobically. The concentration of CO_2 increased from 27 % to a maximum of 34 %. Biogas H_2 increased from values around 200 ppm to over 900 ppm after 29 hours from the step change in starch and dye (Experiment 3.4) and only decreased after 6 days when the feed was changed to low starch and low dye (Experiment 3.1). Therefore H_2 concentration was not only an event marker as claimed by various researchers (Mosey and Fernandes, 1989; Ehlinger *et al.*, 1994) but maintained its response during the event. Quite high concentrations of H_2 were achieved in the reactor biogas considering that in healthy, stable anaerobic digesters, very low pH_2 usually occur in the biogas. Collins and Paskins (1987) found that pH_2 in 20 UK sludge digesters operating with HRT of 8 – 20 days, varied between 15-199 ppm. However, quite high pH_2 were also found by Guwy *et al.* (1997a) using the GMI Instrument when operating 2 fluidised reactors on simulated baker's yeast WW (steady-state B_v = 27-33 COD m^{-3} d^{-1}) at 10.2 – 8.7 h HRT (the setup of Experimental Phase 1). They measured pH_2 levels from 290 to 640 ppm when increasing the B_v from 40 to 63 kg COD m^{-3} d^{-1}. They also found that switching from an older feed, partly-acidified, to a batch of fresh feed, with the same COD, promoted an increase in biogas H_2 content from 200 to 800 ppm, probably as the feed was only cooled to 13-15 °C. In the case of the textile project there was no pre-acidification of the STE as it was stored at 5 °C.

During operation on high dye, high starch a malfunction of the pump delivering concentrated feed to the UASB reactor occurred giving an irregular feed concentration (period A in Figures 4.6 and 4.7). The pump stopped completely during the period between the arrows (B) and restarted at the start of period C delivering high starch, high dye. The effects on biogas parameters are shown in Figure 4.6.

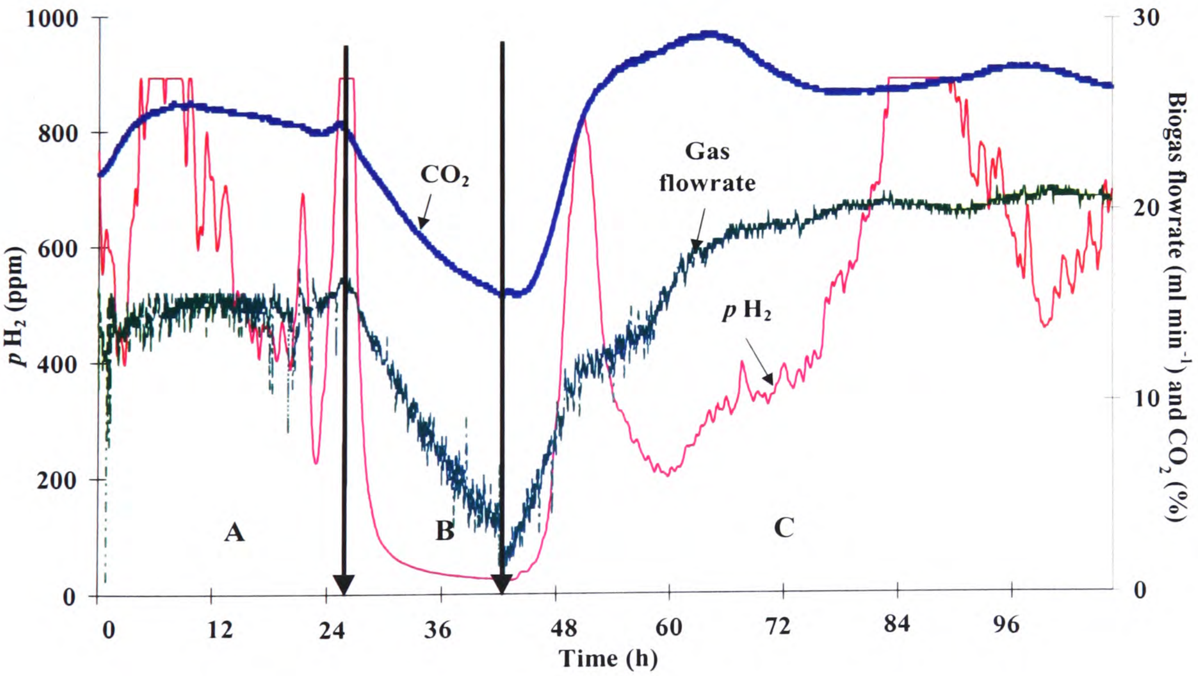


Figure 4.6 - Effects of changes in loading concentrations on UASB reactor gas production, CO_2, and H_2 biogas concentrations

As it can be seen in Figure 4.6 biogas flowrate and %CO_2 were good indicators of a decrease or increase in starch content in the concentrated feed. As high starch, high dye feed was started (start of C) the CO_2 concentration in the biogas increased to a maximum of 28 % and then stabilised at a lower value of 26 %. Hydrogen partial pressure decreased smoothly in B from a value over 900 to 24 ppm. According to traditional kinetic models, the H_2 concentration without loading should be zero. The reason for a residual pH_2 at a feed rate of zero was explained as a threshold value for H_2 consumption exists below which methanogenic bacteria are incapable of H_2 degradation (Cord-Ruwisch *et al.*, 1988) and is dependent on the energetic conditions (Hoh and Cord-Ruwisch, 1996). The H_2 increased sharply straight away when feeding re-started (start of C), and again 10 hours after the reactor was fed once more, corresponding this time to a minimum in pH (Figure 4.7) and a rise in %CO_2, possibly caused by build up of VFAs.

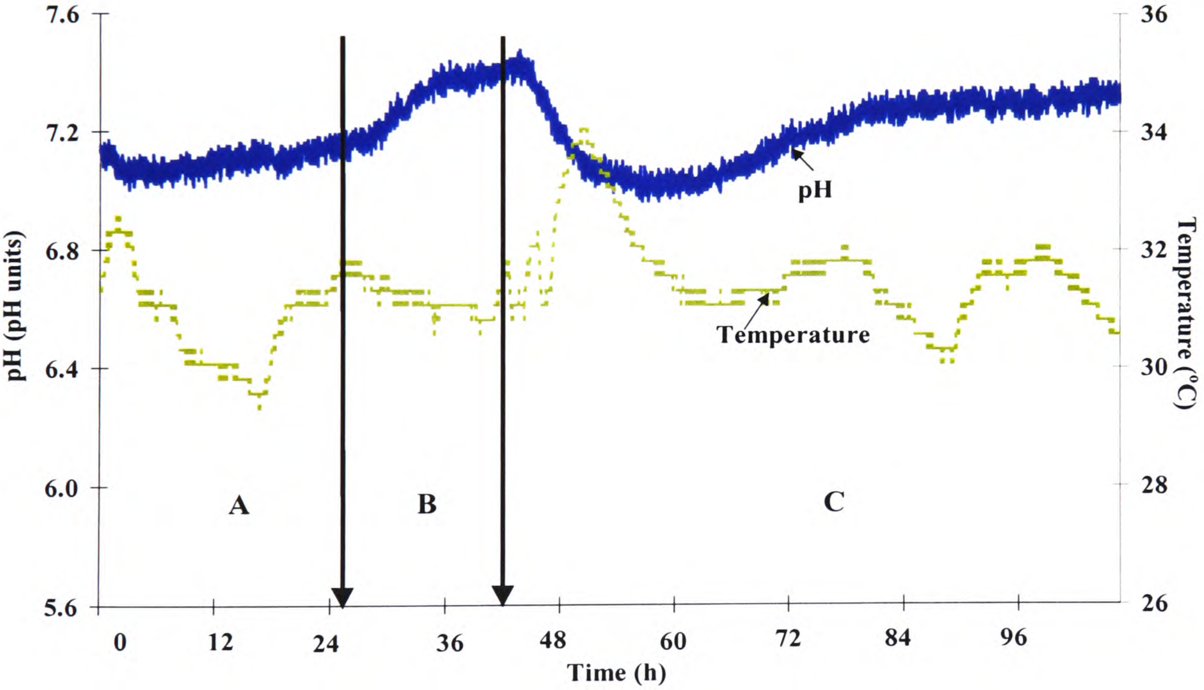


Figure 4.7 - Effects of changes in loading concentrations on UASB reactor pH

During Experiment 3.7, pH_2 went up to 300 ppm and total gas flow rate to 17.8 ml min^{-1}, %CO_2 rose to a maximum of 30 % but was maintained at an average of 26 %. At the medium starch loading rate the UASB reactor value for pCO_2 and pH_2 did not suggest that the reactor was as unstable as during the high starch loading rate. The level of H_2S in the

UASB reactor biogas at medium starch, medium dye was 0.7 % compared to 1.2 % during high starch, high dye. However, this value was below the toxic level of 2 % (Carliell *et al.*, 1996). The H_2S level of 1.2 % corresponds to 62 mg l^{-1} of total sulphide in the liquid phase (at pH 6.9 and 35 °C) (Speece, 1996). This value was below the inhibitory levels for anaerobic processes of 2 – 4 g l^{-1}. After Experiment 3.4 the solution inside the H_2S scrubber was black in colour of the CuS formed together with H_2SO_4 from the reaction of the copper sulphate with the H_2S in the biogas.

Normally, an increase in pH_2 occurred with an increase in starch, however, biogas H_2 levels appeared to vary also unpredictably. Mosey and Fernandes (1989), operating a 20 d HRT laboratory digester on reconstituted skimmed milk, showed that more than a ten-fold change in pH_2 (10 to 120 ppm) could occur without significant changes in bacterial performance. It has been previously suggested that this parameter is not suitable for stand-alone control (Guwy *et al.*, 1997a). Supporting this, Figure 4.8 shows a delayed response of biogas H_2 levels to reactor stress. $NaHCO_3$ was omitted from the feed (to simulate a BA pump failure) at the point shown, leading to a decrease in BA in the UASB to the lowest safe limit of 1000 mg l^{-1} as $CaCO_3$ and a fall in pH. There was an unexpected increase in biogas H_2 during this period, with the sharpest increase in biogas H_2 only after the BA started to rise. During this experiment the reactor acetic acid levels rose from 270 mg l^{-1} to a maximum of 443 mg l^{-1} at the end of the period without BA addition. Propionic or butyric acids did not increase. Biogas H_2 levels only started to fall 50 h after normal levels of BA addition had been established. Therefore, the increase in pH_2 in this case could not be associated with propionic acid as also mentioned by Guwy *et al.* (1997a).

Another simulation, this time of a spillage of acetic acid, and its effect on the UASB reactor can be observed with the addition of an extra 4500 mg l^{-1} of acetic acid to the feed for a 4.5 hour period are shown in Figure 4.9. At the start of the experiment the acetic acid measured in the UASB reactor was 320 mg l^{-1} and 5 hours later was only 450 mg l^{-1}. Propionic acid did not increase. An increase in UASB effluent TOD was observed 3 h from the start of the experiment, reaching a maximum after 14 h, coinciding with a minimum pH. An increase in both CO_2 and gas flowrate was observed. Off-line measurements showed only a slight decrease in BA from 1750 to 1500 mg $CaCO_3$ l^{-1} 10 h after the start of the experiment. During the 4.5 hours of the addition of extra acetic acid, pH_2 decreased from 540 ppm to values down to 50 ppm (Figure 4.9) and remained below 200 ppm for the next 36 hours. This decrease may have been due to the fact that acetate is mainly used by acetoclastic methanogens to produce CH_4 and CO_2. As CO_2 increased it stimulated CH_4 formation by the methanogens reducing the H_2 levels. During the step load of acetic acid, a drop in biogas H_2 content corresponded to a worsening of the UASB reactor effluent quality.

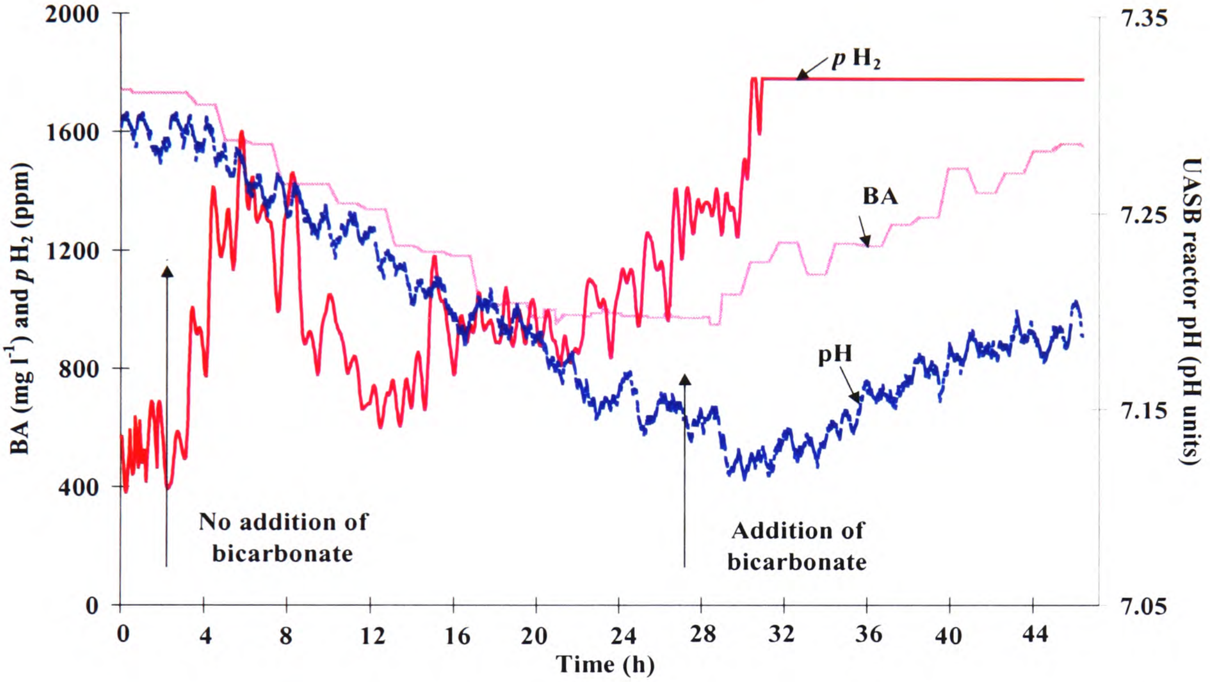


Figure 4.8 - Effects on gaseous H_2, pH and UASB reactor buffering capacity of BA deprivation and addition

Figure 4.10 shows that during Experiment 3.9 (between the arrows) where additional dyes were added (total dye concentration in the STE = 1.05 g l^{-1}), the biogas H_2 increased from 170 ppm to a maximum of 415 ppm during the 5 hours of the experiment. After this, biogas H_2 took 18 hours to return to the previously measured values. The off-line measurements of VFAs followed the H_2 trend although these only returned to the previous values after over 2 days from the end of Experiment 3.9. Increased H_2 levels inhibit the degradation of propionic acid and can also inhibit acetoclastic methanogenesis (Archer *et al.*, 1986). During this experiment the BA did not decrease significantly possibly due to the NaOH used in the hydrolysis process of the dye and the pH remained constant throughout. Both VFAs and biogas H_2 did not increase during Experiments 3.2 and 3.8 where the total dye concentration was 0.75 g l^{-1}. The rise in VFAs and biogas H_2 during Experiment 3.9 could be due to the higher dye concentration coupled with the non-acclimatisation of the UASB reactor biomass to the three extra dyes.

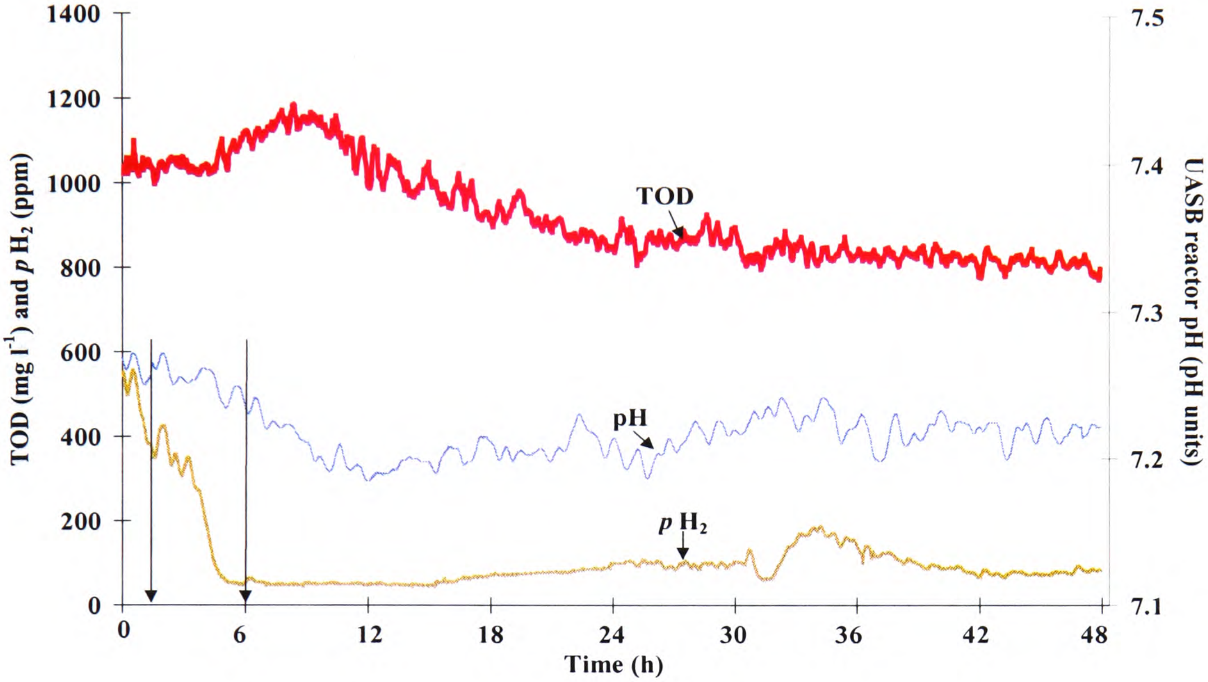


Figure 4.9 - Effects on UASB reactor pH, biogas H_2 and effluent TOD measurements of the addition of 4,500 mgl^{-1} acetic acid

Both VFAs and biogas H_2 increased after 2 hours from the start of Experiment 3.9. Therefore, an on-line VFA monitor could also be useful in predicting problems within the anaerobic process. A dissolved H_2 meter could possibly be more useful than the gaseous H_2 monitor because of the smaller H_2 mass-transfer coefficients in anaerobic digesters (Pauss and Guiot, 1993). This limits the rapidity with which an increase in H_2 concentration in the biomass can be detected in the gas phase of the digester, with the consequence that serious overloading may occur before raised H_2 concentrations in the gas phase are detected (Strong and Cord-Ruwisch, 1995). Therefore, the determination of dissolved H_2 concentration from the pH_2 using Bunsen's coefficient (0.017 cm^3 hydrogen gas dissolves in 1 cm^3 of water at 1 bar and 37 °C) can result in gross under-estimation of the actual dissolved H_2 content. Pauss *et al.* (1990) have shown that dissolved H_2 concentration deviated from gas phase equilibrium by a factor of 40 to 70. Kuroda *et al.* (1991) found that dissolved H_2

concentration was approximately 60-times higher than the theoretical calculated with biogas pH_2. However, as the variation of pH_2 correlated with the operating conditions of the reactor, biogas H_2 was found to be a useful measurement considering also that it was a cheaper and an easier measurement than in the bulk liquid. Also, by knowing the dissolved and the biogas hydrogen it might be possible to determine the hydrogen actually held within the cells, which could be quite an important parameter for detecting instability.

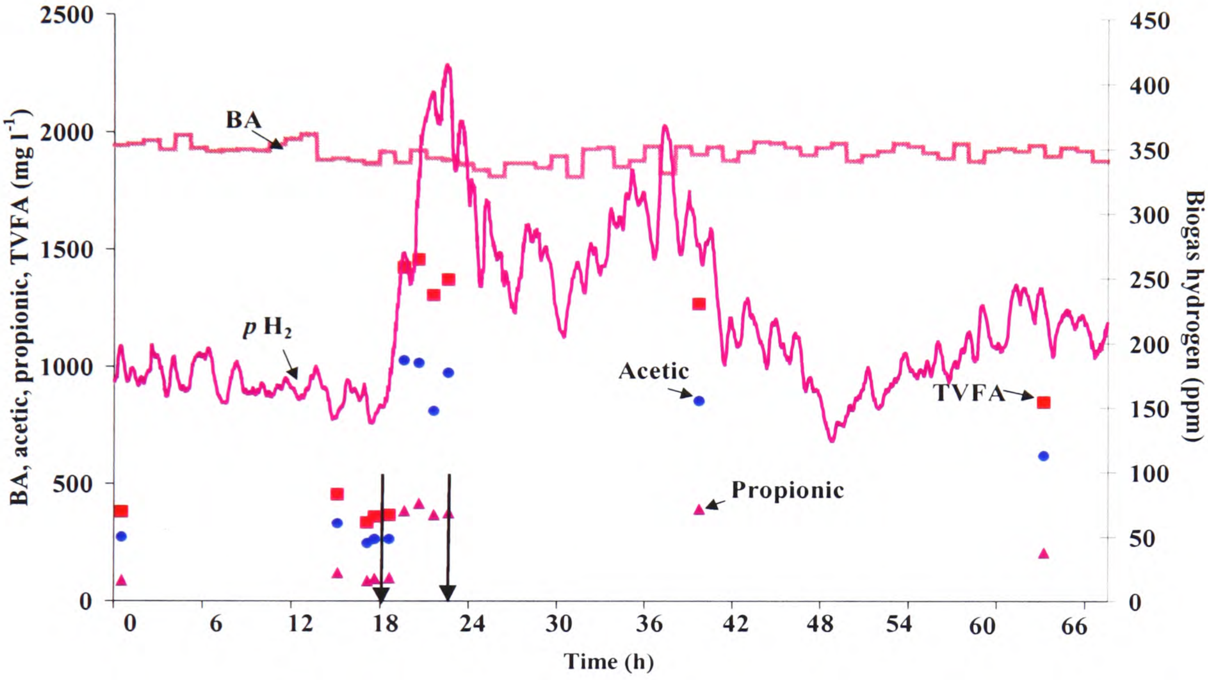


Figure 4.10 – Effects on BA, biogas H_2 and VFAs within the UASB reactor of Experiment 3.9

4.2.3. UASB Reactor BA and pH

The pH and BA values measured on-line reflected those measured off-line but on-line measurements were slightly lower for pH (by 0.1 - 0.2 pH units) and slightly higher (by 200 mg $CaCO_3$ l^{-1}) for BA. This was probably due to volatilisation of some VFAs and loss of CO_2 during off-line measurements.

Figure 4.7 shows that the UASB reactor pH, in this work, could be a good indicator of feed concentration changes. Figure 4.11 shows that the pH in the UASB reactor rose after a change in starch content from high to low starch at low dye concentration. This could be due to a decrease in VFAs within the UASB reactor. However, the rise in pH was then counteracted because there was less NaOH introduced in the feed at low starch concentration (NaOH was used in starch hydrolysis). Hence with this waste, pH alone would not necessarily reflect the health and efficiency of the UASB reactor.

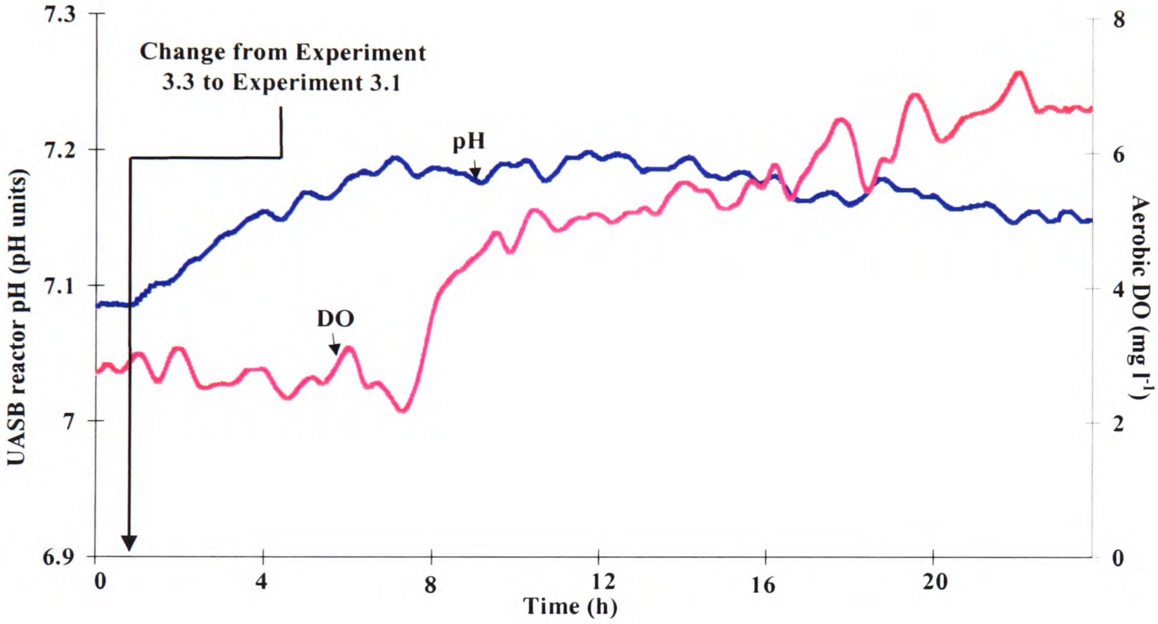


Figure 4.11 – Effects on the UASB reactor pH and aerobic vessel DO by decreasing the starch input from high to low starch at low dye concentration

On changing from low starch to high starch at low dye concentration, the pH and BA in the UASB reactor initially increased due to the higher NaOH level in the feed, but as VFA levels increased pH fell to between 7 and 7.2. The pH in the aerobic vessel also increased and the flowrate of neutralising HCl had to be increased manually to maintain the pH (Section 3.1.1). This demonstrated one of the weaknesses of a simple on-off control system, as there was need for control of the flowrate of the acid.

The intermittent BA monitor operated reliably almost continuously for 18 weeks. Occasionally values of BA were actually over the range of the instrument, or foaming occurred giving false readings (both situations occurred at high starch concentrations). Antifoam addition should be considered if this instrument was used in a control scheme. During the 'Initial Experiment' and Experiment 3.1 the BA values were between 1800 - 2000 mg $CaCO_3$ l^{-1}.

4.2.4. Aerobic Tank DO

DO in the aerobic tank was around 6 mg l^{-1} in the 'Initial Experiment' but increased to 8 - 9 mg l^{-1} (close to saturation) as the MLSS decreased due to a carbon source shortage in the influent to the aerobic stage. On conversion to high starch operation the MLSS increased and the DO levels decreased below 3 mg l^{-1}, automatically starting the second air compressor. At medium starch, medium dye with addition of OECD synthetic sewage an average MLSS of 2.4 g l^{-1} was maintained, with a DO of 6.5 mg l^{-1} with only 1 air compressor operating.

It was found that changes in DO readings were a good indicator of changes in the organic concentration reaching the aerobic vessel, information useful when optimising the complete system. However, absolute values of DO could not be used since it also varied with MLSS. In order to control the MLSS within the aerobic tank, by adjusting the RAS, measurements of biomass activity must be performed. Figure 4.11 shows the DO level in the activated sludge tank. Six hours after a step from high to low starch concentration the DO values rose due to the lower respiration rate promoted by the lack of an organic source. Reverse effects on DO were observed within 6 hours when starch concentration was increased to 3.8 g l^{-1}.

4.2.5. UASB Reactor Effluent Colour

Before Experiment 3.7 deposits were observed on the flowcell of the UV/Visible Spectrophotometer and the tubing walls within two hours. A microscopic examination showed these to be bacterial film (Figure 4.12). The deposits intensified after dilution of the sample from the UASB reactor effluent took place. This could indicate that before dilution the sample was quite toxic and avoided proliferation of the biofilm, perhaps due to amines by-products of azo dye degradation (O'Neill *et al.*, 2000b). All silicone tubing was changed to polyurethane tubing and a broad spectrum biocide (Panabath M, BDH Chemicals Ltd., Poole, UK) was added (in all the experiments after Experiment 3.6) at 0.25 ml l^{-1} made up with DI water, which prevented biofilm formation. At this dilution the biocide showed no absorbance at the 3 measured wavelengths, it did not alter the pH of the sample and did not cause additional foaming. At medium starch, medium dye the colour measurements were shifted by 0.83 absorbance units between the on-line and off-line measurements. For off-line measurements the sample was centrifuged rather than filtered through 185 μm mesh. This suggested turbidity in the on-line samples, requiring a smaller mesh, but physical problems in obtaining filtered samples at a sufficient rate at this scale prevented the use of a finer filter.

The shift of on-line measurements (Section 3.3.6) in relation to the off-line or true colour measurements (Section 3.2.3) can be seen in Figure 4.13, which records the effect of Experiment 3.9 with the addition of PROCION™ Blue, Yellow and Orange. The on-line OD measurements were processed using a 4th order low-pass Bessel filter to attenuate the effect of particles in the sample that occasionally passed through the filter. At the start of Experiment 3.9 the OD at 525 nm was higher than the average OD for 436, 525 and 620 nm, as expected since PROCION™ Red absorbed strongly around 525 nm. After the dye impulse, the average OD was considerably higher than the OD at 525 nm since the extra yellow, orange and blue dyes absorb strongly at 436 and 620 nm. One day after Experiment 3.9 the average OD was still rather high compared to the start of Experiment 3.9 whilst the absorbance at 525 nm was actually lower. This shows the importance of measuring the average OD at three wavelengths.

The TOD measurements followed a similar time evolution to the average OD measurements, achieving a maximum around 4 h (i.e. changing from 1650 mg l^{-1} at the start of the experiment to 1940 mg l^{-1} at 24 h) and then fell to 1700 mg l^{-1}. This increase in UASB reactor effluent TOD was not detected by the DO sensor in the aerobic tank.

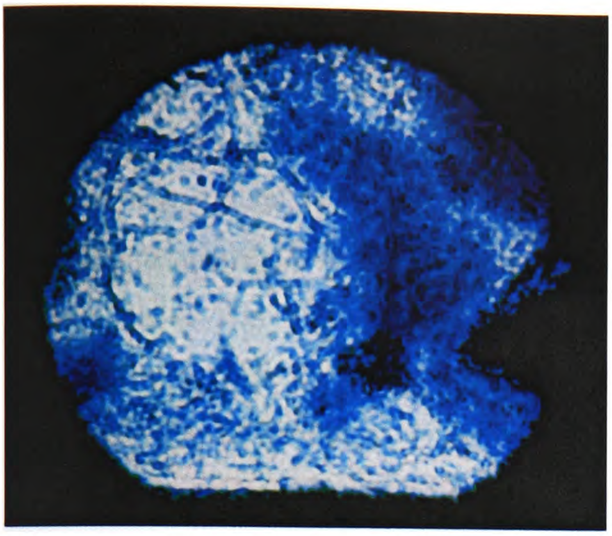


Figure 4.12 – Microscopic photograph of the stained sample of the flowcell residue (100x amplification)

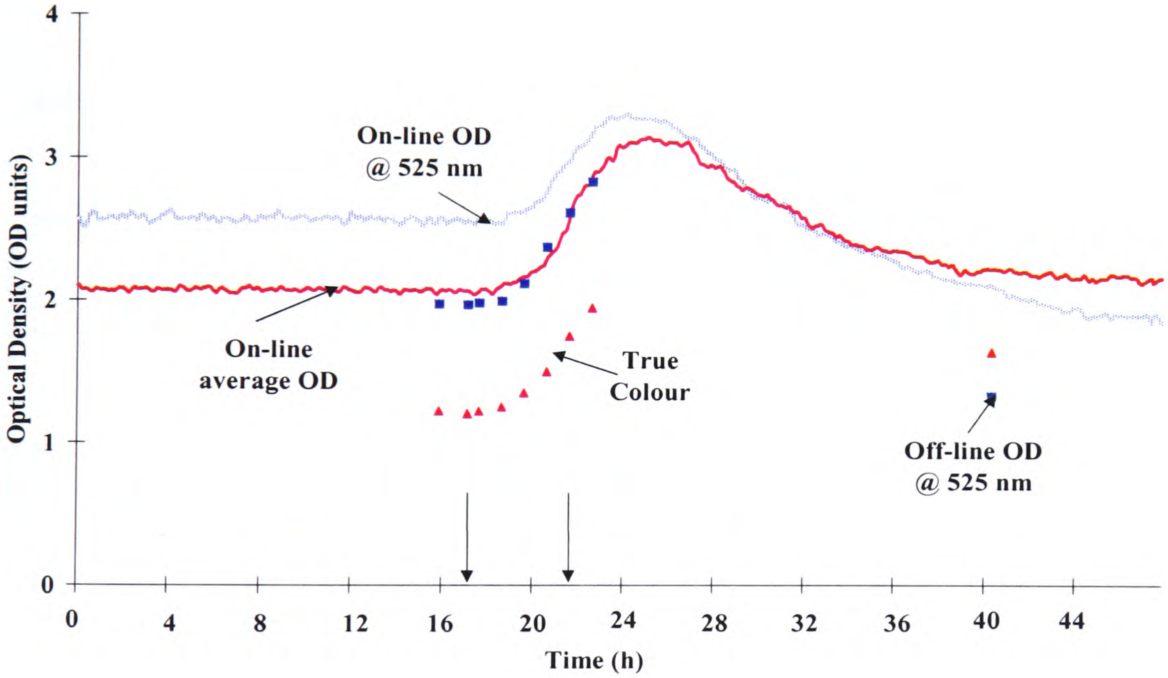


Figure 4.13 - On and off-line average OD and on and off-line OD at 525 nm

4.2.6. Influence of the UASB Reactor Temperature

It is well known that methanogens are more sensitive than acidogens in the anaerobic consortium, and unbalanced metabolism can occur at lower temperatures when acidogens produce VFAs faster than methanogens convert them to methane (Speece, 1996). During one week in the winter the laboratory heating system failed a few times and the flowheater for the

UASB reactor could not cope with the sudden changes in temperature. Figure 4.14 shows the effects of the lower temperature experienced by the UASB reactor and how it affected the gas production and the biogas H_2. During Experiment 3.1 the temperature decreased from 35 °C to a minimum of 31 °C over a period of 23 hours and as expected due to the decrease in the bacterial metabolism gas production was lowered and the %CO_2 in the biogas followed a similar trend. The TOD and colour of the effluent increased slightly during this lower temperature period. At the same time the H_2 in the biogas rose from an average of 150 ppm to a maximum of 860 ppm. As described in Section 4.2.2 the H_2 increased every time the starch concentration increased and this can also be observed in Figure 4.14. Both the hydrogen and gas meter saturated at the end of the period (Figure 4.14). Biogas H_2 was able to detect UASB reactor distress under these conditions.

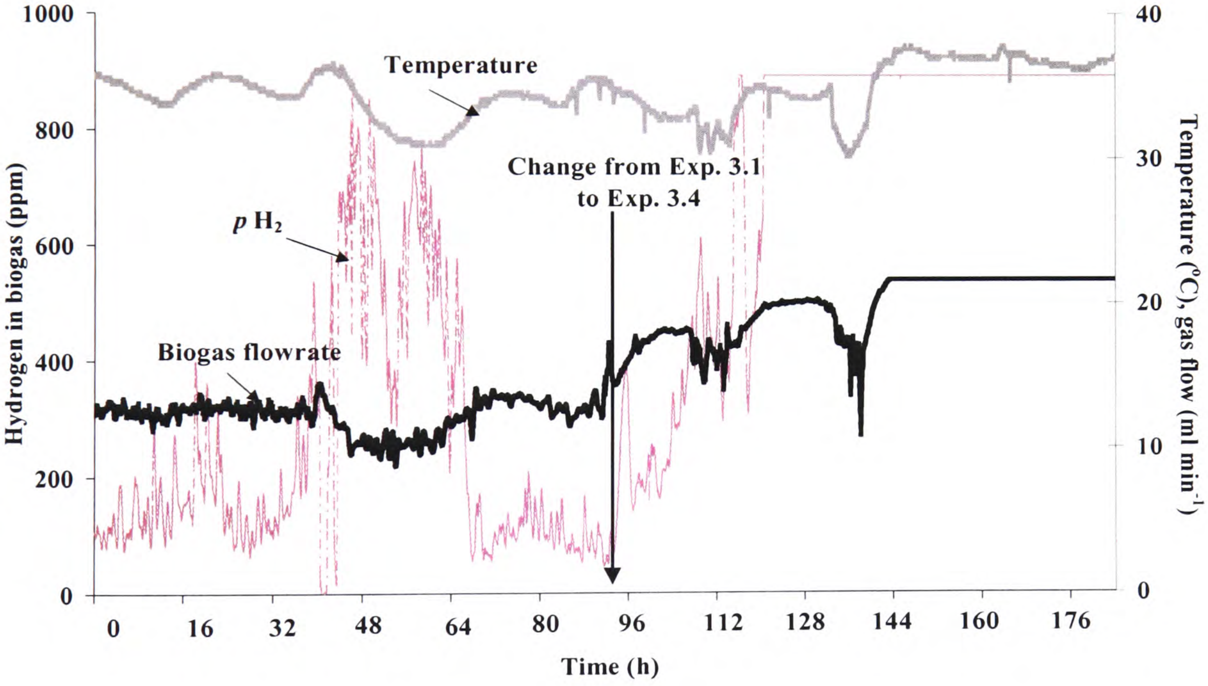


Figure 4.14 – Influence of the UASB reactor temperature on the biogas flowrate and pH_2

4.2.7. Discussion of Results and Conclusions from Experimental Phase 3

After a number of changes in operating conditions, the same performance parameters were obtained on returning to the conditions of the 'Initial Experiment' (except DO and MLSS).

Thus the process was not heavily time variant, making process control easier. BA had to be added to the STE to maintain buffering capacity, suggesting that BA control would be an advantage in industrial biotreatment plants operating on textile effluents. Off-line true colour measurements of the UASB effluent at the same dye concentration showed a decrease in colour at higher starch concentrations. The ratio of starch and dye concentrations must be optimised if good colour removal is to be achieved.

The on-line TOD instrument described may not be used with industrial wastes containing significant mineral content because of blockages. Another instrument for monitoring organic strength at the influent to the UASB reactor and at the effluent are a priority for RTC.

For a system experiencing organic overloads, %CO_2 and gas flow rate are obvious control parameters, with a quick (approximately 1 hour) and in some cases quantitative response. CO_2 levels above 28 % were found to correlate with conditions of anaerobic reactor instability. An on-line gas meter may need re-calibration only yearly and a CO_2 monitor monthly. Both should preferably have a moisture removing step. Biogas hydrogen monitoring has a fast response, and correlated always with a change in the organic loading rate to the methanogenic reactor, however it varied sometimes unpredictably. BA monitoring in anaerobic digesters indicates changes in organic load within approximately 5 hours, in some cases faster than the pH response. However, in the case of this type of industrial waste could also signal changes in NaOH or other parameters (e.g. organic nitrogen) that can lead to changes in the bicarbonate concentration.

Changes in DO in the region above the control set point may quickly indicate a rise in organic load. pH control in an aerobic stage which follows an anaerobic stage operating on waste containing varying NaOH levels may demand more than a set point controller; a proportional controller would be appropriate. A method for colour measurement is a priority for textile effluents. Colour should be measured on-line in a well-filtered sample at several wavelengths, using a suitable biocide to counter bacterial fouling. A colour measurement can be used in the final effluent to warn of breaches of colour discharge consents, but a monitor of colour up-stream of the anaerobic stage is also advised in order to take fast remedial actions.

In order to operate and effectively control a biological treatment process a number of parameters must be monitored, providing complementary information and redundancy in case of instrument failure. Fast remedial actions must be available in the case of inefficient treatment or process instability. Examples of remedial actions are: addition of a buffering chemical; influent dilution/diversion; and effluent recycle for re-treatment. In the case of low organics and high azo dye concentrations in the feed, addition of a carbohydrate electron source for azo bond breakage to the anaerobic stage would be recommended.

4.3. Results from Experiment 4.1

This section describes briefly the UASB reactor 'health' condition after Experimental Phase 3 and compares its performance during Experiments 2.2 and 4.1. Finally, the difference between off-line or true colour and on-line colour measurements is further analysed. The apparatus, instrumentation and control setup for Phase 4 were presented in Section 3.6.4.

4.3.1. 'Health' Condition of the UASB Reactor

After having performed Experimental Phase 3, the UASB reactor was left to operate at steady state for 2 weeks at medium starch and dye concentrations (Experiment 3.7). The bed granules had decreased from Experimental Phase 2 as the granules had risen and found their way through the effluent port. At the end of the two weeks they started to stick together and floated to the top of the UASB reactor. After this period, the gas flowrate was inconsistent due to the granules floating, the temperature of the UASB reactor was erroneous as there were only a few paths between the thick layer of granules chosen by the warmed effluent and they did not pass through the temperature probe. The TVFA within the reactor went from an average of 380 mg l^{-1} to almost 1500 mg l^{-1} of which 500 mg l^{-1} was propionic acid. The pH decreased to 6.9, %CO_2 increased from about 26 % to 31 %, the COD reduction decreased to 20 % from an average of 68 %, and there was only 18 % of colour removal from a previous obtained value of 55 %.

A few attempts to recover its health were performed over a 2 months period: granules were stirred to aid gas release; the reactor effluent was recirculated to cause induce mechanical stress (Kosaric *et al.*, 1990); granules were washed with water in order to get ride of polysaccharides (Shen *et al.*, 1993); the HRT was increased from 1 to 2 days; no salt and dye were included in the STE to reduce possible toxicity and only 1.45 g l^{-1} of starch was used which was hydrolysed with amylase (Section 3.1.2) instead of NaOH also to avoid possible toxicity; $NaHCO_3$ was added up to 3.5 g l^{-1}; extra trace element solution was added (Speece, 1996); the UASB reactor was fed only with water and bicarbonate for 3 times for a period of a few days so that any of the effects of an organic or toxic load would be washed out.

None of these recovery attempts were completely successful enough to be able to operate at a 1 d HRT with the loading conditions of Experiment 3.7. Every time this was tried the gas flowrate decreased and was very irregular due to the floating granules, the CO_2 increased to almost 40 %, the TVFA increased to 740 - 950 mg l^{-1} and pH decreased to 6.8, all indicative of reactor instability. Therefore, the STE for Experiment 4.1 was chosen to be the same as Experiment 2.2 (0.95 g l^{-1} starch and 0.075 g l^{-1} dye concentrations at a 1 d HRT, the lowest organic and dye concentrations used). With this STE the reactor seemed to be stable. The on-line controlled Experiment 4.3 included the ANNBCS controlling a dye step load and the addition of BA. The results of this Experiment are presented in Section 5.3.1.

Some researchers have reported sudden granule disintegration without any obvious reason (Schmidt and Ahring, 1996). However, the floating of granules was thought to have occurred due to one or a combination of three main factors already debated in the literature:

- formation of bacterial aggregates in the UASB reactor because of the production of an external layer of extracellular polymers (Shen *et al.*, 1993).
- the granules within the UASB reactor were black and some white in colour, which according to Thaveesri *et al.* (1995) were susceptible to flotation due to biogas adhering on the surface. According to the authors, grey granules were found to be less susceptible to flotation.
- trapped gas within the granules in a hollow core. SEM examination revealed the presence of cavities inside large-sized granules (∅ > 1 mm) (Hickey *et al.*, 1991). The origin of the hole has been widely debated:

- due to starvation of granules and partial autolisation of biomass (Kosaric *et al.*, 1990). They speculated that if the feed penetrated the granules by diffusion, then when the size of the granule increased beyond a certain limit the concentration of feed in the centre was too small to feed the bacteria resulting in the starvation of the microbes and their subsequent autolysis.
- after a rapid change in load, granules lose their ability to settle and float to the top of the reactor (Blaszczyk *et al.*, 1994). The experiment conducted Blaszczyk and co-workers consisted of a low pH (from 7.5 to 4.7), low temperature (21 °C), high loading rate (from 600 to 1600 mg TOC l^{-1}) and a tenfold increase in sulphate concentration (30 to 300 mg l^{-1}), over a 2 day period. They theorised that the high sulphate concentration decreased the concentration of Ca and other divalent cations, which created links between the bacterial walls and thus improved the settling ability of the granules. This may have occurred with the granules in the reactor, in the work presented here, since the injection tube of the TOD analyser kept blocking due to the Ca content during Phase 3.

4.3.2. Comparison of the UASB Reactor Performance During Experiment 4.1 and Experiment 2.2

The 'Initial Experiment' and the repeats of Experiment 3.1 demonstrated that the UASB reactor performance did not vary greatly during Phase 3, which indicated that control should be easier. However, Table 4.1 shows the comparison of the UASB reactor results from Experiment 2.2 and Experiment 4.1. Both Experiments were performed using the same starch and dye concentrations (i.e. 0.95 g l^{-1} and 0.075 g l^{-1}, respectively). Table 4.1 shows a decrease in the UASB reactor performance during Phase 4, which was a result of a lower biomass concentration within the UASB reactor (decrement of 78 % in TS and VS concentration, and the volume of granules was decreased from 10 to 6 litres) and also due to the health of the granules. Some of the on-line monitoring results will be presented in Section 5.3.1. For Phase 4 on-line measurements of TOD were replaced by TOC measurements. The central computer via the LabVIEW™ VI shown in Figure 4.15 logged TOC measurements. How this VI was linked to the analyser and then to the central computer was explained in Section 3.5.1. The monitoring results were similar between the two repeats of Experiment 4.1.

Table 4.1- UASB reactor monitored parameters (Experiment 2.2 vs. Experiment 4.1)

Parameters	Experiment 2.2	Experiment 4.1
Biogas production (ml min^{-1})	6.7	4.2
CO_2 (%)	25	24
pH	7.1	7.5
BA (mg l^{-1})	1590	1910
STE - VFAs (mg l^{-1})	746	724
UASB reactor - VFAs (mg l^{-1})	Total = 360 Acetic = 335 (*sd* = 32)^1 Propionic = 9 (*sd* = 2)^1	Total = 470 Acetic = 446 (*sd* = 45)^2 Propionic = 12 (*sd* = 4)^2
STE – COD (mg l^{-1})	1610 (*sd* = 120)^1	1409 (*sd* = 96)^2
UASB reactor eff. COD (mg l^{-1})	695 (*sd* = 27)^1	866 (*sd* = 34)^2
COD reduction (%)	57	39
STE – Colour (TCU)	0.6 (*sd* = 0.03)^1	0.6 (*sd* = 0.02)^2
UASB reactor eff. colour (TCU)	0.24 (*sd* = 0.01)^1	0.27 (*sd* = 0.01)^2
Colour reduction (%)	60	55

Note - ^{1} number of samples = 3; ^{2} number of samples = 4. In cases where the values do not present a *sd*, they were monitored on-line and they represent the average of the samples during 2 weeks.

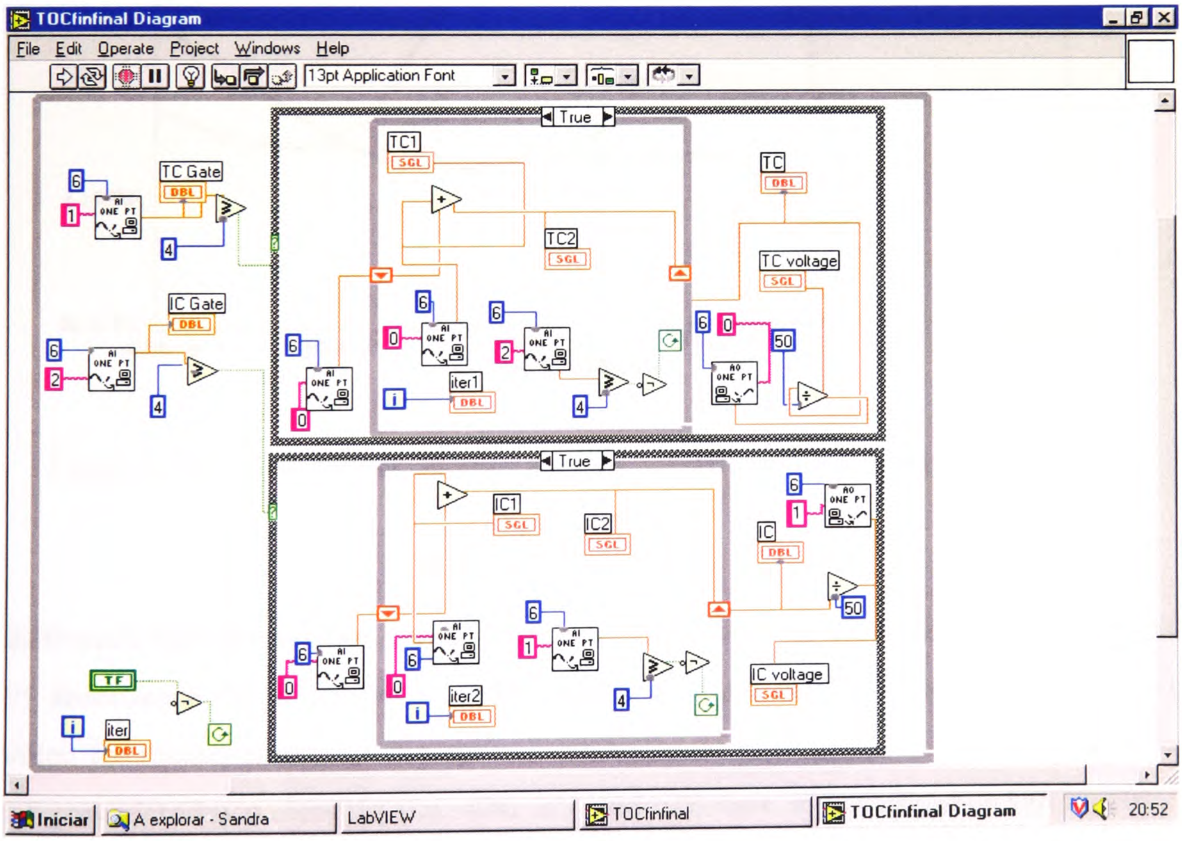


Figure 4.15 – LabVIEW™ VI code for TOC analyser data acquisition

4.3.3. Difference Between Off-line and On-line Colour Measurements

As the UV/Visible Spectrophotometer would be on-line during Phase 4 and the accuracy of measurements was a priority for the ANNBCS performance the difference between the results off and on-line was studied here in depth. Figure 4.16 shows spectrums of the UASB reactor influent and effluent during Experiment 4.1. The dye degradation effect can be noticed, as there was an absorbance decrease only for some part of the visible spectrum. If the colour reduction was felt throughout then it could have been mainly due to adsorption.

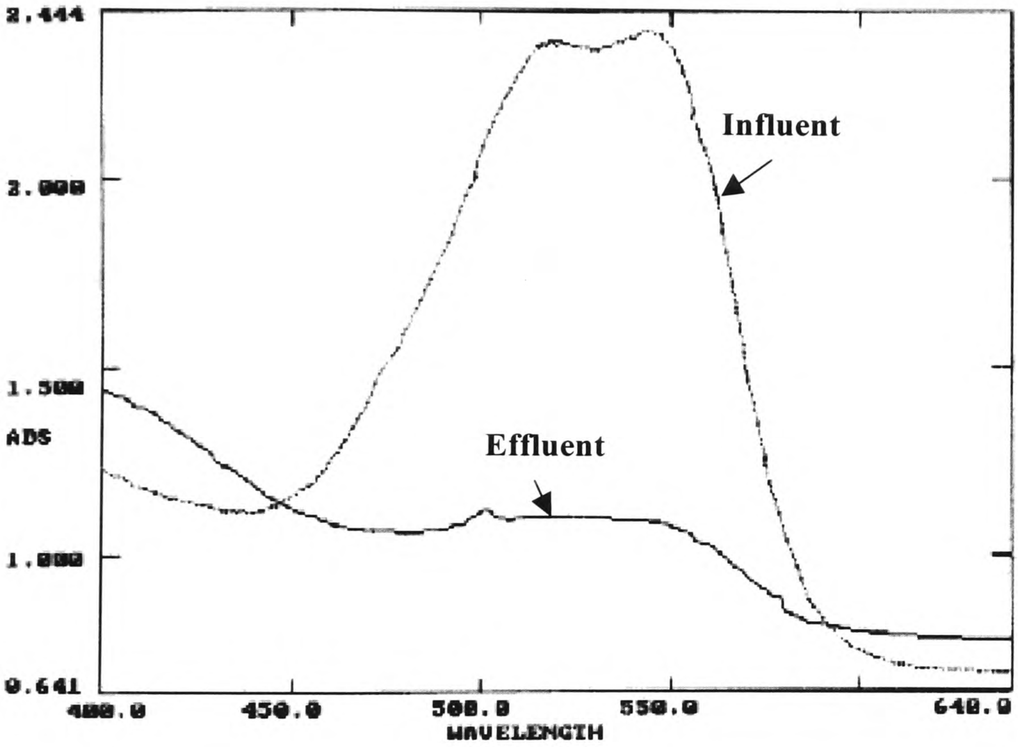


Figure 4.16 – Absorbance spectrum of the UASB reactor influent and effluent (Experiment 4.1)

The difference between on-line and off-line measurements was found to be 0.83 (*sd* = 0.003, n = 9) absorbance units, for both UASB reactor influent and effluent. The n in this case accounted for measurements in 3 repeated spectrums of Experiment 4.1 and the absorbance at the 3 considered wavelengths (i.e. 436, 525 and 620 nm). This difference can be observed in Figures 4.17 and 4.18. The off-line measurements were performed with centrifuged samples and measured with the cell (Section 3.2.3). Also measurements were performed using the flowcell and a manually filtered sample using a 185 μm mesh (Industrial Fabrics -

Sericol Ltd., Broadstairs, UK) in which case there was no effect due to the flow, as the sample was placed statically in the flowcell. However, the flow effect did not seem to have affected the readings when comparing the off-line and on-line measurements (Figure 4.13) during Experiment 3.9 (the difference was 0.83 absorbance units). Therefore, in order to try to keep on-line colour measurements the same as the true colour (i.e. acquired off-line) a tighter mesh was used during Phase 4 except the first 3 days of Experiment 4.1. The mesh had an aperture of 60 μm supplied also by Industrial Fabrics - Sericol Ltd. (Broadstairs, UK), and it was used with the self-cleaned cross-filter (Filter 3) (Section 3.4). The results for Experiment 4.2 will be shown in Section 5.3.1, as the UASB reactor was controlled by the ANNBCS.

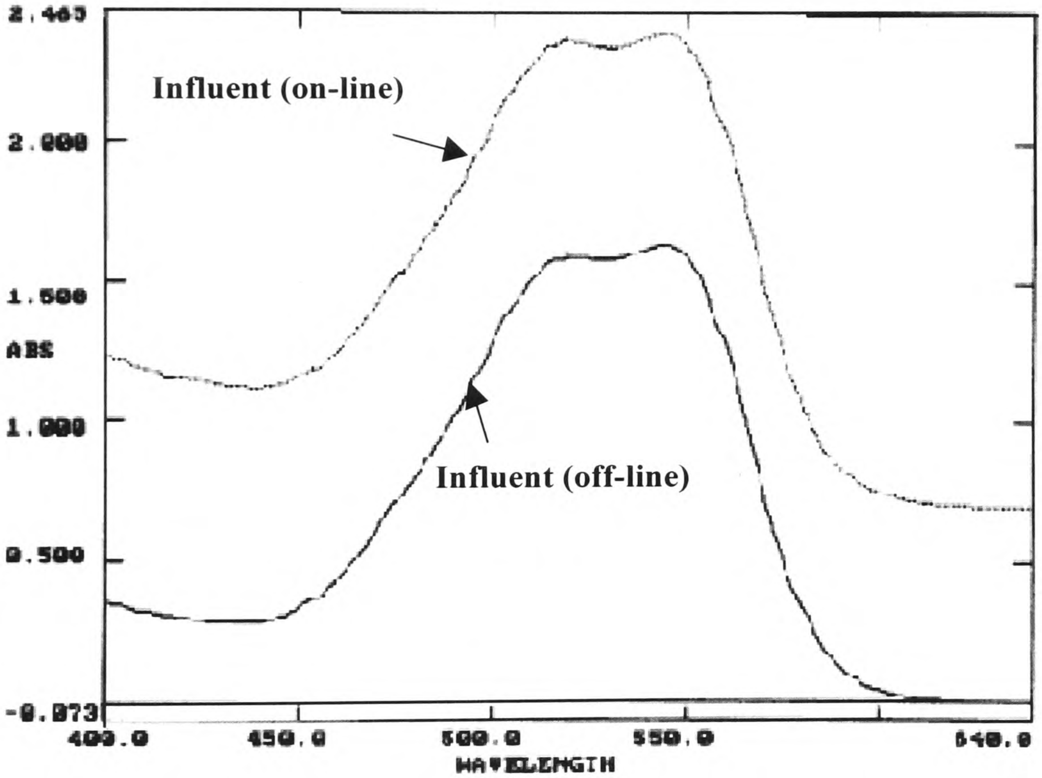


Figure 4.17 – Comparison between the spectrum of the UASB reactor influent on-line and off-line (Experiment 4.1)

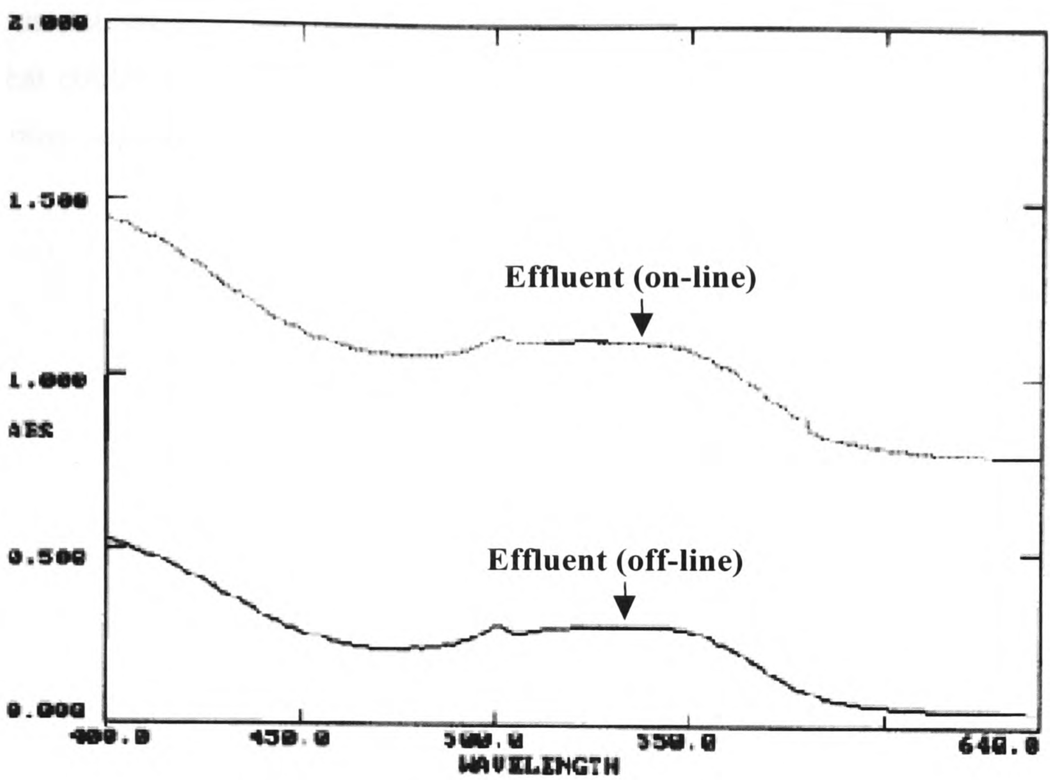


Figure 4.18 - Comparison between the spectrum of the UASB reactor effluent on-line and off-line (Experiment 4.1)

4.3.4. Conclusions from Experiment 4.1

A constant B_v of 3.14 g COD l^{-1} reactor d^{-1} (medium starch, medium dye) during a period of 1.5 months with no lower loading rates in between may have caused some 'distress' to the UASB reactor biomass, not noticed by the performed on-line measurements.

The UASB reactor demonstrated to be a time varying process due to instability and reducing biomass, two situations which must be avoided by a control scheme and should result in a less time varying system. A control scheme based on all the on-line sensors used for the UASB reactor and even from the off-line analyses (e.g. VFAs and H_2S) could have not anticipated the problems, which occurred with the reactor granules. An on-line monitor that could measure the biomass within the reactor or the rate of granules loss could have probably indicated that something adverse was happening. Over 2 months were spent trying to bring the biomass left in the reactor to a healthy state, this meant that if this would have happened in an industrial site it would have been catastrophic. Most of all, there were no remedial actions that could be taken that could tackle straight away the floating problem. Therefore, even the most sophisticated control scheme would have had the same difficulty.

Temperature within the UASB reactor must be maintained at all times to avoid changes in metabolism. It would be very important to measure it on-line in different locations of the reactor if the measurements would be connected to a close control loop for the UASB reactor. Temperature sensors may fail or provide erroneous information.

The sample for on-line colour measurements must be filtered through a tighter mesh than 185 μm (possibly 60 μm), otherwise a difference of 0.83 absorbance units would occur between on-line and off-line (i.e. true colour) measurements. The same difference was found for the influent and effluent samples of the UASB reactor.

4.4. Results from Experiments 5.1 and 5.2

In order to test the ability of the ANNBCS to control the aerobic stage, an experiment was undertaken where the concentrated STE was diverted to the aerobic stage without any pre-treatment by the UASB reactor. Data was collected from Experiments 5.1 and 5.2 to allow training of the ANNBCS for these particular situations. On-off control of pH and DO was performed during these two experiments (Section 3.1.1, Figure 4.2).

The experiments consisted of a step increase in the STE starch concentration from 1.5 g l^{-1} to 5.5 g l^{-1} and a step decrease to 1.5 g l^{-1} (Section 3.6.5). There were no advantages in trying to perform a colour step load as there was no dye degradation in the aerobic stage without prior anaerobic treatment. Throughout the experiment, parameters such as aerobic stage effluent TOC, pH and DO in the aerobic vessel and colour of the STE were monitored on-line. Off-line measurements of the colour and COD were performed on the STE and at the outlet of the aerobic stage. The on-line average OD was measured only in the STE to evaluate the effect of pH changes in the sample on this parameter as the extra starch was hydrolysed with amylase and the pH was adjusted to roughly 6 pH units. In addition, off-line measurements of SCA were also carried out.

4.4.1. Aerobic Stage Effluent TOC and Aerobic Tank DO and pH

The changes in the effluent TOC of the aerobic stage and pH within the aerobic tank during the step load and recovery period can be seen in Figure 4.19. As a result of the starch impulse loading (arrows indicate 'Start' and 'End' of the impulse) there was a sharp decrease in effluent treatment efficiency and therefore, there would be a need to control the biological process when similar situations occur in practice. The TOC level rose to 720 ppm in 7 hours and decreased to the previous level of 110 ppm 23 hours after the starch load was reduced.

Figure 4.20 displays the response of DO and corresponding air compressor voltage provided by the on-off controller constructed in LabVIEW™ (Section 3.1.1). The DO signal has been filtered to attenuate noise resulting from large bubbles of air impacting on the DO probe.

It can be seen that the pH and DO measurements responded quicker than TOC for each of the organic load changes (i.e. step up and down in starch concentration). This was because the two electrode measurements were performed *in situ* i.e. within the aerobic tank, whilst TOC samples experienced a time lag of approximately 16 minutes while measurements were made. Moreover, the measurement of pH and DO were taken from the aerobic tank (16 h HRT) whilst the TOC sample was taken from the top of the settler (and extra 3 h added to the 16 h of HRT). Another reason for the delay in the TOC response was that the aerobic bacteria, just after the load change were very active and therefore able to cope with the new load until the F:M ratio was too high for further degradation. It can also be observed that both DO and pH levels decreased after the load increase and started to rise again with the same time lag of 1.5 hours after the load returned to its original level (Figures 4.19 and 4.20). The decrease in DO levels supports the increase in bacterial activity. The two most probable reasons for the decrease in the pH were that:

- the starch was hydrolysed with amylase instead of NaOH and in order to stop the reaction the pH was lowered to 2. However, the solution was pH adjusted with NaOH, the pH was not quite at a neutral condition (i.e. pH ≈ 6);
- a build up of long chain acids in the aerobic tank since the anaerobic stage was not degrading all the starch in the STE.

The pH in the aeration tank rose after the end of Experiment 5.2 to values slightly above normal (i.e. from 7 to 7.16). When the organic load increased the DO reduced from an average of 6.2 to 3.2 mg l^{-1} and the extra compressor was switched on as shown by the voltage trace (the unfiltered values of DO went below 3 mg l^{-1}). After the end of the impulse the DO did not return to the previous average of 6.2 mg l^{-1}, but instead was maintained around 4.4 mg l^{-1}. This possibly occurred because of biomass growth and an increase in biomass activity resulting in a higher oxygen demand. Measurement of the solids concentration and also the SCA made before, during and after the starch impulse indicated that this was the case.

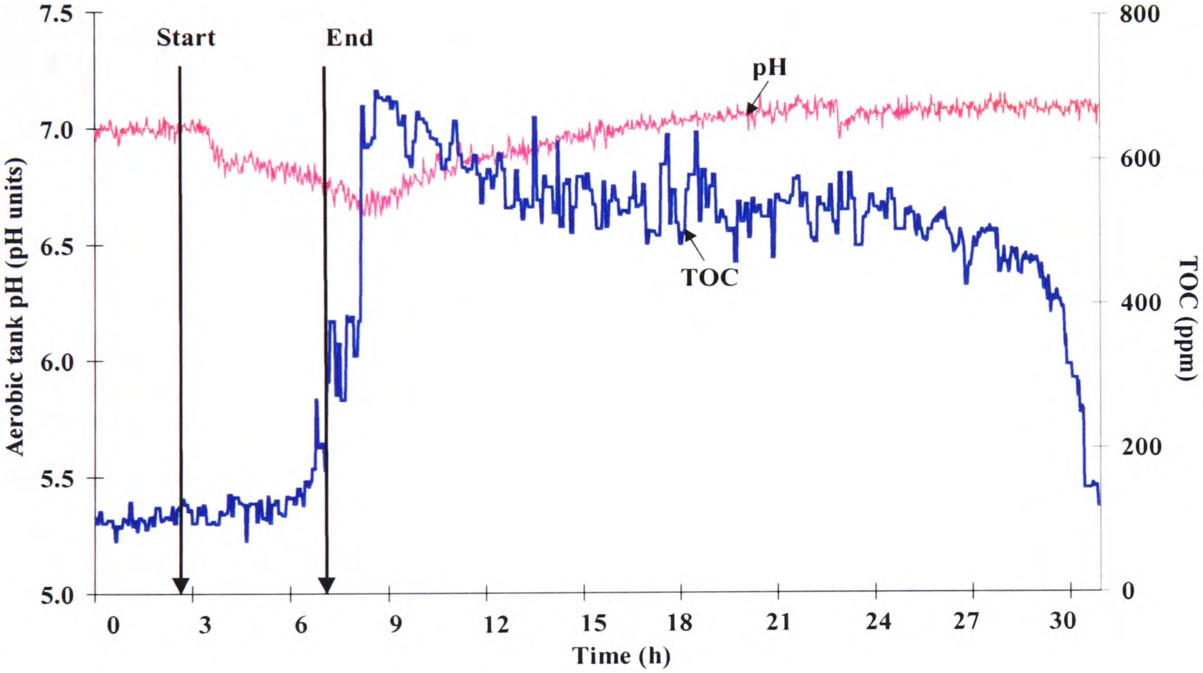


Figure 4.19 - TOC of the effluent of the aerobic settler and pH within the aerobic tank (Experiments 5.1 and 5.2)

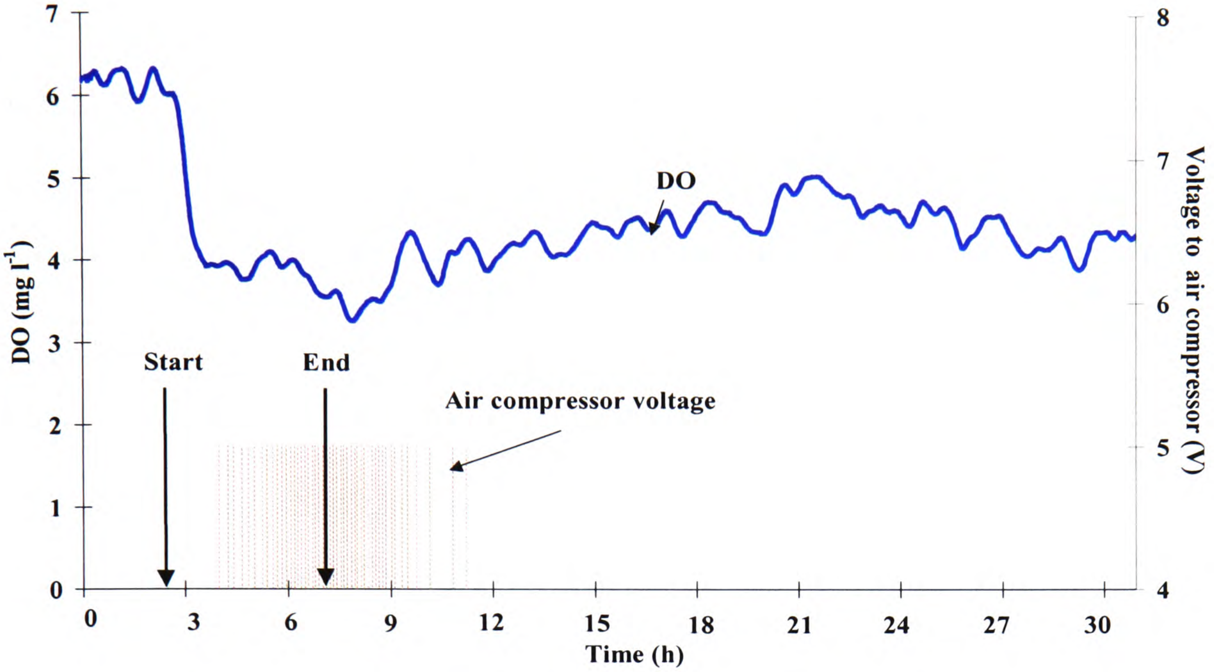


Figure 4.20 - DO within the aerobic tank and air compressor voltage (Experiments 5.1 and 5.2)

DO proved to respond very quickly although it was concluded that it could not be used to control the flow of additional starch, or the RAS (Section 4.4.2). However, the regulation of the pH and DO parameters were vital in order to ensure that good treatment conditions prevailed. The control of the additional starch flow was thought to be best achieved using the TOC measurements. These were performed on-line and responded 3 h from the start of the starch step load (Section 5.3.2). However they did not indicate how biodegradable the effluent was or any information on the biomass concentration and activity.

4.4.2. Aerobic Tank Solids and Biomass Catalase Activity

As illustrated in Section 3.2.9, SCA was calculated based on two variables: the oxygen evolved which was measured using the LFM 300 gas meter within the semi-automated novel biomass meter (Guwy *et al.,* 1998) and the VSS of the activated sludge samples which were determined off-line (APHA, 1995). Sludge samples were taken from the aerobic tank, namely 30 ml for measuring MLSS and VSS and 150 ml for measuring biomass activity.

Table 4.2 shows that both MLSS and VSS increased after the starch step impulse. The MLSS:VSS ratio was 1.14 (*sd* = 0.0092, n = 6 (6x each triplicate)) and the SCA results were obtained based on the VSS. This demonstrated that not only was there biomass growth but its specific activity had also increased during the starch impulse. Although, the VSS decreased slightly in the 4th measurement, the specific biomass activity was the highest. The SCA started to decrease by the 5th measurement.

Table 4.2 - MLSS, VSS and SCA recorded during Experiments 5.1 and 5.2

Samples	**MLSS (g l^{-1})**	**VSS (g l^{-1})**	**SCA (catalase units g^{-1} VSS)**
1	2.42	2.13	8,240
2	2.43	2.15	8,285
3	2.96	2.57	10,541
4	2.78	2.44	14,203
5	2.92	2.59	12,045
6	2.76	2.41	11,710

Note: Both MLSS and VSS were the average of the triplicates.

Figure 4.21 shows a plot of the SCA against TOC measurements. It can be observed that there was an increase in SCA from 8,240 to 14,203 catalase units g^{-1} VSS after 21 hours, from the starch impulse, which approximately corresponds to the aerobic tank HRT. These SCA values obtained were roughly within the average values found from activated sludge samples from WWT Plants of Halifax (9,420 catalase units g^{-1} VSS) and Owlwood pocket 4 (12,950 catalase units g^{-1} VSS) (Guwy *et al.*, 1998). These results suggest that the biomass monitor could provide information related to the organic load applied to the aerobic stage since an increase in the measured catalase activity should shadow an increase in the organic load. Hence, it could be very useful for controlling the organic loading rate and also the RAS.

Figure 4.22 shows that the highest SCA did not correspond to the lowest DO levels within the aerobic tank, although the lowest SCA corresponded to the highest DO values. Therefore biomass activity has no direct relationship with DO levels. However, the SCA after the 3rd sample seems to respond in parallel to the DO values.

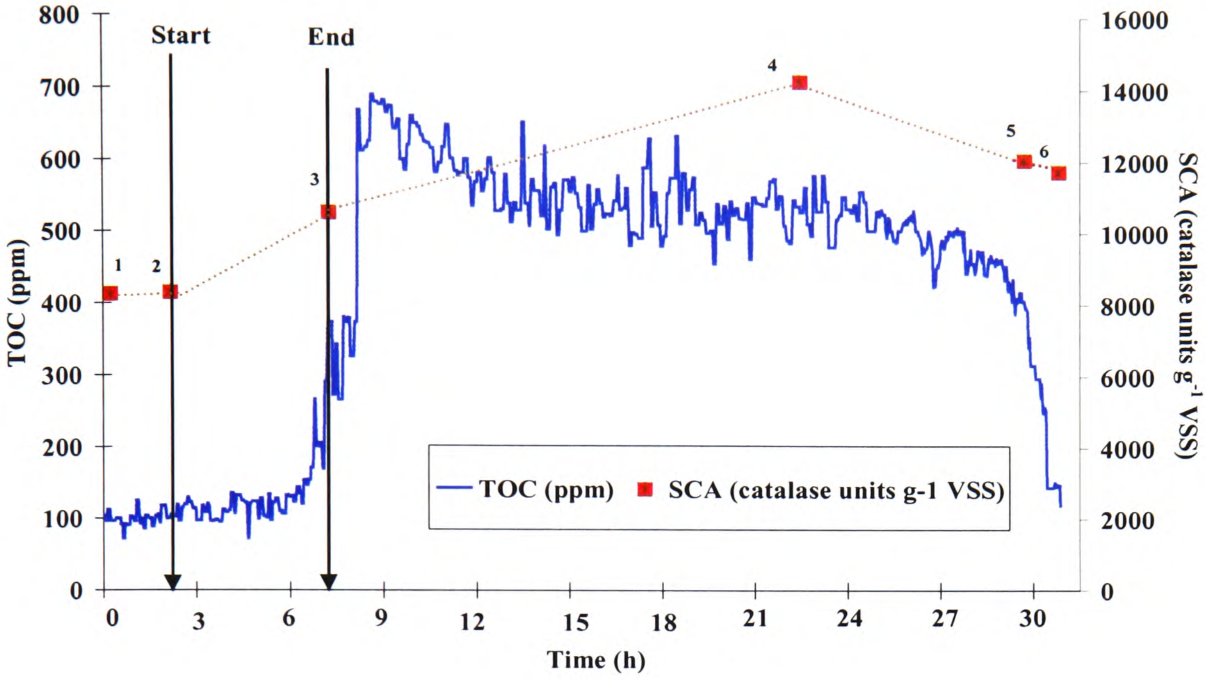


Figure 4.21 - Aerobic stage effluent TOC vs. SCA of the aerobic vessel sludge (Experiments 5.1 and 5.2)

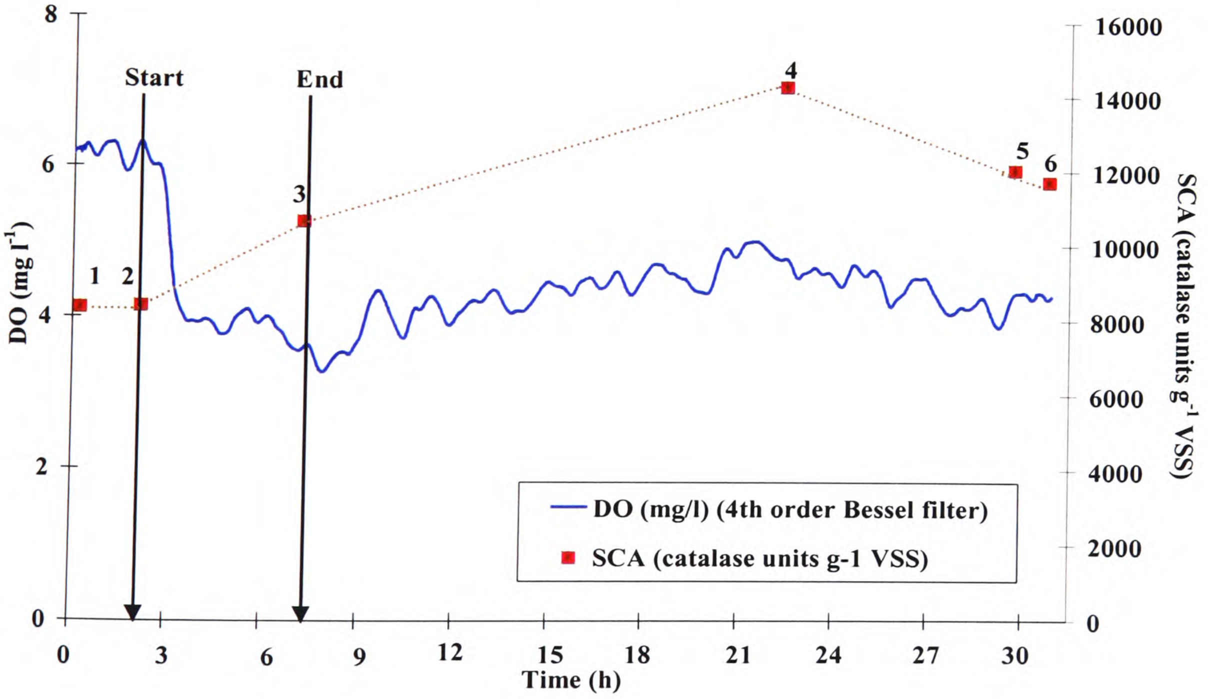


Figure 4.22 – SCA of the biomass vs. DO within the aerobic tank (Experiments 5.1 and 5.2)

4.4.3. On-line Colour Measurement of the Influent to the Aerobic Stage

On-line colour measurements exhibited very little OD variation in the STE. The average OD of the STE before and after the end of the starch step load was 0.56 TCU. A value of 0.83 absorbance units were subtracted from the on-line colour measurements, as the mesh used here was the 185 μm with Filter 1 as Filter 3 was being used with the settler effluent. During the starch step change in load the OD was 0.77 TCU possibly due to the extra starch effect and not due to pH changes.

4.4.4. Conclusions from Experiments 5.1 and 5.2

DO and pH levels within the aerobic tank must be controlled, however they cannot be the only inputs used to control the organic input or the RAS.

A TOC analyser must be placed at the inflow to the aerobic stage if the organic content is unknown and at the effluent of the aerobic stage in order to control changes in the load.

Biomass catalase activity reflected plant performance with varying organic load. It responded faster than TOC measurements of the aerobic effluent to the starch impulse. The measurements of biomass catalase activity also proved that as expected a higher biomass activity was possible with lower biomass concentration. Therefore a biomass activity monitor, working on-line with an appropriate sampling frequency, could be used to control the RAS and off-line measurement of MLSS or VSS would not be needed and also it could enable a control scheme to vary the addition of nutrients and other supplements.

5. DEVELOPMENT AND ON-LINE TESTING OF THE CONTROL SCHEMES

This Chapter is divided into three main areas of work. The first Section presents the development of the ANNBCS by selecting the most appropriate type of ANN to use. The second Section includes the selection of an appropriate hybrid ANNBCS in order to cope with sensor failure. Finally, a third section, which includes the on-line testing of two ANNBCS (1 and 2) to both stages: aerobic and anaerobic, respectively. In the three Sections training and off-line testing of the different ANNs have been performed. All the ANNs that make up the Control Schemes were configured, trained and tested using the MATLAB® Neural Network Toolbox (Demuth and Beale, 1994).

The suitable configuration of the ANNs was dependant on the data sets (i.e. number of sets and their relevance) and the initial values of the weights. However, throughout this Chapter where it was thought appropriate information on the number of layers, neurones per layer, learning rates, momentum values, and so on, are presented. This information may be useful, as a guideline, for people starting to use these ANNs for similar purposes.

5.1. Artificial Neural Network Selection

The best ANN to use in the control scheme was first selected using data gathered during another project (Guwy *et al.,* 1997a) defined as Experimental Phase 1, which was briefly described in Section 3.6.1.

As presented in Chapter 2 an ANN is characterised by its topology, the connection strength between neurones, known as weights, neurone properties including its transfer function, and a learning rule (Demuth and Beale, 1994). The arrangement and the nature of the neurones' inter-connections determine the structure of a network, whilst the manner in which weights are adjusted during training to achieve a desired overall behaviour is governed by its learning algorithm.

The following ANNs were configured, trained and tested:

- Linear network (Widrow and Sterns, 1985);
- BP network (Rumelhart and McClelland, 1986);
- RBFN (Chen *et al.*, 1991);
- Elman network (Elman, 1990);
- SOM (Kohonen, 1989).

All the networks are of the FF type except the Elman network, which is partially recurrent. In addition, all the network training was supervised except for the SOM.

5.1.1. Network Architectures and Off-line Training

The inputs and outputs of the networks are described below, except for the SOM, since it is an unsupervised network. Structure and training conditions for the SOM will be dealt with in Section 5.1.2.

The inputs to all five ANNs included the on-line results, which were obtained from the BA, pH, H_2, biogas flow and CO_2 measurements in four different operating conditions:

- reactor response during 'normal' operation;
- reactor response during step changes in the organic load;
- simulated data for failure of the BA sensor;
- simulated data for fouling of the pH sensor.

The outputs of each supervised network were the two remedial actions, as listed below, proposed for the anaerobic digester:

- increase the bicarbonate buffering when BA is below 1.8 g $CaCO_3$ l^{-1}, in order to maintain the BA at a safe level for stable bacterial activity (the lowest recommended level of BA is 1000 mg $CaCO_3$ l^{-1} without adversely affecting the bacterial population (Metcalf and Eddy, Inc., 1991);

- adjust the feed flow rate if there is an organic overload (it was arbitrarily decided that a 50 % diversion of the incoming load was necessary, if such condition arises). With normal operation the full load (100 %) should be applied. A schematic of this is shown in Figure 5.1.

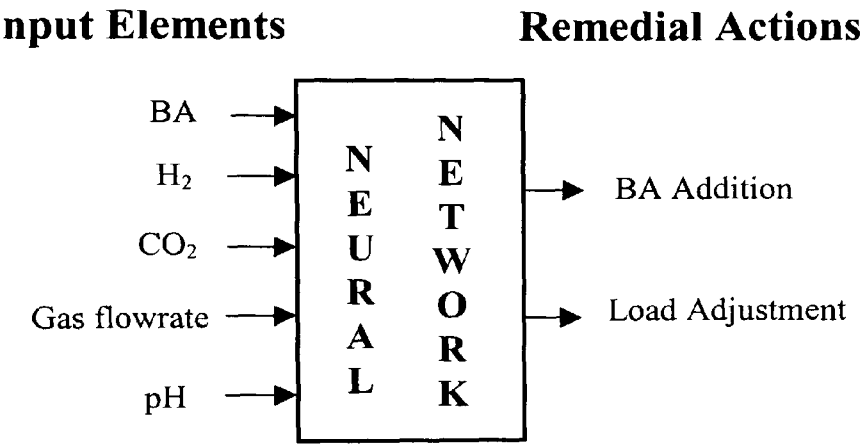


Figure 5.1– Diagrammatic representation of the ANN controller with the sensorial information as inputs and remedial actions as outputs (except for the SOM)

Twelve days of sensorial data resulting from the feeding pattern (Figure 5.1) was pre-selected so as to decrease the number of data points to be dealt with by the ANNs. Each different type of network was trained using 2 sets of data. The first consisted of 124 data sets, which did not include simulated sensor failure conditions and the second consisted of 346 data sets, which included simulated sensor failure conditions. For the various ANNs the 'optimum' configuration and the overall error goal of the network in order to minimise the training period and at the same time improve the accuracy of the network predictions were obtained empirically. For the configuration of the ANNs parameters such as the number of layers and hidden neurones, learning rate, transfer and training functions, spread constant (i.e. the width of each data cluster for each hidden neurone), used by the RBFN were experimentally determined. The final configuration for each of the networks is described below.

The *linear network* had a two neurone single linear layer, due to the use of the Widrow-Hoff rule for training. However, the employment of multiple layers does not result in a more powerful network, as is generally known linear networks can only solve linear problems (Demuth and Beale, 1994). The Widrow-Hoff rule calculates small changes for the neurone's weights and biases in a direction that decreases the neurone's error. This rule is then implemented by making changes to the weight in the opposite direction and updating the learning rate. The initial learning rate was set as 0.9. It was trained, by presenting the inputs and the outputs to the network, as in Figure 5.1. It used the linear transfer function resulting in a minimum SSE of 1.6 (i.e. the sum squared differences between the network targets and actual outputs for both training data sets), after 45 and 50 epochs for the first and second data sets, respectively.

The *BP network* had an input layer of 5, a hidden layer 30 (first data set) and 35 (second data set) of arbitrarily chosen neurones and an output layer of 2 neurones (for the two remedial actions). The characteristic of the neurones in each layer was of the logarithmic sigmoid transfer function and the network was trained with the Levenberg-Marquardt optimisation technique (Demuth and Beale, 1994) to speed up learning. The network employed momentum (to make it less likely to get caught in a local error minima) with a value of 0.1 and an initial learning rate of 1×10^{-4} with adaptive factors. The SSE achieved was 0.01 after 25 and 37 epochs for the first and second data sets, respectively.

The *RBFN* employed a 2-layer configuration with the input layer hosting the RBF neurones and the output layer consisting of linear neurones. A maximum of sixty neurones was selected for the hidden layer and two were used for the output layer. During training, neurones were added to the network until the SSE fell beneath an error goal or the maximum number of neurones had been reached. In order to determine the strengths of the connection between the hidden and the output layer, the generalised least square minimisation was used. This consisted of utilising the output vectors of the hidden layer and the desired output target (Demuth and Beale, 1994). The SSE achieved after 10 (first data set) and 17 (second data set) epochs was 0.1 with the spread constant set to 0.1 for the RBF layer.

The *Elman network* reached a SSE of 1.0 while set to use an initial adaptive learning rate of 0.01. It consisted of 2 layers, one being recurrent using a hyperbolic tangent sigmoid transfer function and the other an output layer with a linear transfer function. The ratio used to increase the learning rate was 1.05 whilst the ratio to decrease the learning rate was 0.7, the error ratio was 1.04 and the momentum constant was 0.95. The SSE achieved was 0.1 after 22 and 50 epochs for the first and second data sets, respectively.

5.1.2. Results and Discussion

Each ANN was validated with data not presented during training. The validation of the network consisted of classifying the 4 different Scenarios (Table 5.1), which aimed to simulate the following events:

Scenario 1 – 'normal' operation;
Scenario 2 - organic overload condition;
Scenario 3 - faulty operation of BA analyser;
Scenario 4 - pH probe fouling.

Ten data vectors for each of the Scenarios (a total of 40 data sets) were used to validate each of the ANNs. For Scenarios 3 and 4 (sensor failure) five data sets where related to normal operation and the other five to an organic load increase.

Table 5.1 - Typical values, of the four different Scenarios, used to test the ANNs

Scenarios	**BA ($g\ l^{-1}$)**	**H_2 (ppm)**	**CO_2 (%)**	**Biogas Flow ($ml\ min^{-1}$)**	**pH**
1 Normal	1.8	150	32	48	7.0
2 Organic load increase	1.2	400	55	50	6.6
3 BA analyser fault	0.0	150	32	48	7.0
4 pH probe fault	1.8	150	32	48	4.0

Table 5.2 shows the remedial actions suggested by each network for Scenarios 1 to 4. It must be noted here that each network was only trained with data covering Scenarios 1 and 2, therefore its predictions for Scenario 3 and 4 can be treated as a validation exercise, namely for generalisation. It can be seen that each network acted quite appropriately by making

sensible decisions, and did not respond catastrophically in any of the validation situations. For example, in Scenario 2 where the organic load was increased, all networks activated the addition of BA, which had fallen below 1.8 g $CaCO_3$ l^{-1} and decreased the incoming load. In practice, some load would be temporarily diverted to a buffer tank with the flow to the digester perhaps being diluted with treated effluent. In Scenario 3 the actual BA in the reactor was 1.8 g $CaCO_3$ l^{-1} despite the false sensor reading, which was simulated to provide a much lower reading to the networks.

Table 5.2 - Comparison of the different ANNs predictions to the desired targets when not trained for sensor failure (%)

Type of Network	**Scenario 1**		**Scenario 2**		**Scenario 3**		**Scenario 4**	
	BA Add.	Load Adj.	BA Add.	Load Adj.	BA Add.	Load Adj.	BA Add.	Load Adj.
Linear	6	101.9	57	58.2	-13	89.79	126	66.8
BP	6	99.9	60	46.5	6	83.5	125	64.9
RBF	11	94.1	40	56.2	24	76.0	25	76.2
Elman	0	92.5	56	62.8	-60	92.3	97	90.2
Target	**0**	**100**	**60**	**50**	**0**	**100**	**0**	**100**

However, the performance of all networks for Scenarios 3 and 4 was unsatisfactory. For example, in Scenario 4 the Linear, BP and Elman networks all resulted in the maximum addition of BA even though the BA reading was normal. It was felt that by including fault conditions of BA and pH in the training data that the performance of the controller would be improved. This can be seen in Table 5.3 where there was an improvement in all the networks for Scenario 1. In Scenario 2, remedial action of BA addition at a rate of 60 % of the maximum and a deviation of 50 % of the incoming load to a buffer tank were proposed. These remedial actions were achieved most closely by the Linear and BP networks while the Elman network did not propose any addition of BA. In Scenario 3, the Linear, BP and RBF networks have an improved performance but the Elman network predictions had deteriorated with respect to BA addition, although improvement with respect to load adjustment was demonstrated. The Elman network as expected was the least able to cope with sharp changes in the data such as a sudden sensor failure due to the network's inherent need for a smoother temporal pattern. In Scenario 4 all networks had improved performance, and had delivered satisfactory results.

Table 5.3 - Comparison of the various ANN predictions to the targets when trained for sensor failure (%)

Type of Network	Scenario 1		Scenario 2		Scenario 3		Scenario 4	
	BA Add.	Load Adj.	BA Add.	Load Adj.	BA Add.	Load Adj.	BA Add.	Load Adj.
Linear	0	102.9	50	57.7	-7	92.0	20	103.0
BP	4	100.0	68	50.3	0	100.0	0	98.4
RBF	8	94.4	34	60.0	8	91.1	4	93.3
Elman	-14	101.2	5	50.2	21	98.4	27	98.1
Target	**0**	**100**	**60**	**50**	**0**	**100**	**0**	**100**

The ability of a linear network and a multi-layered FF network, MIMO, in modelling the identical data set was studied by Premier *et al.* (1999). These authors showed that even a linear network obtained reasonable performance. These linear models were able to follow the behaviour of the reactor by utilising a piece wise linear approximation of the non-linear process. In this study there was no data 'window' that could be altered as necessary so all the data sets were presented to the linear network and the results were satisfactory. However, the BP network in general was more capable, suggesting more accurate remedial actions than the linear network, which is to be expected since the network itself is more capable in representing non-linear relationships (Hornik *et al.*, 1989).

The selected ANN must also be able to cope with the data from the four Scenarios, as the performance of the ANN is vital to maintain satisfactory operation of the plant. In order to assess the suitability of each network, it was decided to aggregate the errors for both ANN outputs (i.e. BA and load adjustments) and express the error as the percentage achieved for each Scenario to give a final overall error (Table 5.4). As can be seen, the percentage error of the predictions were, for each of the networks, reasonably small although the best performance was given by the BP network with a total overall error of 13.9 %, from which 60 % arises from Scenario 2. This could be possibly further reduced if more data relating to this Scenario was presented to the network during training.

Table 5.4 - Aggregated network error expressed as a percentage

Type of Network	Scenarios				Total Error
	1	2	3	4	
Linear	2.9	17.7	15.0	23.0	58.6
BP	4.0	8.3	0.0	1.6	13.9
RBF	13.6	36.0	16.9	10.7	77.2
Elman	15.2	55.2	22.6	28.9	121.9

In practice sensor failure may go unnoticed by a conventional controller and the process might be uncontrolled for several days, during which time, severe reactor breakdown could occur. For the data used here, the BP network considerably outperformed the other networks. It can also be concluded from this work that in order to deal with a wider range of sensor failure conditions, a large training data set would be necessary thus leading to excessive training time (Demuth and Beale, 1994) and lower accuracy due to contradicting sensorial information. A better approach would be to employ a pre-processing stage which classified the data prior to feeding the sensorial information to an appropriately trained BP network, in this case the 'specialised' network would be the most able to recognise the situation.

The SOM, being an unsupervised ANN, could easily be used to classify different operating scenarios including sensor failure conditions. It has been found to be superior for the classification of faulty conditions, to the standard BP network (Silva, 1997). The use of the SOM does not readily lend itself to taking control actions rather, the network is best suited to the classification of incoming data into distinct classes. This is due to the nature of the network's output, as it is a single output which define classes e.g. 1, 2, to n and not continuously variable outputs which are needed for the remedial actions (e.g. voltage outputs). An SOM could be used to pre-process the incoming information in order to select the most appropriate BP network to take the necessary control actions. This pre-processing ability would perhaps become quite significant when there was a combination of faulty sensorial information that could persist for an extended period of reactor operation. Figure 5.2 demonstrates the ability of the SOM at classifying the two loading states of the reactor accurately using a simplistic one-dimensional Kohonen layer with 2 neurones. The larger circle denotes the dominant (or the winning neurone), and the smaller circle denotes the neighbour neurone.

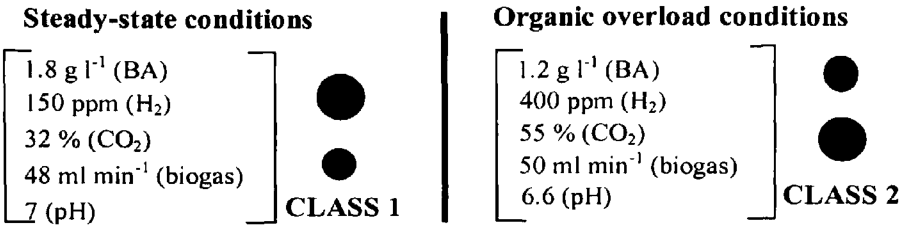


Figure 5.2 - SOM classification of loading conditions to distinguish Scenario 1 from Scenario 2

Figure 5.3 demonstrates that the use of a two-dimensional SOM can independently classify the two loading conditions (Scenarios 1 and 2). It can also classify conditions of BA failure as in Scenario 3, although faulty pH sensor conditions were confused with Scenario 2. This may have resulted from the limited data set that was available to represent the conditions of failure in the pH probe failure, something that should be remedied with more training data. However, Figure 5.3 shows that the SOM recognised a fourth class that represents the intermediate loading condition demonstrating its ability to generalise.

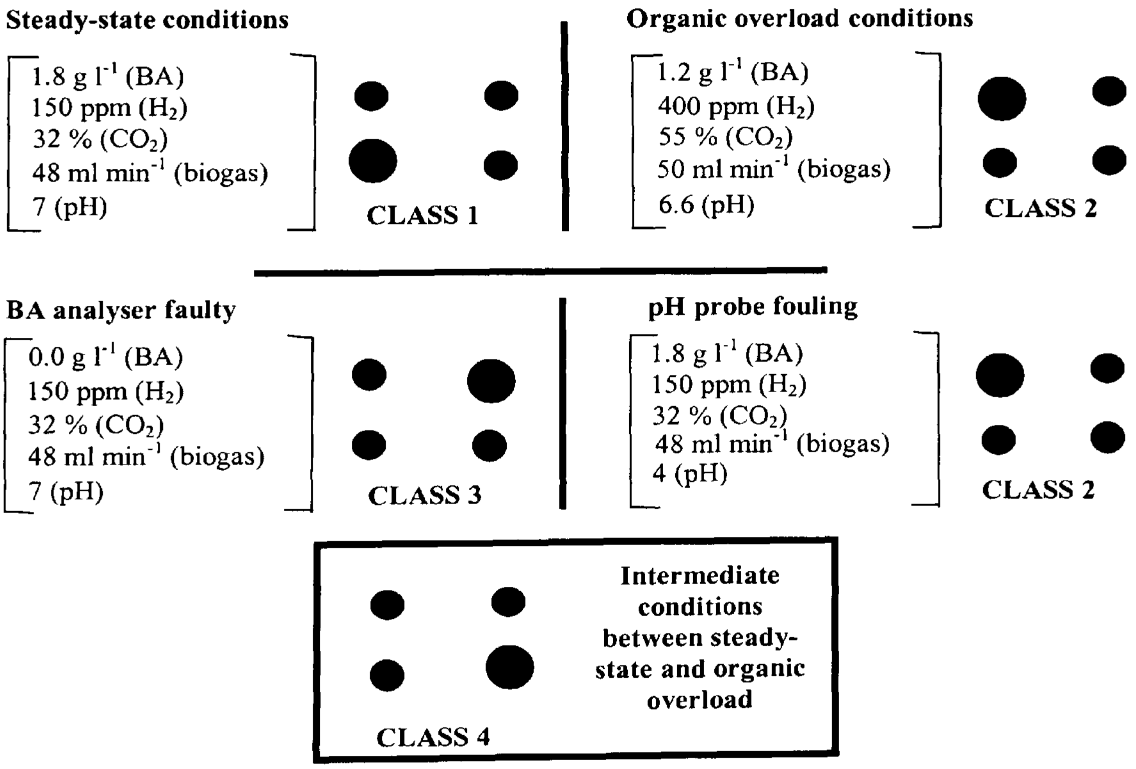


Figure 5.3 - SOM classification of loading and fault conditions to distinguish Scenarios 1 - 4 and an intermediate loading Scenario

5.1.3. Conclusions from the ANN Selection

For the Linear, BP, RBF and Elman networks with the increase in the number of training data sets the number of training epochs have increased in order to maintain a constant SSE.

There are benefits from using ANNs to control the biological treatment process, however, training can be exhaustive with the determination of the optimum configuration of the network is highly empirical. All the tested network configurations were arrived at experimentally in order to improve predictions and decrease the training time.

All the ANNs were structured as MIMOs. The most suitable network to be employed for the purpose of controlling the plant would be the fast BP algorithm. However, the network would then have to be trained for a series of conditions and combinations of sensor failure if it was to be robust enough to act as a 'controller'. The SOM was able to classify clusters of data from various operating conditions and situations of sensor loss and would therefore be a useful tool for pre-processing sensory information before passing it to the BP network for devising suitable remedial actions.

Therefore, a control strategy based on the combination of two ANNs, the SOM and BP, should provide the best solution for the development of a 'simple' ANNBCS for the WWTP. This control scheme would take spot checks on the conditions of the biological process and decide on the appropriate remedial actions ignoring the dynamics of the process. This could result in a general control scheme that could be applied to a range of different processes. The advantage of this would be that instead of training one ANN with all the possible data and fault conditions, a group of smaller ANNs would be trained for classes of data suggested by the SOM classification network.

5.2. Control Scheme Development

The development of the ANNBCS was performed using data gathered from the textile WWT rig during Experimental Phase 2 (Section 3.6.2). The ANNBCS aimed to maintain the BA level, so as to maintain the health of the reactor with a suitable organic and colour loads to the reactor for stable operation and also for discharge compliance. The development of this ANNBCS, for future on-line application, is detailed below under Control Schemes 1 to 4.

5.2.1. Introduction, Data Gathering and Selection

The Control Schemes consisted of one or two ANNs this last being a hybrid structure. They were based on the FFN trained with the BP algorithm alone or coupled with a classification network such as SOM or LVQ network (Pham and Oztemel, 1994). The inputs to the network were the data collected from the sensors, and the outputs were the suggested remedial actions that would be needed for the proper functioning of the reactor.

The four Control Schemes were designed to improve the predictive performance, in terms of the remedial control actions, and also to minimise the training time. Table 5.5 shows six suggested combined remedial actions, which are based on different associations of the four single remedial actions presented in Figures 5.4 to 5.7. The data used to train and test the networks off-line consisted of representative data sets collected during the non-steady state experiments of Experimental Phase 2 (Section 3.6.2). Normal operating conditions were associated with Experiment 2.2, organic step change with Experiment 2.1, colour step change with Experiment 2.4 and organic and colour step changes with Experiment 2.3 (Table 3.2). In addition, there was also simulation of data for the following conditions: reactor instability and low organic and colour loads. The reactor's BA should be maintained above 1000 mg l^{-1}, as $CaCO_3$, to prevent reactor failure together with a pH level between 6.8 and 7.8 (Speece, 1996). These two levels of BA and pH are inter-linked as the bicarbonate ions represent the main pH buffering species in anaerobic digesters (Speece, 1996).

Table 5.6 presents 6 operating conditions with an example of validation data sets. The same validation data was used to test the performance of the four Control Schemes. Thirty-six data sets (sensorial data and remedial actions) concerning non-failure conditions were used. An additional thirty six data sets, which included sensor failure conditions were used (e.g. 3 data sets for operating condition 1 with failure of the BA monitor and three other data sets for operating condition 2 with a failing BA monitor). Similar sets of data were included for pH failure and the other operating conditions.

Table 5.5 - Six suggested remedial actions

Scenarios	**Remedial actions**
i) a temporary increase in organic load with which the digester can just cope	i) BA adjustment if this was below 1200 mg $CaCO_3$ l^{-1} and/or pH was 6.8 or below
ii) reactor instability due to an organic overload	ii) BA adjustment and load reduction
iii) a sustained increase in organic and colour loads that would make the digester unstable	iii) reduction of organic and colour loads if these were above 1200 mg TOD_{out} l^{-1} (UASB effluent) and the average OD at 436, 525 and 620 nm was above 0.3
iv) treatment of any WW previously diverted to a temporary storage tank during periods of high load	iv) load increase from a textile WW holding tank when both levels of organic strength and colour were below the set limits above
v) low starch to dye ratio for successful decolourisation of the WW	v) addition of a carbon source if the TOD_{out} was below 1000 mg l^{-1} and the average OD was higher than 0.3
vi) sensor failure: BA monitor and pH meter	vi) advise on the fault, ignore it when predicting by taking into consideration only reliable information

Table 5.6 - Representative data of the 6 operating conditions

Operating Conditions	**Inputs to the ANNs**					
	BA (mg l^{-1})	TOD (mg l^{-1})	CO_2 (%)	Gas flowrate (ml min^{-1})	pH (pH units)	Av. OD (TCU)
1 Normal operation	1500	1100	25	7	7.0	0.27
2 Organic step load	1700	1900	32	11	7.2	0.30
3 Colour step load	1450	1050	25	8	7.1	0.37
4 Organic & colour step load	1650	1900	31	13.5	7.2	0.40
5 Reactor instability	950	2000	33	10	6.8	0.30
6 Low organic & colour load	1460	1000	25	6	7.1	0.26

Operating condition 5, represents reactor instability which led to the production of VFAs and a consequent drop in the BA level below the safe limit (Speece, 1996). The TOD measured at the outlet of the UASB reactor rose together with the % CO_2 in the biogas, whilst the gas flow rate and the pH decreased. Condition 5 can be distinguished from the case of an organic step load (operating condition 2) where the VFA levels remained low, which was accompanied by an increase of BA levels due to the NaOH content of the influent. This was also followed by an increase in the gas flow rate, although the % CO_2 remained fairly constant. A colour step load (operating condition 3) mainly increased the OD, which is undesirable if compliance to environmental legislation is to be maintained. The effect of operating condition 4 (organic and colour step load) was a combination of both operating conditions 2 and 3, which are both undesirable.

All four Control Schemes were trained and validated with simulated sensor failure conditions with the exception of Control Scheme 1, which did not include such conditions. When simulated sensor failure occurs BA and pH have taken the values of 0 g $CaCO_3$ l^{-1} and 6.5 pH units. The values were chosen because they occurred a few times during the experimental work. The 0 g $CaCO_3$ l^{-1} occurred when there was no sample going to the BA analyser and readings of 6.5 pH units occurred when the pH sensor fouled.

The next five Sections will present and discuss how the different Control Schemes were devised, trained and validated and will analyse how well the four Control Schemes performed in terms of the suggested remedial actions. It will also conclude as to the best Control Scheme to use for this biological treatment process.

5.2.2. Control Scheme 1

Control Scheme 1 was setup considering the results obtained in Section 5.1 where the BP network was found to be the most suitable ANN to use.

Architecture and Development of Control Scheme 1

Figure 5.4 shows a diagrammatic representation of the first Control Scheme. This was based on a standard 3-layer BP algorithm with 15 neurones allocated to the hidden layer. The neurones of each layer possessed a logarithmic sigmoid transfer function and the network was trained with the Levenberg-Marquardt optimisation technique (Demuth and Beale, 1994) to further speed up learning. The network employed momentum, to prevent being caught in local minima, with a value of 0.1 and an adaptive learning rate initially set to $1x10^{-4}$. The increment and decrement factors of the learning rate were $1x10^{-3}$ and 10, respectively and the target SSE was set to 0.02. Eighty-five data sets were used during training of the Control scheme 1.

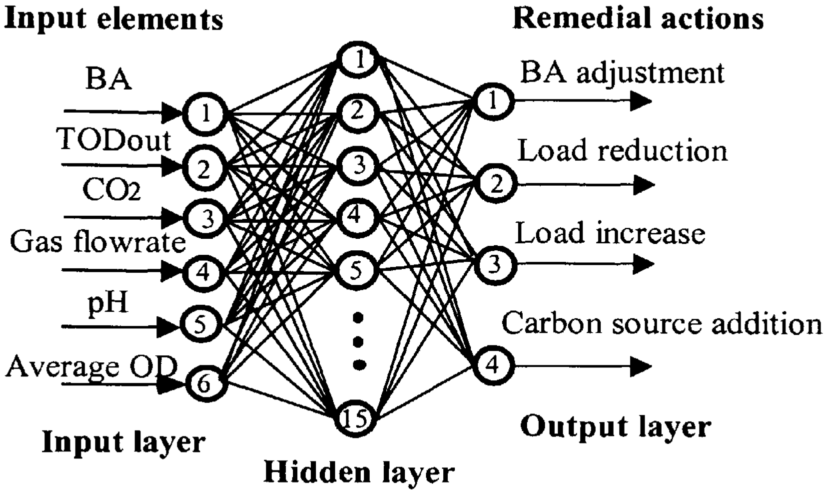


Figure 5.4 – The 3-layer FFN structure for the Control Scheme 1

Results and Discussion

The ANN controller decisions are presented in Table 5.7. These would ultimately be transferred to the plant actuators e.g. pumps or valves on a normalised scale of 0 to 1 (where 0 means off or closed and 1 means maximum output or fully open). The schematic of this arrangement can be found in Figure 3.7 (Sections 3.5.1 and 3.5.3). As can be seen from Table 5.7 the network made sensible predictions as to the valve openings and pump speeds in the event of a change in the input to the process. For example, for normal operation, the network would leave the process alone, whilst when the organic load was increased the network would reduce the organic content entering the system by 80 %. In the case of operating condition 5 (reactor instability) in which the BA fell to 950 mg $CaCO_3$ l^{-1} and the pH dropped to 6.8 (Table 5.6) the activation of the remedial action is almost full BA addition and maximum reduction of the load strength.

Table 5.7 also displays the ANN decisions for the simulated failure of the pH and BA sensors. As it can be seen, the neural network did not respond well and has made inappropriate decisions. It suggested switching on the bicarbonate dosing pump, reduction of the load, and in the case of BA instrument simulated failure it also predicted an addition of carbon source during all operating conditions, something that was not appropriate. This last condition happened as the BP networks work on the principal that during training the overall range of the data should be shown to the network (Demuth and Beale, 1994).

Table 5.7 - ANN predictions for the 6 different operating conditions and two cases of simulated sensor failure (Control Scheme 1)

Scenarios	**Outputs of ANN**				**Outputs of ANN (pH electrode fouls)**				**Outputs of ANN (BA instrument failure)**			
	BA adj.	Load red.	Load inc.	Carbon add.	BA adj.	Load red.	Load inc.	Carbon add.	BA adj.	Load red.	Load inc.	Carbon add.
1 Normal operation	0	0	0	0	0.9	1	0	0	1	0.9	0	0.5
2 Organic step load	0	0.8	0	0	1	1	0	0	1	1	0	0.5
3 Colour step load	0	0.5	0	0.4	1	1	0	0	1	0.9	0	0.4
4 Organic & colour step load	0.3	1	0	0	0.9	1	0	0	1	1	0	0.5
5 Reactor instability	0.9	0.9	0	0	0.9	1	0	0	1	1	0	0.4
6 Low organic & colour load	0	0	0.6	0	0.9	1	0	0	1	0.9	0	0.5

5.2.3. Control Scheme 2

The second Control Scheme was setup as for the Control Scheme 1 and trained and validated with additional conditions.

Architecture and Development of Control Scheme 2

The diagrammatic representation of Control Scheme 2 is the same as Control Scheme 1 as the second control scheme resulted from re-training the first Control Scheme with sensor failure conditions (Section 5.2.1), in addition to the original training set. This it was thought would increase the tolerance of the network to possible sensor failure. The number of hidden neurones was also increased from 15 to 25 in order to cope with the increase in the number of data sets (Demuth and Beale, 1994). The network was trained until it reached an acceptable accuracy. During training of the BP network comprising the Control Scheme 2, 124 data sets were used.

Results and Discussion

Table 5.8 displays the results for the six operating conditions encountered in Control Scheme 1. However, in this Control Scheme the failure of the pH and BA instruments has been included in the examples used to train the ANN. As can be seen in Table 5.8, the ANN outputs for cases when the instrument failed have improved over that achieved for Control Scheme 1 (Table 5.7), where no inappropriate BA addition was made. However, the network predictions for conditions when no instrument failure occurs were not as satisfactory, this was perhaps due to the large and contradictory set of training data. For example, in the case of the reactor being unstable (operating condition 5) the ANN decided that no appropriate remedial actions were required when it should have suggested reduction of the load strength and an increase of the BA flow. This means that this Control Scheme no longer reliably classifies the BA instrument output. Similarly, the load was not increased in operating condition 6 when the BA and pH sensors failed. From the results of Control Scheme 2, it can be concluded that an improved means of controlling such a complex process where sensor failure is often an occurrence is needed.

Table 5.8 - ANN predictions with two cases of sensor failure, when these have been included in the training data (Control Scheme 2)

Scenarios	**Outputs of ANN**				**Outputs of ANN (pH electrode fouls)**				**Outputs of ANN (BA instrument failure)**			
	BA adj.	Load red.	Load inc.	Carbon add.	BA adj.	Load red.	Load inc.	Carbon add.	BA adj.	Load red.	Load inc.	Carbon add.
1 Normal operation	0	0	0	0	0	0	0	0	0	0	0	0
2 Organic step load	0	0.5	0	0	0	0.5	0	0	0	0.5	0	0
3 Colour step load	0.2	0.6	0	0	0.1	0.5	0	0	1	0.2	0	0
4 Organic & colour step load	0	0.9	0	0	0	0.8	0	0	1	1	0	0
5 Reactor instability	0	0.8	0	0	0.1	0.7	0	0	0.1	0.5	0	0
6 Low organic & colour load	0	0	0.3	0	0	0	0	0	0	0	0	0

5.2.4. Control Scheme 3

A hybrid neural Control Scheme was setup, trained and validated. Control Scheme 3 was developed to cope with the large and contradictory sensorial information and will be described in this Section. The SOM was used for classification based on the positive results shown in Section 5.1.2.

Architecture and Development of Control Scheme 3

Figure 5.5 shows, a schematic, of the third control scheme, which consisted of a SOM and 6 BP networks. The SOM was used to pre-process the incoming data, to detect when a sensor had failed together with the operating conditions, and to select the most appropriate neural network component from a suite of multilayered perceptron networks in order to optimise the WWT process for the most probable situation. The 2-dimensional SOM was constructed with 6 neurones in the Kohonen layer, an initial learning rate of 1 and the training data sets were presented to the network 1000 times. As the nature of training was of the unsupervised type, the use of the SSE was not required in this case. The accuracy of the predictions depends on the number of classes attributed and the number of times that the training data sets are presented to the network. The degree of certainty of classification by the network is reflected by the emergence of a dominant neurone (denoted by the larger circle). The persistency of the emergence of a pattern reflects the network's certainty of the class being presented to the network. Figure 5.5 shows the case when neurone 1 of the SOM is dominant (largest) and this then selects the appropriate BP network to make the necessary remedial actions (being in this case BP1). Neurone number 2, 3 and 4 are neighbours to the dominant neurone and the fifth and sixth are termed dead neurones, these ones are furthest away from the dominant one.

The six BP networks, each representing a set of remedial actions designed to cope with the expected range of undesirable reactor behaviour, consisted of similarly structured networks employed in Control Schemes 1 and 2. The difference here is that the data used to train the Control Schemes 1 and 2 was subdivided into six distinct data clusters. Training of the six BP networks was performed only after the SOM predictions were obtained. The data sets for training of the BP networks were defined according to the SOM classification.

By experience, a smaller SSE (0.001) was thus achievable in order to improve the accuracy. Although the training data sets decreased as they were subdivided, the numbers of hidden neurones (15) were maintained so as to allow for the improvement in the error goal. The number of data sets used during training by the different ANNs of Control Scheme 3 were as follows: SOM - 124; BP1 and BP2 – 30; BP3 – 6, BP4 – 14; BP5 – 21; and BP6 – 23.

Results and Discussion

Table 5.9 displays the results obtained using Control Scheme 3 and includes the result of the SOM classification. The second column of Table 5.9 shows which neurone, from the 6 attributed during training was dominant. Each dominant neurone represents one class of representative data.

As can be seen for the case when all sensors were operational, this Control Scheme performed similarly to Control Scheme 1, whereas for the two cases of simulated sensor failure Control Scheme 3 performed significantly better than Control Schemes 1 and 2 (Tables 5.7 and 5.8). This is best observed for the cases of organic step load and reactor instability where Control Scheme 3 would provide the appropriate buffering and load reduction for instances where either sensor failed. However, carbon was not added in operating condition 3 when either sensor was failing, and BA was added in this case. This could perhaps be due to insufficient training data and/or to disadvantages in using an unsupervised learning network such as SOM in this type of Control Scheme. The observed disadvantage was that slightly different data fired the neighbour neurone and not the dominant one (i.e. it predicted a different class), consequently there was less accurate predictions, as the BP network selected was not trained with data similar to the validation data set.

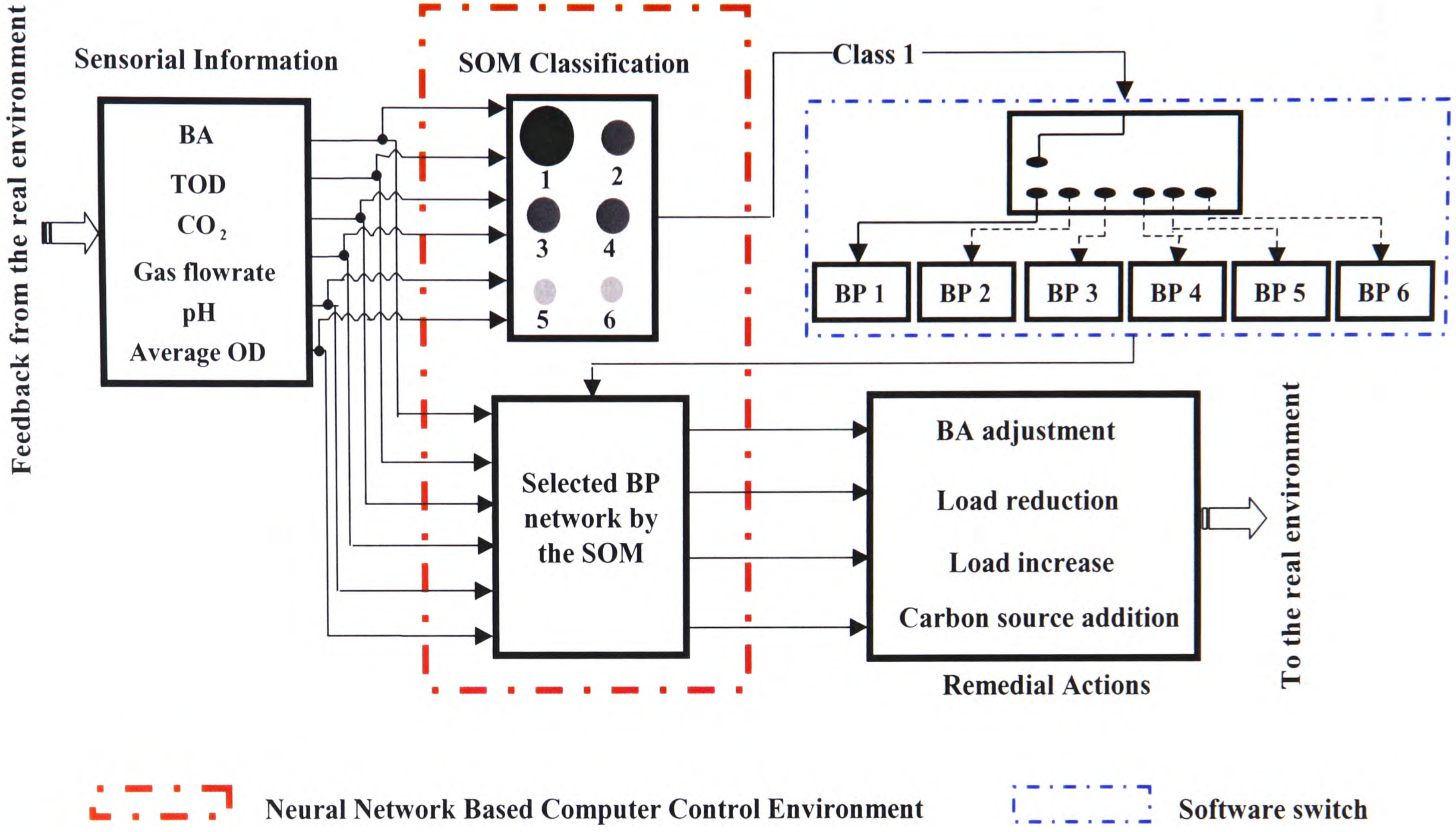


Figure 5.5 - Hybrid Control Scheme 3 (SOM + BPs)

Table 5.9 - ANN predictions for Control Scheme 3

Operating Conditions	**SOM Classification**	**ANN Outputs**			
		BA adj.	Load red.	Load inc.	Carbon add.
1 Normal operation	**5**	0	0	0	0
2 Organic step load	**2**	0	0.6	0	0
3 Colour step load	**3**	0.2	0.6	0	0.2
4 Organic & colour step load	**2**	0.4	0.8	0	0
5 Reactor instability	**2**	0.5	0.8	0	0
6 Low organic & colour load	**5**	0	0	0.5	0
BA Failure					
1 Normal operation	**1**	0	0	0	0
2 Organic step load	**2**	0	0.7	0	0
3 Colour step load	**1**	0	1	0	0
4 Organic & colour step load	**2**	0.7	1	0	0
5 Reactor instability	**2**	0.5	0.8	0	0
6 Low organic & colour load	**1**	0	0	0.4	0
pH Failure					
1 Normal operation	**5**	0	0	0	0
2 Organic step load	**6**	0	0.9	0	0
3 Colour step load	**6**	0	1	0	0
4 Organic & colour step load	**6**	0	0.9	0	0
5 Reactor instability	**3**	1	0.4	0	0.2
6 Low organic & colour load	**5**	0	0	0.3	0

5.2.5. Control Scheme 4

It was thought from the experience of running the biological treatment process that using a supervised learning classification network such as the LVQ network could be more useful for the control purpose than utilising the SOM. If the LVQ network is used, the BP networks can be trained even before the classification network as the data sets for the different classes and consequently the data for training of the various BP networks is selected by the user. The sensorial data corresponding to the different operating conditions are quite distinctive from each other, so that there is no need to use an unsupervised classification network. The use of the LVQ network presents also another advantage as for the predictive accuracy due to its learning structure. The advantages of the LVQ network in comparison to other competitive networks will be summarised in the next two Sections.

Architecture and Development of Control Scheme 4

As outlined previously, the SOM is an unsupervised learning network, meaning that the network itself learns to detect regularities in the training data. The neurones of competitive networks learn to recognise groups of similar input vectors. However, the classes that the competitive layer finds are dependent only on the euclidian distance between input vectors. If two input vectors are very similar, the competitive layer will probably put them into the same class. There is no mechanism in a competitive layer design to dictate whether or not any two input vectors are in the same class or different classes. LVQ networks, on the other hand, learn to classify input vectors into target classes chosen by the user in a supervised manner. LVQ networks classify input vectors into target classes by using a competitive layer to find subclasses of input vectors, and then combining them into the target classes. The use of an LVQ network therefore is a safer option in comparison to the SOM. LVQ networks can classify any set of input vectors, the only requirement is that the competitive layer must have enough neurones, and each target class must be assigned enough competitive neurones.

In terms of training time, LVQ networks can take longer as the user has to define the corresponding outputs. The LVQ network training takes longer providing that a small software program is built when using the option of SOM + BPs (Control Scheme 3). This program must assist in the automatic division of the data sets (i.e. depending on the predicted class) into smaller data sets for the training of the BP networks, otherwise the use of the SOM can also be time consuming. Another disadvantage of the SOM, for this type of Control Scheme, is that some of the data vectors had a dominant neurone e.g. 2 but 1, 3 and 4 were neighbours of the dominant neurone meaning that a similar data set could actually belong to one of the neighbouring neurones. Since the build up of the data set to train each BP network is only based on the dominant neurone classification, it might result in a degradation of the predictions made by the BP network, as they might not contain similar data when trained.

Therefore, a supervised classification network such as the LVQ network was thought to perform better in this type of Control Scheme. Validation of the Control Scheme (Figure 5.6) with the same data sets as for Control Scheme 3 was performed and the results are presented in the next section. For simplicity, only a box represents the LVQ network. The structure of this network is shown in more detail in Figure 5.7. This ANN contains 2 layers: a competitive hidden layer with a size of 24 neurones (i.e. 6 classes with 4 subclasses each).

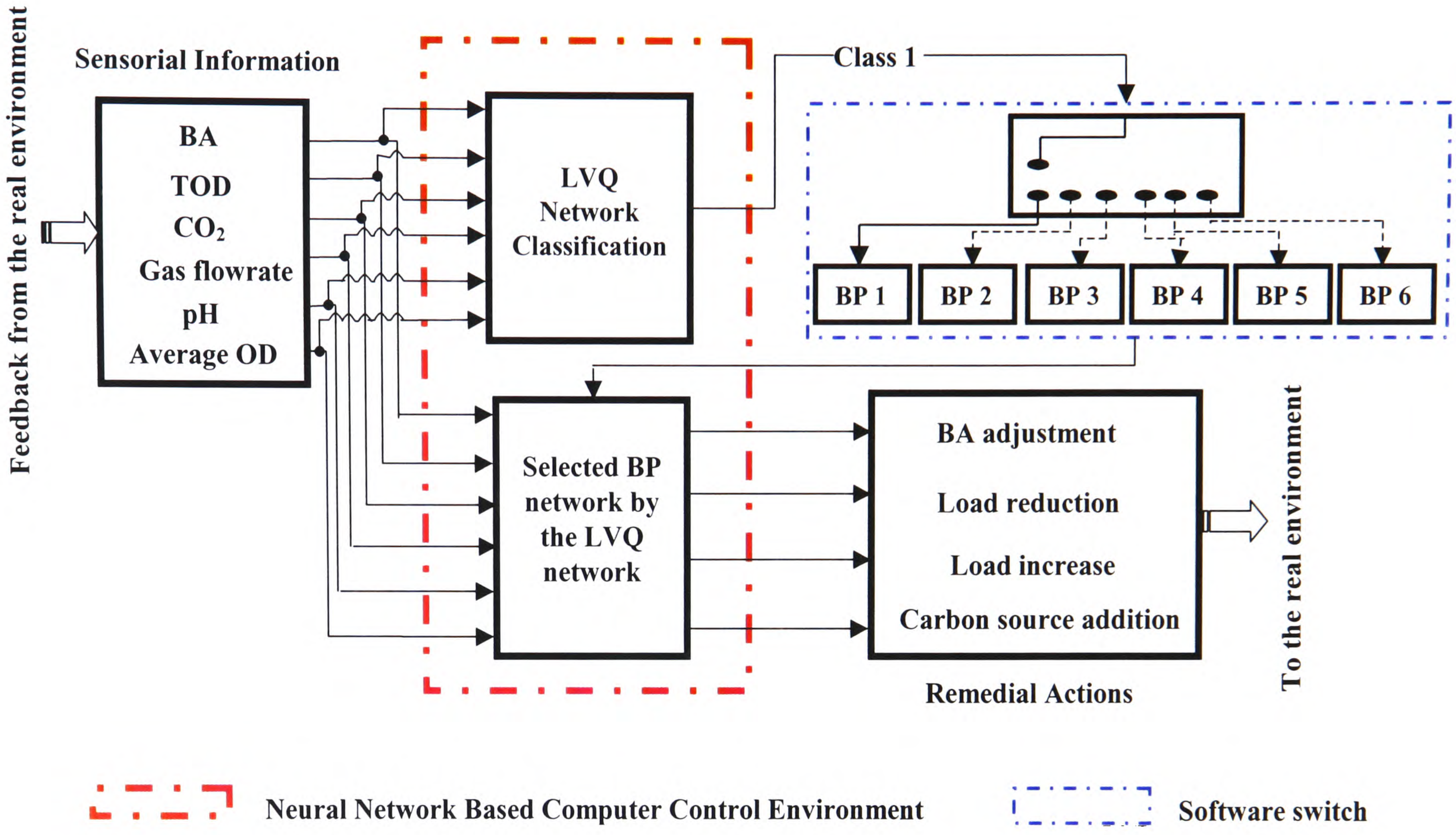


Figure 5.6 – Hybrid Control Scheme 4 (LVQ + BPs)

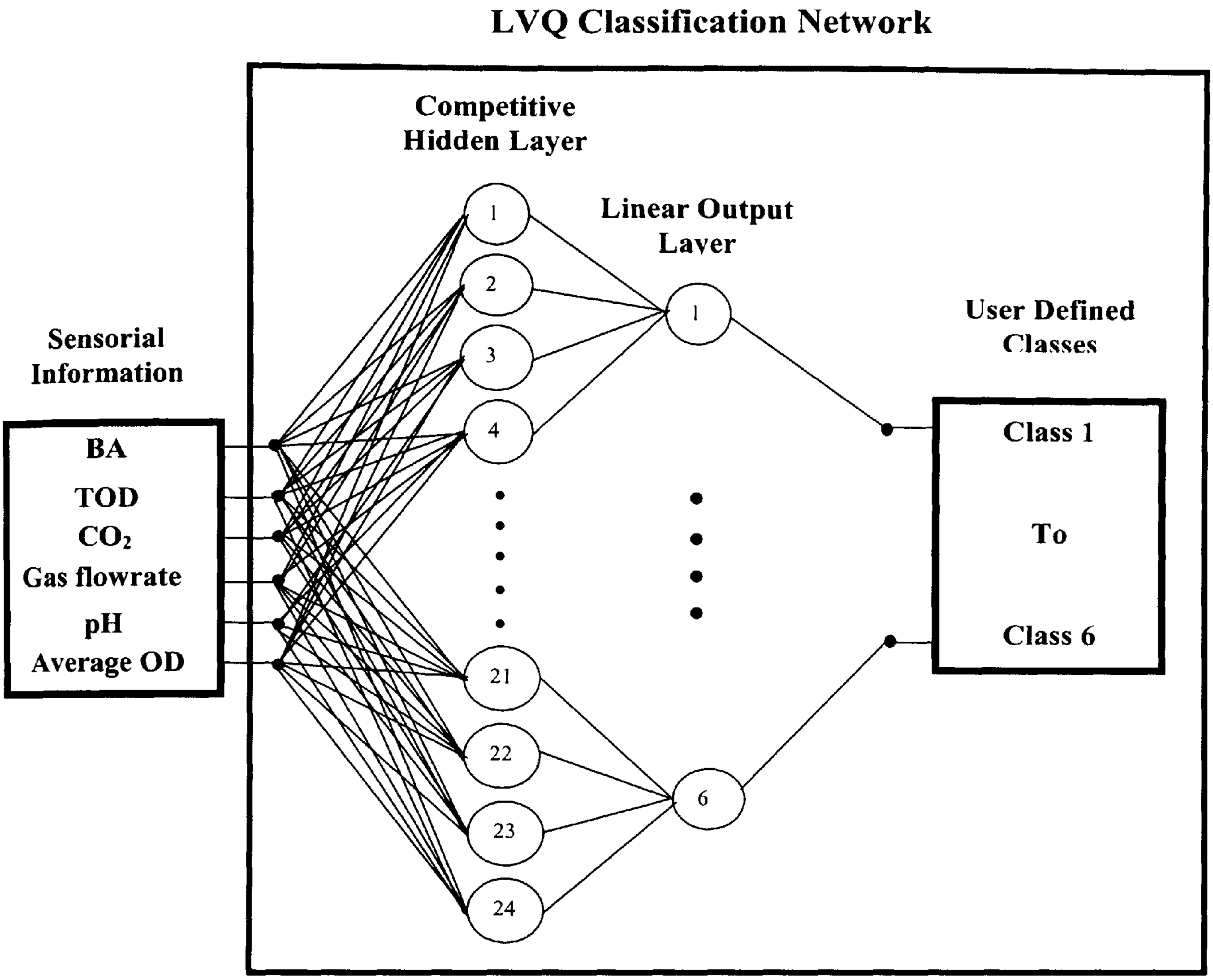


Figure 5.7 – LVQ network used in the Control Scheme 4

Table 5.10 shows the classification work and the respective pre-defined outputs of the LVQ network. Training was set (as for the SOM) at 1000 epochs. The data defining each class (output of the LVQ network) was pre-defined by the user (i.e. supervised learning). The use of six different classes (as the outputs of the LVQ network) were chosen based on previous experience and also to maintain the number of classes used by the SOM for a fairer comparison of predictions. The six BP networks, each representing a set of remedial actions designed to cope with the expected range of undesirable reactor behaviour and sensor loss, consisted of similarly structured networks employed as for Control Schemes 1 to 3.

The number of data sets used during training by the different ANNs of Control Scheme 4 were as follows: LVQ - 124; BP1 – 18; BP2 – 20; BP3 – 19, BP4 – 32; BP5 – 18; and BP6 – 17.

Table 5.10 – Training of the LVQ Network for Control Scheme 4

LVQ network input data from sensors	LVQ network output designation
Normal operation	[0 0 0 0 0 1] → **Class 1**
BA failure	[0 0 0 0 1 1] → **Class 2**
pH failure	[0 0 0 1 1 1] → **Class 3**
Organic step load & colour step loads (joint and separately)	[0 0 1 1 1 1] → **Class 4**
Reactor instability	[0 1 1 1 1 1] → **Class 5**
Low organic and colour loads	[1 1 1 1 1 1] → **Class 6**

Results and Discussion

Scheme 4 similarly to Scheme 3 was used to detect bad reactor operation and whether a sensor had failed and subsequently, to select an appropriate BP network to cope with the situation. Table 5.11 displays the remedial actions predicted by the Control Scheme 4 and also the result of the LVQ network classification.

As can be seen for the case when all sensors were operational, this Control Scheme performed similarly to Control Scheme 1, it predicted better than Control Scheme 2 and considerably better than Control Scheme 3, especially for operating condition 3 (i.e. colour step load). In this case Control Scheme 4 did not suggest additional intake of BA and suggested a more appropriate carbon addition in order to improve dye degradation.

For sensor failure conditions Control Scheme 4 outperformed all the other Control Schemes. However, a closer comparison to Control Scheme 3 will be made here, as they proved to be the two best Control options. The fourth Scheme during a colour step load (i.e. operating condition 3) predicted well for both sensor failure conditions, very similar to the case of no sensor failure. Another improvement was for the case of reactor instability and simulated failure of the pH probe. In these cases Control Scheme 3 suggested an increase in carbon addition and no load reduction, both undesirable control actions. On the other hand, Control Scheme 4 only increased the BA intake and reduced the load strength to the reactor; it did not suggest any carbon addition, all appropriate suggestions. Control Scheme 4 also improved the predictions for operating condition 6 (low organic and colour load) where it dictated a 0.5 load strength increase, with loss of both sensors, instead of 0.4 and 0.3 for BA and pH sensors failure, respectively, in Control Scheme 3.

Table 5.11 - ANN predictions for Control Scheme 4

Operating Conditions	**LVQ network Classification**	**ANN Outputs**			
		BA adj.	Load red.	Load inc.	Carbon add.
1 Normal operation	**1**	0	0	0	0
2 Organic step load	**4**	0	0.7	0	0
3 Colour step load	**4**	0	0.5	0	0.4
4 Organic & colour step load	**4**	0.3	0.8	0	0
5 Reactor instability	**5**	0.6	0.9	0	0
6 Low organic & colour load	**6**	0	0	0.6	0
BA Failure					
1 Normal operation	**2**	0	0	0	0
2 Organic step load	**2**	0	0.7	0	0
3 Colour step load	**2**	0	0.7	0	0.3
4 Organic & colour step load	**2**	0.4	0.8	0	0
5 Reactor instability	**2**	0.6	0.9	0	0
6 Low organic & colour load	**2**	0	0	0.5	0
pH Failure					
1 Normal operation	**3**	0	0	0	0
2 Organic step load	**3**	0	0.7	0	0
3 Colour step load	**3**	0	0.7	0	0.3
4 Organic & colour step load	**3**	0.4	0.9	0	0
5 Reactor instability	**3**	0.6	0.9	0	0
6 Low organic & colour load	**3**	0	0	0.5	0

5.2.6. Conclusions from the Control Scheme Development

Training of all the Control Schemes was relatively quick. None of the BP networks took more than 49 epochs, which represents the number of presentations of each data set to the network for 6 input elements together with the 4 targets elements, and the calculation of new network parameter (weights and biases) necessary to minimise the SSE. The training of the SOM and LVQ networks was also performed quickly.

Comparing each of the four Control Schemes in this manner is subjective. In order to achieve a quantitative comparison, the actual response of each scheme was compared with the target and the calculated SSE is displayed in Table 5.12. There is a relatively large error for Scheme 1, which is reduced considerably with the inclusion of the sensor failure conditions in Scheme 2. A further 48 % reduction in prediction error was achieved for Scheme 3 in comparison with Scheme 2. The best Scheme was Control Scheme 4 with a SSE of 0.027, with an extra 29 % prediction improvement compared to Scheme 3.

Table 5.12 - SSEs for the four Control Schemes

Control Schemes	SSE
1	0.206
2	0.073
3	0.038
4	0.027

Note: SSEs achieved for the same validation data set

5.3. Further Development and On-line Implementation of Two ANNBCSs to Control the UASB Reactor and the Aerobic Stage

The work presented in this Section follows the results obtained from Section 5.2 where the best ANNBCS was found and was based on a hybrid ANN based structure (LVQ + BPs). The objective was to test on-line a more complete controller (i.e. more inputs and outputs) based on the structure of Control Scheme 4 for controlling both stages at the same time. However, as mentioned in Section 4.3 the UASB reactor could not cope with an organic step load and therefore two separate ANNBCSs (1 and 2) were trained in order to test on-line their performance at controlling the UASB reactor and the aerobic stage, respectively.

This section presents the off-line training and on-line testing of the ANNBCSs (1 and 2). For the anaerobic stage the control actions were to alter the dye concentration to be fed to the UASB reactor in order to maintain good effluent quality (i.e. average OD < 1.65 TCU) and to maintain BA and pH levels above 1700 mg $CaCO_3$ l^{-1} and 7.4, respectively, despite the simulated failure of the BA analyser. Control over the aerobic stage was achieved through the regulation of the extra starch to the stage using TOC as the control parameter, and also by maintaining the DO level above 3 g l^{-1} and the pH level below 7.2. ANNBCS (1 and 2) were trained with data gathered from Experimental Phases 4 and 5 (Sections 4.3 and 4.4), respectively.

5.3.1. On-line Control of the UASB Reactor (ANNBCS (1))

The monitoring and hardware control setup and the data gathered for the training of the ANNBCS (1) have been described in Sections 3.6.4 and 4.3. As mentioned earlier, only a colour step load could be performed, due to the health of the reactor. For the same reason, it was not advisable to perform an uncontrolled dye step load, therefore, the data used to train the ANNBCS (1) was gathered only from Experiment 4.1. The data, which represents the increase in the STE dye concentration, of colour, BA, pH and TOC was constructed from the previous experience of running the reactor. First of all, what was good or bad reactor operation was defined using the sensorial data for pH and BA (within the UASB reactor), also for colour and TOC in the effluent of the reactor. From here, ANNBCS (1) was re-organised and re-trained based on the same principle as Control Scheme 4 (Section 5.2). From the four remedial actions presented previously, only two were adopted: adjustment of the dye pump, which was pumped separately from the concentrated STE and adjustment of the BA pump. The other two remedial actions, carbon addition and increase in organic and dye concentration were not used due to the health of the UASB reactor. The ANNBCS (1) was tested on-line to maintain: steady BA concentration within the reactor, even during simulated failure of the BA monitor, which should maintain a constant level of pH, and a good UASB reactor effluent quality in terms of colour.

Two experimental runs were carried out to test the ANNBCS (1) on-line and similar results were obtained. The following parameters were monitored on-line: BA, TOC, pH, and colour. The TOC and colour analysers sampled the effluent from the UASB reactor. The dye step load and the non-addition of a constant flow of BA together with the STE were performed 1 h into experiment (Figures 5.14 to 5.18) and was then returned, to its initial level 7 hours after the step load started. Control was effected by peristaltic pumps (Watson Marlow 505U, Cornwall, UK) (calibrated as in Section 3.5.3). During Experiment 4.2 the pumps were controlled by the ANNBCS (1) with some of the LabVIEW™ code to integrate the remedial actions from MATLAB® and send them to the actuators being shown in Figure 5.8. The program written in MATLAB® for controlling the UASB reactor on-line is shown in Section B.3 - Appendix B.

The additional dye pump was controlled by BP(a) an integral part of ANNBCS (1). The concentration of the additional dye was stored at a concentration of 50 g l^{-1} and the maximum flowrate for the pump, provided when BP(a) suggested a 5 V output, was 9.925 g l^{-1}, so that a total of 10 g l^{-1} could be pumped to the reactor when joined with the constant flow of dye in the concentrated STE. During the same time period no BA was pumped with the STE. Instead the BA addition was controlled by the combination of the 3 ANNs: LVQ and both BP(b1) and BP(b2). These three elements of the ANNBCS (1) controlled the pump for the concentrated BA solution (60 g $NaHCO_3$ l^{-1}). This solution was prepared just before the experiment took place and its concentration was chosen so that it was as close to saturation as possible so that the smallest alteration to the UASB reactor HRT could be achieved. From Experimental Phase 3, it was known that increasing the STE dye concentration would not affect the levels of BA and pH within the UASB reactor. The main objective in controlling the BA level was to be able to test the predictions on-line of the hybrid part of ANNBCS (1) during BA monitor simulated failure.

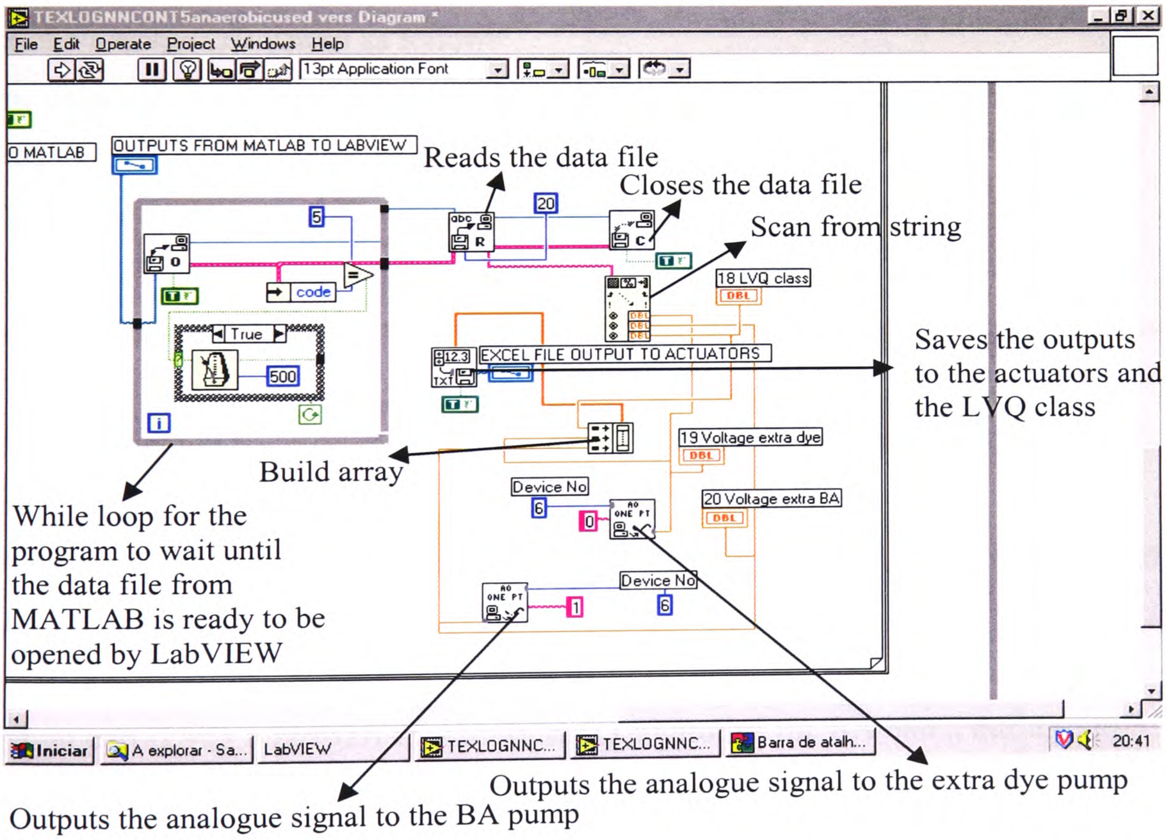


Figure 5.8 – LabVIEW™ VI code to integrate data from MATLAB® (ANNBCS (1))

Training of ANNBCS (1) to Control the UASB Reactor

Four ANNs were designed and trained which made up ANNBCS (1) to control the UASB reactor on-line. Three of them were the standard 3-layer FFN using the fast BP algorithm (i.e. BP(a), BP(b1) and BP(b2)), the fourth was the LVQ network for classification of the functional status of the BA monitor (working or failing) and to subsequently select the appropriate BP network either (b1) or (b2). Two classes were found sufficient to classify the BA and pH sensorial data, in order to control the BA feed rate and also detect if there was a loss in the BA sensor. If the colour readings would be included in the LVQ classification network the number of classes would have to be increased and most probably ANNBCS (1) would have less accurate predictions. The design and training parameters of the ANNs are presented in Table 5.13. The training input and output sets ranged as indicated in Table 5.14.

Measurement of on-line TOC could be used as another input to BP(a), however since there was no knowledge of the average OD and TOC relationship when the additional dye concentration was in place, TOC measurements could not be used. BP(a) was trained based only on the average OD values as follows:

- the delivered flow would be decreased accordingly for an average OD between 0.7 and 1.65;
- the flow would be completely turned-off for an average OD above 1.65.

The hybrid part (LVQ + BP(b1)) of ANNBCS (1) that controlled the BA pump was trained as follows:

- the pump would be stopped for values of pH and BA above 8 and 2250 mg $CaCO_3$ l^{-1}, respectively;
- there would be a variable flowrate of BA solution for values between 6.8 and 8 of pH and 800 and 2200 mg $CaCO_3$ l^{-1}.

The hybrid part (LVQ + BP(b2)) of ANNBCS (1) controlled the BA pump during simulation of BA monitor failure (i.e. assumed to be when BA < 700 mg $CaCO_3$ l^{-1}). During this situation BP(b2) would take only the values of pH into consideration and not the BA monitor measurements since they were not valid (Tables 5.13 and 5.14).

Table 5.13 - Structure and training parameters of ANNBCS (1)

Type of ANN	**Data sets**	**Input features (sensor measurements)**	**Hidden nodes**	**Network outputs (V to pumps)**
		Control of extra dye pump		
BP(a)	110	1 (UASB reactor effluent OD)	30	1
		Control of BA pump		
BA monitor working **BP(b1)**	31	2 (UASB reactor BA and pH)	30	1
BA monitor failing **BP(b2)**	20	1 (UASB reactor pH)	35	1

Table 5.14 - ANNs input and output range of ANNBCS (1)

Type of ANN	**ANN Input range**	**ANN Output range**
BP(a)	Av. OD [0 to 5] (OD units)	Dye pump [0 to 5] (V)
LVQ	pH [6.8 to 8] (pH units) BA [0 to 2300] (mg $CaCO_3$ l^{-1})	2 Binary Classes [0 1 or 1 0])[1]
BP(b1)	pH [6.8 to 8] (pH units) BA [0 to 2300] (mg $CaCO_3$ l^{-1})	BA pump [0 to 5] (V)
BP(b2)	pH [6.8 to 8] (pH units)	BA pump [0 to 5] (V)

[1] There was a 'software switch' which translated the binary values as follows:
[0 1] → Class 1 → BP(b1); **[1 0]** → Class 2 → BP(b2)

The setup and calibration of the two control pumps can be observed in Table 5.15 (Section 3.5.3). It is important to notice that in order to maintain the BA and pH values, within the UASB reactor, of 1700 mg $CaCO_3$ l^{-1} and 7.4, an equivalent of 0.545 ml min^{-1} of the BA solution had to be pumped (i.e. ANN output for the BA pump of 2.5 V).

Table 5.15 – Control pumps setup and calibration for control of the UASB reactor

Designation of the pumps	**Maximum voltage (V)**	**Maximum speed (RPM)**	**Maximum flowrate (ml min^{-1})**
BA pump	5	12	1.090
Additional dye pump	5	25	4.506

Note: Minimum voltages and corresponding flowrates were 0 V and 0 ml min^{-1}, respectively.

The three BP networks were trained as before and achieved a satisfactory SSE of 0.02 after training. The LVQ network was trained to recognise two distinctive patterns with a total of 51 data sets. It was constructed with 8 neurones (4 neurones for each subclass in the competitive layer). As explained previously, the prediction accuracy depended on the number of times the data was presented to the network. From observation of the training plot after

1500 presentations (Figure 5.10) the learning had stabilised, therefore 1500 was felt to be the adequate number of presentations to adopt. The initial learning rate was set to 0.1 and the bias time constant of 0.99. Class 1 contained data where the intermittent BA monitor was fully operational (values above 700 mg $CaCO_3$ l^{-1}) and the second class included data when the monitor was faulty (values below 700 mg $CaCO_3$ l^{-1}). The corresponding BP networks associated with the two possible operating conditions, were the fully functional and faulty BA monitor were BP(b1) and BP(b2), respectively.

All the networks had the input and output data normalised to values between 0 and 1, except the outputs of the LVQ network, as the outputs were of the binary form. The training sequence for the four networks is presented in Figures 5.9 to 5.12.

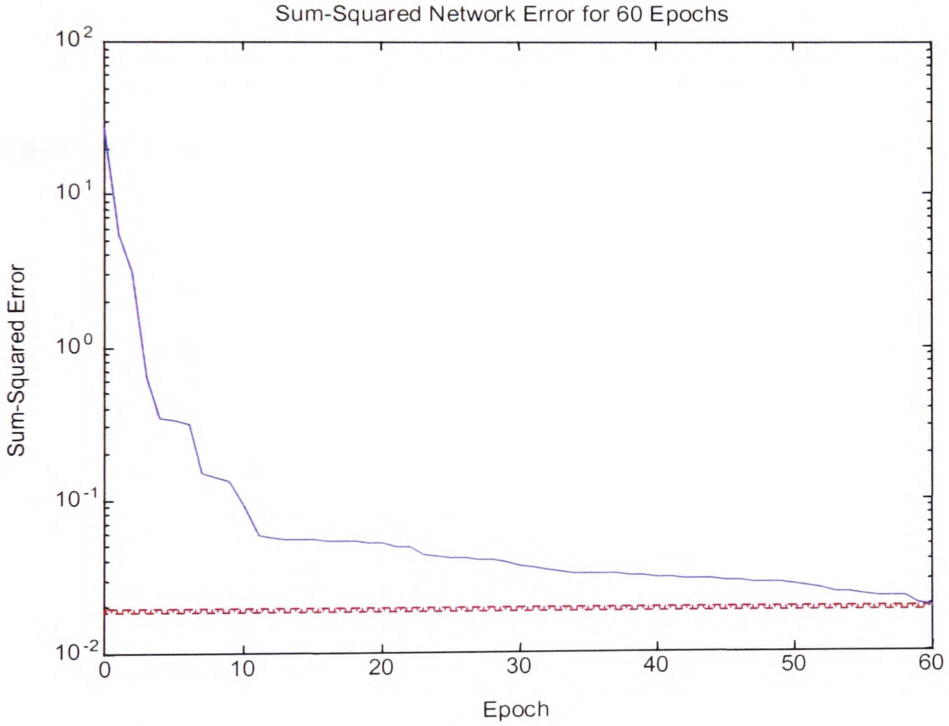


Figure 5.9 - Training sequence for BP(a) vs. SSE

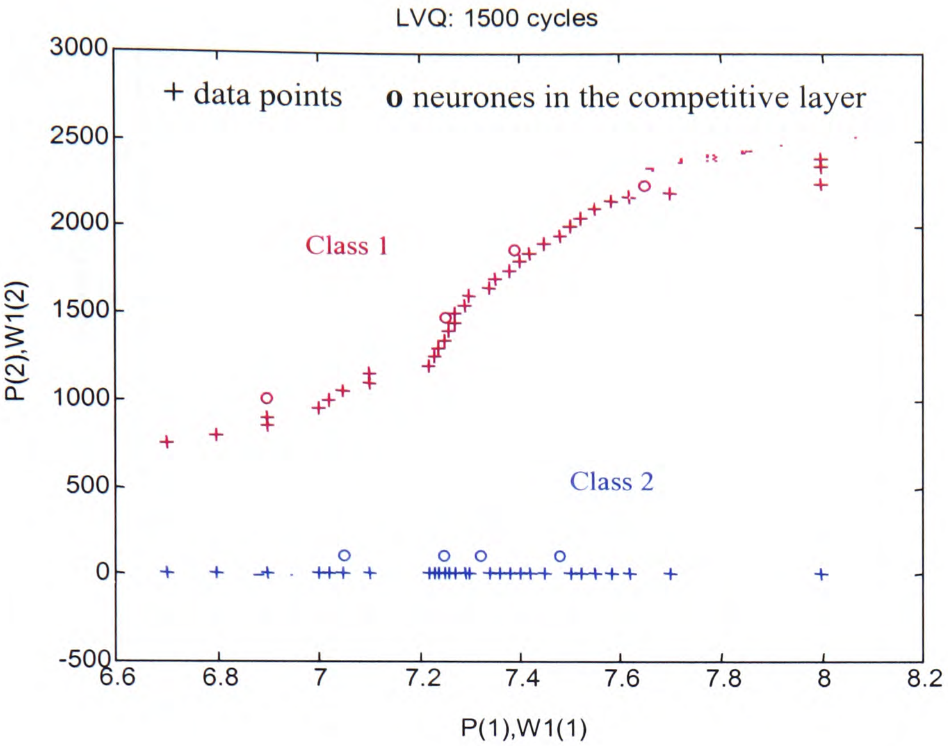


Figure 5.10 – Final position of the competitive neurones after 1500 epochs

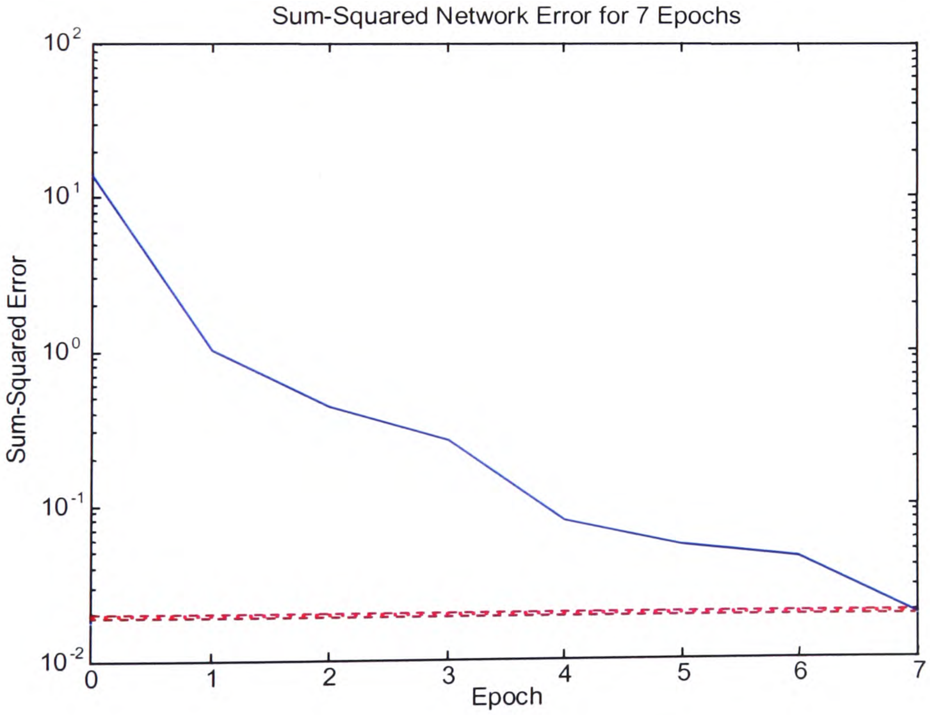


Figure 5.11 - Training sequence for BP(b1) vs. SSE

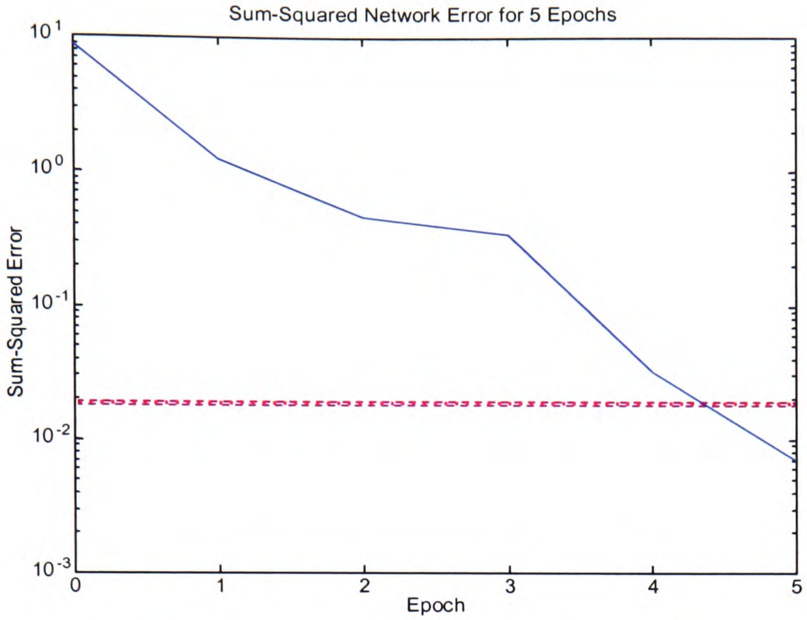


Figure 5.12 - Training sequence for BP(b2) vs. SSE

Results and Discussion of the On-line Control Experiments

Figure 5.13 shows a screen capture of the panel of the LabVIEW™ VI during the second run of Experiment 4.2.

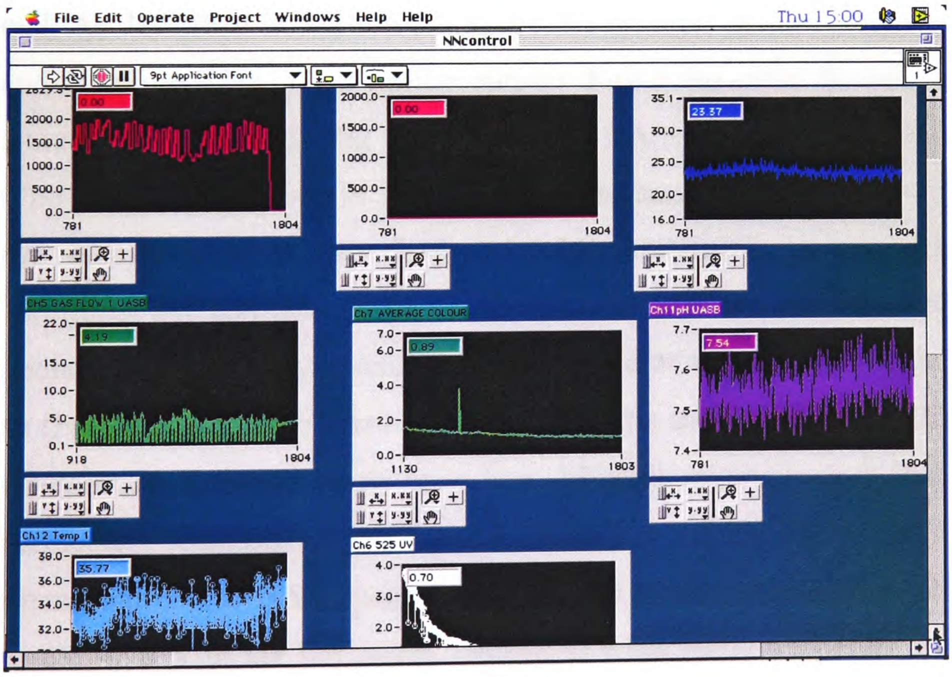


Figure 5.13 – Screen capture of part of the LabVIEW™ VI panel during the second run of Experiment 4.2

UASB Reactor BA and pH

During Experiment 4.2 there were variations in the on-line BA levels in the UASB reactor. These variations were due to the irregularity in the volume of the sample that reached the instrument. This phenomenon was caused by the rapid flowrate used by the re-circulating pump which was necessary in order to promptly remove the granules so as to avoid them being deposited in Filter 2 (Section 3.4). Therefore, during the entire experiment the BA monitor provided measurements that were not correct. However, the measurements were maintained within a range which ANNBCS (1) was not trained as being failure conditions. Consequently, in order to test the hybrid part of the control scheme a simulation of the BA monitor failure had still to be performed (i.e. BA values lower than 700 mg $CaCO_3$ l^{-1}).

Despite the BA measurement variations, ANNBCS (1) behaved quite sensibly throughout the experiment. Off-line BA measurements (Jenkins *et al.*, 1983) were performed every hour and an average value of 1830 mg $CaCO_3$ l^{-1} (*sd* = 85, n = 9) was found indicating that as in Figure 5.14 only the highest levels recorded by the on-line BA monitor were realistic. The lower values obtained by the analyser were due to less sample being pumped to the reaction chamber. The constant level of pH within the upper part of the UASB reactor can also be observed in Figure 5.15, indicating that changes in the BA measurements were not completely real. As described in Section 3.3.3, the BA monitor operated intermittently every 30 minutes and therefore BP(b1) had similar outputs during the entire 30 minute period. The variations of the ANN outputs during the time where BA measurements remained constant were due to the small fluctuations in the pH measurements. From the arrow labelled start until the arrow labelled end (Figures 5.14 and 5.15) ANNBCS (1) was actuating on the pumps (i.e. additional dye and BA). However, before and after the arrows ANNBCS (1) was outputting predictions based on the sensorial measurements without controlling the pumps. Its predictions (Figure 5.14) in the first half-hour before the start of the controlled experiment showed how important it would be to have two different measurements that can provide similar/complementary information to a control scheme. It can be seen in the first half-hour of the experiment that if only BA measurements were to be considered by ANNBCS (1), the predictions would be 0 V to the BA pump, instead of what really happened because BP(b1) also considered the pH measurements.

As shown in the Figure 5.14, there was a simulation of BA failure where the instrument output was set to 0 mg $CaCO_3$ l^{-1}. The LVQ network correctly classified the 'failure' and selected BP(b2) in order to deal with the problem as represented by Class 2 in Figure 5.15. BP(b2) predictions were quite noisy in comparison to the BP(b1) as they were related only with pH measurements and not to the BA values since these were understood not to be correct.

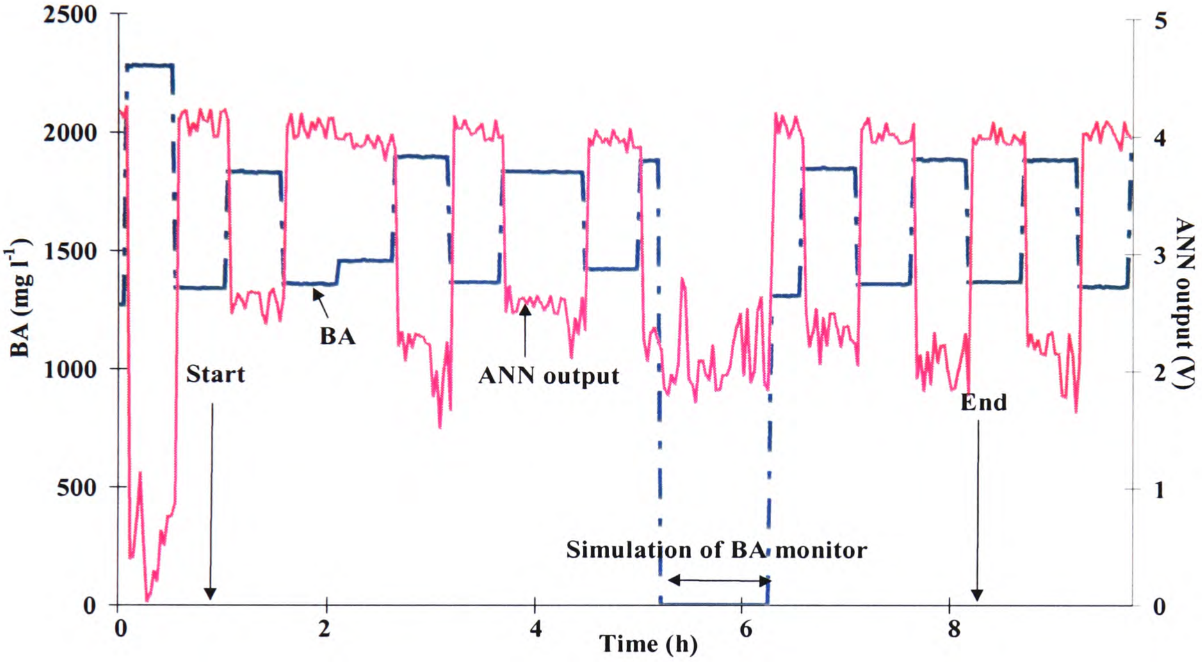


Figure 5.14 - ANN control output (BP(b1) and BP(b2)) vs. UASB reactor BA level

It would be very important to have various monitors capable of providing complementary information so as to enhance the tolerance of the ANNBCS. It is well known that WW sensors can fail quite frequently and the ANNBCS has to be able to cope with these losses. Even if not all the sensor failure conditions are to be taken into account at least the most common ones should be. For example a similar intermittent BA monitor to the one used here, could return various unreal measurements:

- higher readings due to foaming or to slacking of its feeding tube or air leaking into the reaction chamber;

- lower readings due to a tube blockage, air bubble trapped and as in this case the re-cycling pump for the filter produced a sucking effect of the sample, decreasing the amount of flow to the monitor;
- zero or constant readings due to an electrical failure of the monitor or a computer software problem.

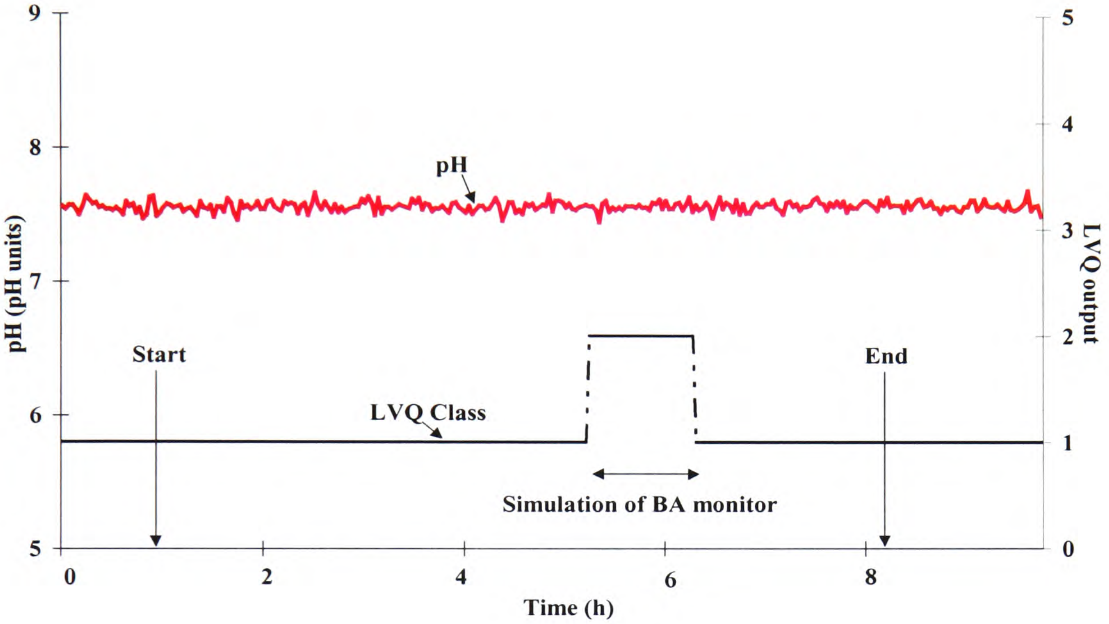


Figure 5.15 - UASB reactor pH and LVQ network output (Classes 1 or 2)

UASB Reactor Effluent Colour

Figure 5.16 shows the response of the BP(a) network output in attempting to reduce the dye addition when the colour quality of the UASB reactor effluent deteriorated. As there was only one flowcell that could be read by the spectrophotometer, and the UASB reactor was fed with a STE (i.e. the OD and the TOC of the feeding WW were known) it was decided that the spectrophotometer would measure the UASB reactor effluent only. This brought known complications for the controller. The increase in colour and TOC content of the UASB reactor effluent was not immediate due to adsorption, degradation, sampling delay (approximately 16 minutes) and mostly due to the flow dynamics of the reactor. In real life the OD and TOC of the feed would have to be measured as well and be input to the ANNBCS so that these two parameters would dictate the major remedial actions to be taken. In order to safeguard the health of the reactor, compliance with legislation and to be able to reuse the WW by the industrial process if required, the feed dye load would have to be reduced prior to its entrance in the reactor. This reduction would be based on the measured OD of the reactor feed and the known average reactor removal efficiency. Small adjustments could then be made from the observed reactor efficiency at a particular time based on the measurement of the UASB reactor effluent, as for example:

- removal efficiency has increased due to biomass growth then an increase in dye load in the feed is possible or;
- removal efficiency has decreased due to loss of biomass or the effect of a toxic agent, then the dye load in the feed has to be reduced.

A combination of remedial actions such as reduction of the colour load, addition of a carbon source, addition of biomass and addition of nutrients could actually be performed by the ANNBCS.

The main objective of this experiment was to assess the generalisation capacity of ANNBCS (1) as only the UASB reactor effluent colour and TOC were measured and only the reduction of the dye concentration was considered as a remedial action. It can be seen from Figure 5.16 that, the additional dye pump was only switched off after 6.3 h from the start of Experiment 4.2. However the flow was gradually reduced 4.5 h from the start of the experiment.

Apart from the good generalisation capability of the ANNBCS (1) a positive control effect could also been observed. The pump for extra dye was completely switched off before this was done manually and the flow started to reduce 1 h before that. The effect of the ANNBCS (1) could be more significant if the additional load of dye instead of being available for a 7 h period would be available for one whole night e.g. 14 hours. However, when operating with real effluent where the dye concentrations are unknown the colour analyser would be measuring the influent and then a much efficient ANNBCS control action would easily be achieved. Figure 5.17 shows that the maximum average of the UASB reactor effluent OD was attained approximately 13 h after the commencement of the step increase in the dye. It also shows the time required for near full recovery and the correlation between on-line measurements and the five off-line measurements (true colour) of the average OD, which correlated well with the introduction of the 60 μm mesh.

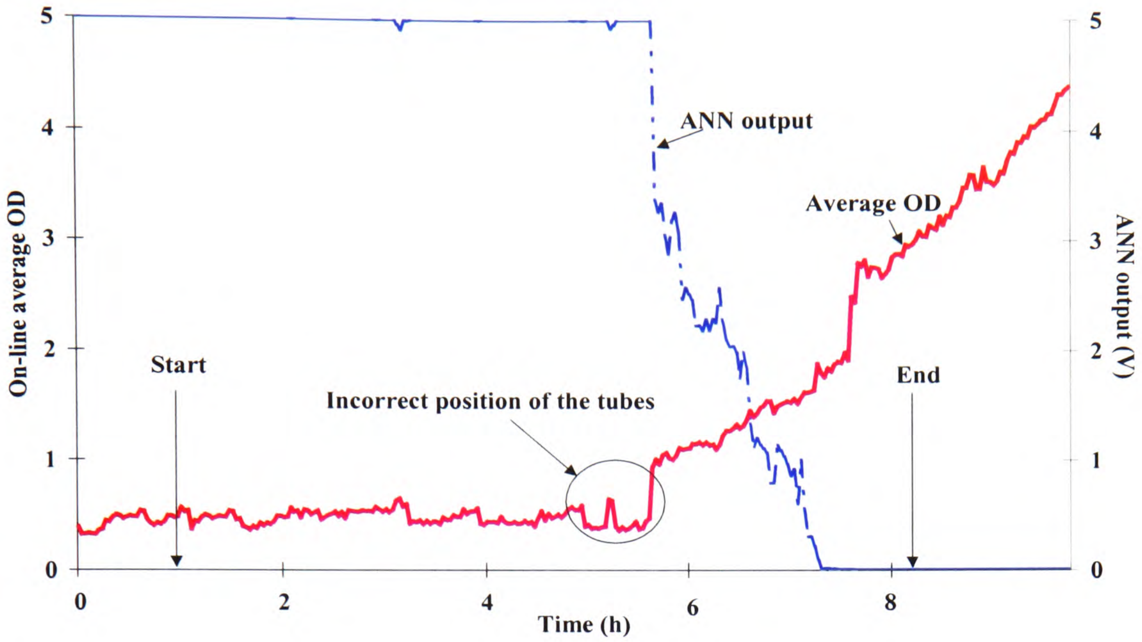


Figure 5.16 - ANN output (BP(a)) vs. on-line UASB reactor effluent average OD

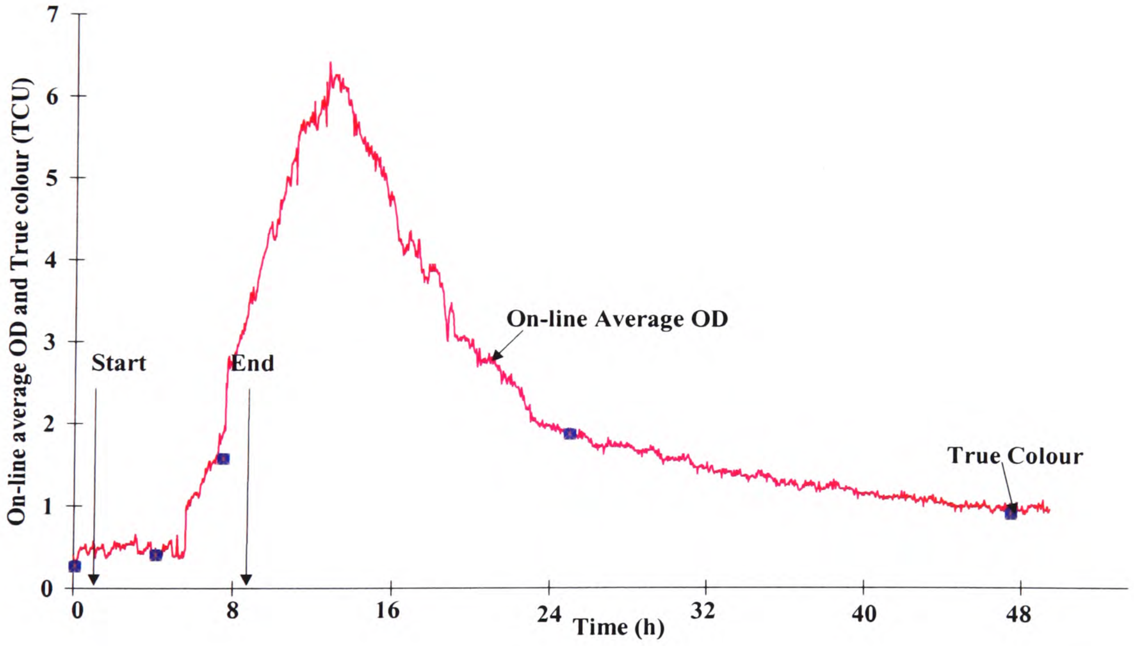


Figure 5.17 - On-line average OD and off-line true colour

UASB Reactor Effluent TOC

The additional dye contributed to the increase in the organic content fed to the UASB reactor, which was detected by the measurements of the UASB reactor effluent TOC. Figure

5.18 shows the TOC evolution during the experiment. TOC values were filtered using a 4th order low-pass Bessel filter constructed with LabVIEW™ VI code. As it can be seen, during the two days experiment the TOC of the UASB reactor effluent increased from an average of 400 mg l^{-1} to a maximum of 1000 mg l^{-1} within 14 hours. It can be observed from Figures 5.17 and 5.18 that both values of the average effluent OD and TOC were still unsatisfactory 2 days after the step increase in the dye load. This was probably resulted from the dye being adsorbed onto the granules and its slow release.

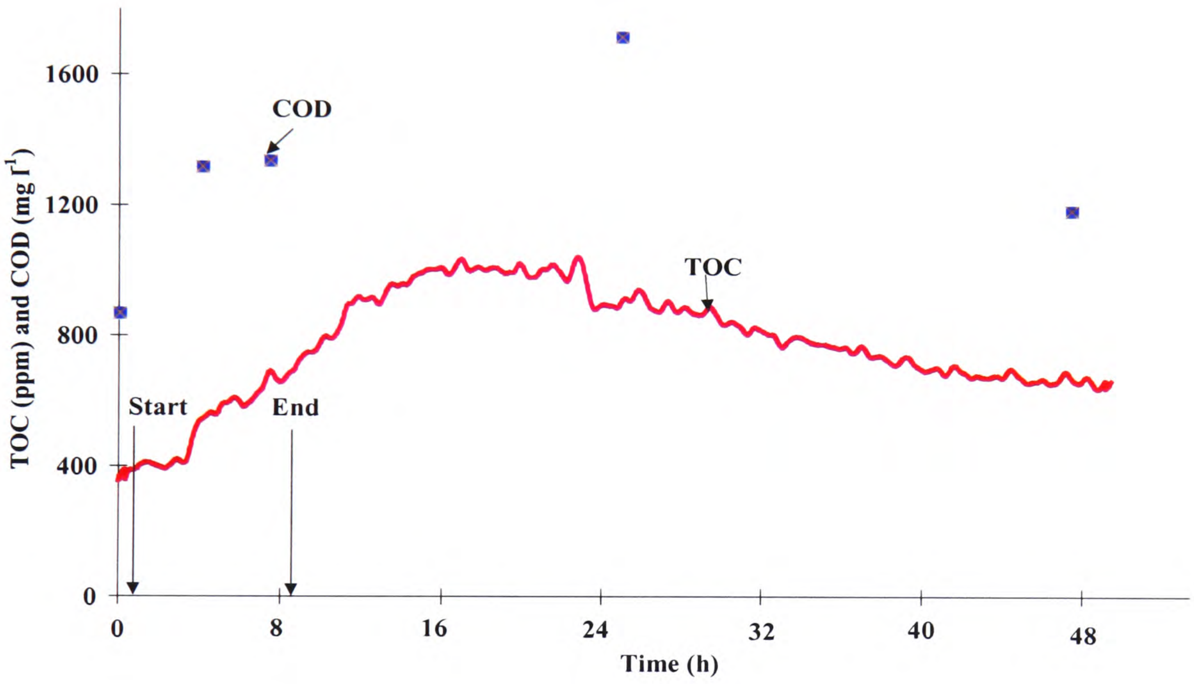


Figure 5.18 - Off-line COD vs. on-line TOC

The legislation concerning discharge consents does not yet refer to TOC limits, only to BOD and COD. In order to find a correlation between TOC and COD for this particular waste, off-line COD measurements were performed during these experiments. The off-line COD measurements can be related to the TOC, as shown in Figure 5.18. COD values as expected were higher than those obtained with TOC analysis. A COD:TOC ratio of 2.1 was determined. For control purposes it is important to use some sort organic content measuring technique. With the COD:TOC ratio, the TOC analysis could be performed and compared to the legislative restrictions. Ionics (1993) published COD:TOC ratios of 1.8 – 2.2.

5.3.2. On-line Control of the Aerobic Stage (ANNBCS (2))

The data of Experimental Phase 5 was selected and used to train the different networks involved in the control scheme has been defined in Section 4.4. In Section 3.6.5 there is a full description of the on-line monitoring used to gather the data and control setup adopted to test the prototype ANNBCS. The following two Sections will detail the training of ANNBCS (2), and the results of the on-line control of the aerobic stage.

Training of ANNBCS (2) to Control the Aerobic Stage

The control scheme objective was to optimise the pH and DO levels within the aerobic tank and also the TOC of the aerobic stage effluent. As explained earlier the extra starch due to difficulties was hydrolysed with NaOH rather than with amylase. This lead to the decrease of pH during Experiment 5.2, which would most probably not have occured when hydrolysed with amylase. Therefore, ANNBCS (2) would add acid (1 M HCl) in order to control the pH level increase within the aerobic tank. The maximum flowrates for each control action to be applied during the control experiment were as presented in Table 5.16 and zero as the minimum flowrates by the three actuators.

Table 5.16 – ANNBCS (2) control outputs based on the combined action of three separate ANNs (maximum voltages and flowrates)

ANNBCS	Actuators	Max. ANN output (V)	Max. corresponding flowrate (ml min^{-1})
1	Additional starch pump	1.7	0.5
2	Air compressor	5	7 000
3	Acid (HCl) pump	5	1.14

All the BP networks were trained using the same learning parameters as for the networks used for the on-line control of the UASB reactor. Neurones of the logarithmic sigmoid transfer function were also employed for each of the layers. They were of a SISO structure. Seventy-three data sets were used for all the networks. The three networks achieved a satisfactory SSE of 0.002 after training.

ANN Controller 1 – Consisted of one input element (TOC from the aerobic settler) and one output (voltage to the additional starch pump). The voltage would be reduced when the controller was informed of the decrease in the effluent quality. The values of outputs (i.e. voltage to the additional starch pump) considered for the training set were as follows:

- 1.7 V for TOC values below 100 ppm (fully on providing the maximum flowrate set for the calibration);
- 0 V (completely off) when the TOC of the effluent was above 600 ppm for Experiment 5.2 and 300 ppm for Experiment 5.3; and
- it would vary for values in between.

ANN Controller 2 – The second network had DO readings as input and the voltage to the additional air compressor as an output. For the case when the level of DO was lower than 3 mg l^{-1} the extra compressor fully switched on as the compressor was not of a variable type. As a consequence the training of the ANN controller was based on an on-off remedial action.

ANN Controller 3 - Its response was based on monitored values of pH in the aerobic tank. Similarly with the acid pump, the third ANN controller output consisted of the on-off response despite the fact that the pump for the acid was of the variable type. This simplistic on-off response was thought to be adequate since the sampling rate of 2 minutes was fast enough to deal with the size of the tank and the acid flowrate. Furthermore, the tank was well mixed due to the aeration motion. The acid pump would be activated should the pH level rise higher than 7.2, in order to deal with the increase of pH due to the NaOH used to neutralise the amylase hydrolysed starch.

Results and Discussion of the On-line Control Experiments for the Aerobic Stage

Two runs of Experiment 5.3 were performed as indicated in Section 3.6.5. During the first the TOC analyser was faulty (i.e. it did not hold the calibration and the injection arm was constantly getting stuck) and therefore ANNBCS (2) performed differently for the two runs.

TOC of the Settling Vessel Effluent and Aerobic Tank SCA

A similar step increase in the starch intake to the one of Experiment 5.2 was performed in order to test the ANNBCS (2) response with the TOC analyser being fully operational. The increased starch intake was available for 5 hours and was then manually turned off as indicated by the arrows in Figure 5.19. Prior to the start of the experiment (i.e. 18 h before) some drops of hydrogen peroxide were used to break-up the flocs so that there was not to much loss of solids (Metcalf and Eddy, Inc., 1991). Just before the start of the experiment the sludge MLSS concentration in the tank was quite high (approximately 3.5 g l^{-1}) and as the experiment progressed, significant sludge bulking problems were experienced at the top of the settler. The TOC analyser worked well throughout this experiment and there was actually an increase in organic degradation despite the poor quality of the sludge. This efficiency enhancement had repercussions for the ANN control action. The corresponding controller output when the TOC value was above 300 ppm was set to 0 V. Due to this treatment efficiency improvement the effluent TOC was only above 300 ppm after the additional starch pump had been manually switched off. In between the start and the end, the controller did try to regulate the additional flow of starch, although its actions were not restrictive enough as can be seen in Figure 5.19. Tighter control should have been applied during training of the first ANN (e.g. full turn-off of the extra starch pump for TOC measurements higher than 150 ppm).

As the sludge was very fine it inevitably lead to bulking at the top of the settler where the sampling point of the TOC analyser was located meaning that both Filters 2 and 3 were significantly blocked at the end of Experiment 5.3. These filters had to be cleaned and hence no recovery period was monitored. However, there was very little difference between Experiment 5.2, which was uncontrolled (Figure 4.20) and Experiment 5.3, which was controlled (Figure 5.19). Figure 5.20 shows a similar effect to that of Experiment 5.2, between the SCA and the TOC levels. However, in this case the five SCA results were slightly higher than the ones during Experiments 5.1 and 5.2 and this could have been due to one or a combination of the sludge condition and the addition of the hydrogen peroxide to break-up the flocs. The DO levels were also higher in the tank in comparison with Experiment 5.2, but this would possibly indicate a lower and not higher biomass catalase activity.

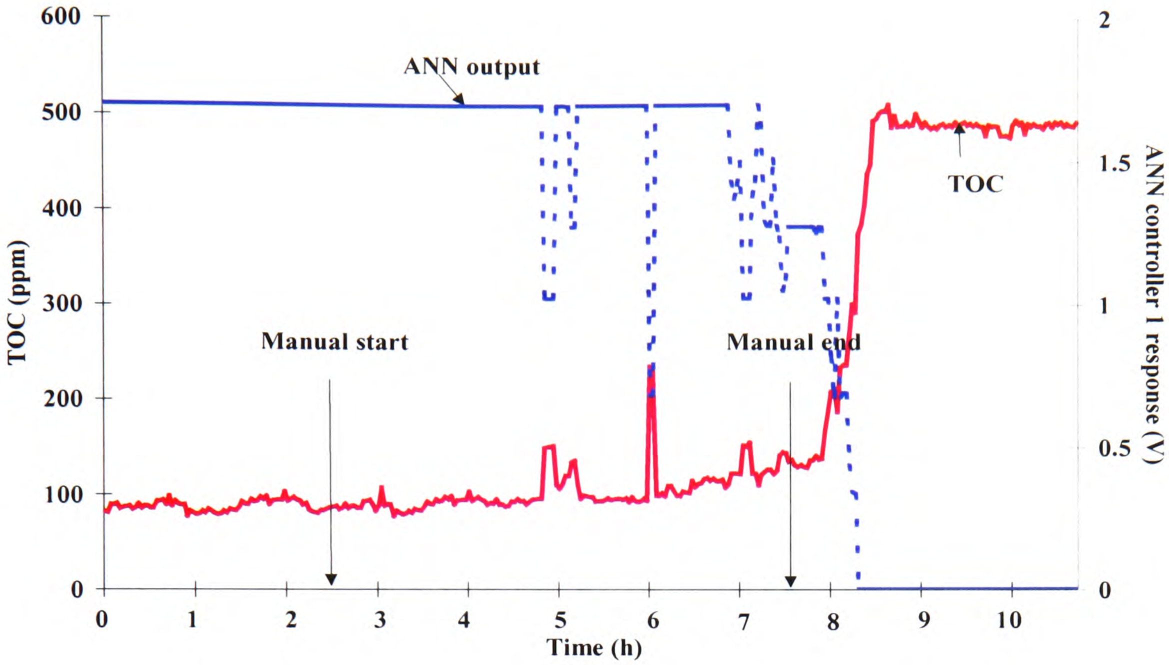


Figure 5.19 - ANN control of the aerobic stage effluent quality (Experiment 5.3)

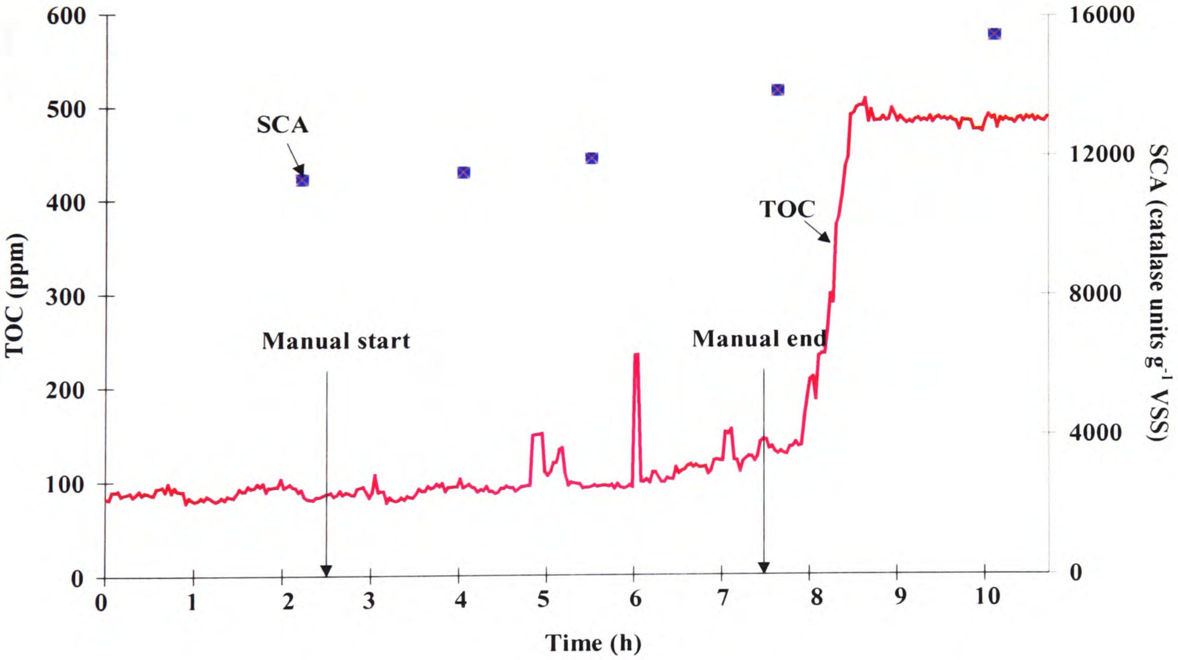


Figure 5.20 - Aerobic stage effluent TOC vs. SCA of the aerobic tank (Experiment 5.3)

Aerobic tank DO and pH

The other parameters that the ANNBCS simultaneously controlled were the DO and the pH in the aerobic tank. There was no need for pH control throughout the second run of Experiment 5.3, which was safely maintained within the range of 7 to 7.18 and also no effort was required to control the level of DO control (Figure 5.21). The DO results were filtered using a 4th order Bessel filter so to attenuate the effect of the large bubbles, which impinged on the DO probe. However, the demand for oxygen through the experiment varied. The DO increased just after the commencement of the increase in starch intake and decreased as soon as ANN Controller 1 started to reduce the additional starch pump speed and was maintained at a slightly lower level than at the start of this experiment.

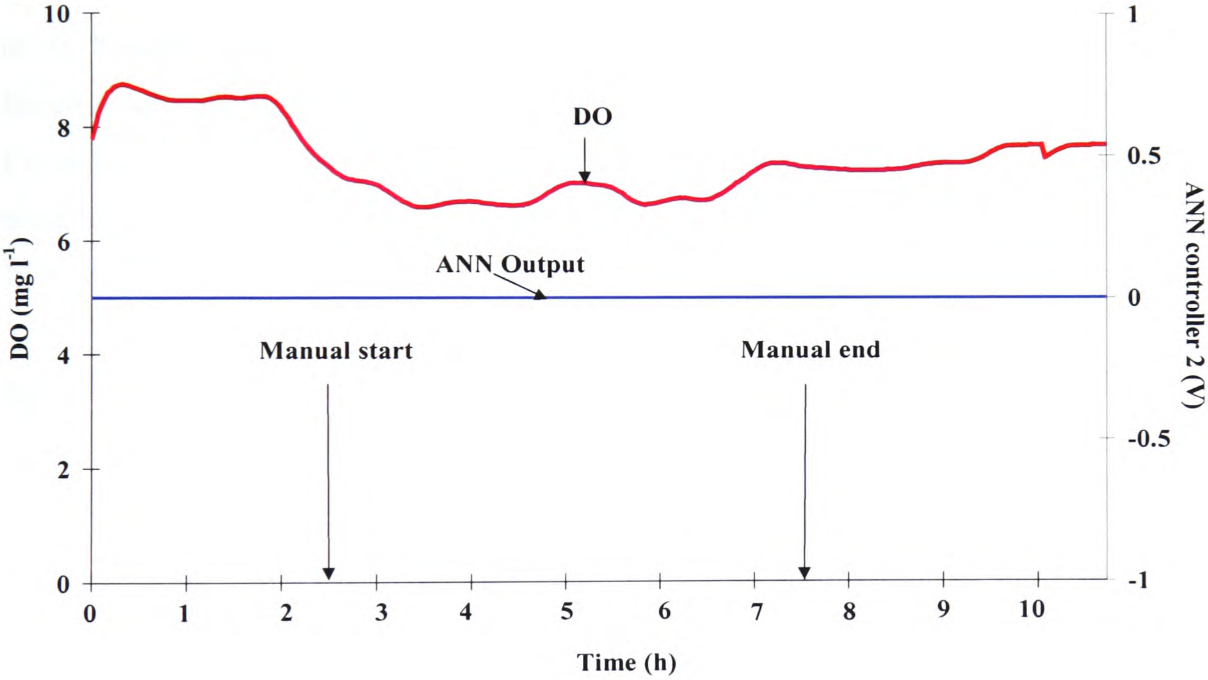


Figure 5.21 - ANN Control of the DO in the aerobic tank (Experiment 5.3)

5.3.3. Conclusions from the On-line Implementation of the ANNBCSs

The ANNBCSs (1 and 2) used here were successful as they suggested the appropriate remedial actions and responded accordingly to the training examples. The on-line control examples highlighted the need for complementary parameters with which to establish a control action since most of the on-line analysers used in WWTPs are still quite unreliable. The provision of additional information to the ANNBCS would definitely increase the tolerance to any loss of information.

Sensors for TOC and colour have to measure both the influent and effluent of the UASB reactor and aerobic stage. The information provided about the influent and effluent in terms of organic and colour loads would be used by the ANNBCS in order to balance remedial actions, as for example: diversion/dilution, addition of nutrients, GAC or powered activated carbon (PAC) addition to the UASB reactor, addition of carbon source, variation of the RAS, re-treatment of the effluent. The influent characteristics have to be known so as to decide on the remedial actions before it flows to the system and also during the treatment process. It is very important to know the characteristics of the WW before it is allowed inside the system. This is because any system has a maximum treatment capacity no matter how many enhancing substances or actions are used. At the same time other remedial actions could be put in place to cope to the adverse WW characteristics. The additional sensing requirements needed to ensure robust monitoring and control are significant additional costs and perhaps only more stringent legislation will force the increased use of instrumentation.

Despite the aerobic biomass condition i.e. sludge bulking, its activity followed the increase in organic load to the system.

6. MODELLING OF THE UASB REACTOR USING ANNs AND FURTHER EVALUATION ON THE ANNBCS PERFORMANCE IN A COMPUTER SIMULATION

The 'black-box' modelling technique using ANN is adopted here to model the behaviour of the UASB reactor via the use of data gathered from the UASB reactor operation during a period of 7 months (i.e. Experimental Phase 3 except Experiments 3.5, 3.6, 3.8 and 3.9). The Neural Network Based Identification Toolbox developed by Nørgaard (1995) has been extensively used in this Chapter for the system identification task. Also an ANNBCS was constructed using the same hybrid structure as in Chapter 5 (i.e. LVQ + BPs) using the Neural Network Toolbox by Demuth and Beale (1994). The two toolboxes were both for use with MATLAB®. The 'black-box' models were subsequently evaluated with unseen data and their prediction accuracy verified before being used to further evaluate the ANNBCS within a computer simulation environment. The ANNBCS was re-trained with 3 inputs (TOD, average OD and CO_2) and 2 outputs (adjustment in starch and dye loads).

6.1. The Purpose of the Chapter

This Chapter serves to demonstrate the feasibility of using the ANNBCS developed in this work to optimise the treatment achieved by the UASB reactor in relation to organic and colour loads. One on-line control experiment was successfully carried out on the UASB reactor to evaluate the ANNBCS performance in coping with a sudden step load in the STE dye concentration (Chapter 5). Unfortunately no further on-line control experiments could be carried out, as the health of the UASB reactor deteriorated after Experimental Phase 3. Therefore the concentration of the starch and dye had to be decreased to lower values than the ones adopted during Phase 3. Several attempts to increase the loading rate of the UASB reactor to previous levels were performed but with little success. During these starch increases the VFAs in the reactor built up and the pH decreased quite quickly which indicated reactor instability (Chapter 4). Also the bed of granules rose to the top of the reactor and at this point it was decided that the reactor was no longer usable for starch step loads.

As a large number of experiments were undertaken where the starch and dye concentrations were changed in a step manner with the response of the UASB reactor being measured by instruments such as the TOD analyser, UV/Visible Spectrophotometer and a CO_2 analyser, neural models of the anaerobic process could be developed. This would enable further evaluation tests to be carried out on the ANNBCS in simulation. Although comprehensive on-line control experiments were not possible, the advantage of the simulation work on the ANNBCS using models was that it would be possible to better investigate the controller's performance.

It has been widely claimed that ANNs are able to model non-linear and time varying processes (Narendra and Parthasarathy, 1990; Chen and Billings, 1991). The following sections are dedicated to investigating the performance of the ANNBCS in a computer simulation environment during the following three different operating conditions:

- Low dye and starch concentrations;
- High dye and low starch concentrations;
- High dye and starch concentrations.

6.2. Background to the ANN Based System Identification

Identification is about finding a model that best regenerates the original output signals when subjected to the same input signals based on previous observation (e.g. Ljung, 1987). It is a tool, which could be utilised to 'model' a process without having to take into account the many complex physical laws that govern the system. Identification using ANNs in many ways is similar to the parametric identification approach, as multi-layered ANNs are versatile non-linear maps (Chen and Billings, 1991). These identification networks are techniques to estimate the parameters in a given model structure, which basically entails a numerical search for suitable values of the associated parameters of the model in order to deliver satisfactory results (Ljung, 1995). In the case of system identification using FFNs, the number of neurones in the hidden layer(s) will have to be pre-allocated depending on the complexity of the input-output relationship, and this is generally determined through trial and error. The task of system identification using ANNs essentially involves finding a suitable model structure and subsequently finding good numerical values for its parameters (weights and biases of the network). The theoretical basis of non-linear modelling by using ANNs has been well established in the last decade by authors such as Hornik *et al.* (1989). They have shown that a 2-layer FFN hosting sufficient numbers of neurones (of continuous, bounded and non-constant activation function) in the hidden layer can approximate any continuous function.

The work involved in identifying the dynamics of a process can be summarised into four sequential basic steps:

Step 1: gathering experimental data;

Step 2: selecting a model structure;

Step 3: estimating the model (i.e. find suitable model parameters); and

Step 4: validating the identified model with new data sets.

Step 1 – One vital requirement in such technique is that the training data should cover the entire operating region of interest and is gathered with the correct choice of sampling frequency (Ljung, 1995; Nørgaard, 1995). All the dynamic modes of response should be reflected in the gathered data collected, which will be used to teach the 'black-box' ANN model. Additionally, this information must come from a continuous stream of data gathered from the same experiment in order to avoid any discontinuities or irregularities in defining the dynamics of the process.

Step 2 - In this work an ANN of the MLP architecture was adopted as the framework of the model structure. In addition to the adopted model structure, the tasks also involved choosing an appropriate set of regressor vectors (Nørgaard, 1995) (since the process to be identified was non-linear in nature). The ARX regressor structure was chosen based on its popularity in linear system identification and hence was extrapolated to include the ANN models. More sophisticated regressor structures could well reduce the error during training but the added complexity of the model was not justified by the possible improvement in forecasting

(Premier *et al.*, 1997). The regressor structure ARX uses previous inputs and outputs in order to deliver an output prediction at one or more sample periods in the future (Ljung, 1995; Premier *et al.*, 1997). The function *nnarx* in the Neural Network System Identification Toolbox, allows the user to utilise such a regressor structure in the context of ANN modelling, and therefore the models were denoted as NNARX models. The associated regressor vector and the predictor terms are defined as:

$\varphi(t) = [y(t-1)...y(t-n_a), u(t-n_k)...u(t-n_b-n_k+1)]^T$ and $g(\varphi(t), \theta)$, consequently.

Where:

$\varphi(t)$ is a vector containing the regressors;

θ is a vector containing the weights and biases of the network;

g is the function realised by the ANN model through learning;

n_a and n_b denote the number of past outputs and inputs required, respectively;

n_k denotes the delay of the system, expressed as the number of sampling periods.

There are other advantages according to Nørgaard (1995) in using the *nnarx* function. His work stated that it has a static predictor (feed-forward with no feedbacks), which is more stable in its prediction than other model types, which are recurrent (i.e. the future network inputs depend on past network outputs) and further suggested its use as a general rule of thumb. In addition, the presence of noisy signals can be considered to be insignificant as the raw signals can be filtered prior to its use by the models, which further justifies the use of the *nnarx* function (Nørgaard, 1995).

Step 3 - When developing NNARX models there are difficulties that can be encountered. The tasks of choosing the appropriate order of the model (i.e. number of past inputs and outputs that the model has to take into consideration) and also the output delay (i.e. time taken for the output of the system to start changing) are relatively laborious for non-linear processes. In addition, other parameters such as the number of neurones in the hidden layer and the desired error goal will have to be determined empirically. However, in the absence of more precise knowledge, some intuitive physical insight will to some extent enable the designer to suggest the orders and delays of the system (Nørgaard, 1995; Ljung, 1995).

Step 4 - Once the 'black-box' model has been constructed with the desired error goal during training, the final stage is to validate it. This involves passing unseen data to the model and then comparing the actual responses to the model predictions. The average sum squared error (ASSE) between the actual response and the model prediction was used by Chong (1999) and Chong *et al.* (2001) to evaluate the model's predictions and is defined below:

$$\mathrm{ASSE} = \frac{\sum_{i=1}^{N}(Y_i - y_i)^2}{N}$$

Where: Y_i is the actual plant response; y_i is the model prediction and N is the number of samples within the data set.

The comparison of the ASSE for the testing and validation exercise is vital, as it provides an indication of the model's ability to generalise. In linear system identification, the auto-correlation function of the residuals and cross correlation function between the input signals and residuals are employed to quantify the model's performance. In the case of non-linear models, the main approach is by a visual comparison of the predictions of the derived model with the plant response of the validation data sets. Therefore, the choice of the test data must be carried out carefully in order to fully investigate the predictive ability of the model.

Should the predictions prove to be unsatisfactory, then the designer would have to refer back to the two previous steps in the identification procedures (i.e. steps 1 and 2). Very often this will involve a single or combined action such as: choosing a different regressor structure; altering the delays, number of past inputs and outputs; or adopting a different network architecture, for instance the number of neurones in the hidden layer. Unfortunately, this is an empirical exercise that has to be carried out until satisfactory results are obtained. If the predictions still proved to be unsatisfactory, additional feature(s) could be added as inputs to the model in order to improve the ANN learning of the functional relationship (Premier *et al.*, 1999). Again, the inclusion of additional feature(s) hinges solely on the degree of influence that these parameters have on the process being modelled and again is primarily based on the experience of the expert operator(s).

6.3. Feed-Forward MLP Neural Network – Architecture of the NNARX Models

Numerous ANN applications in system identification and data classification involve the so-called FF MLP ANNs (Chong, 1999; Narendra and Parthasarathy, 1990). These are, at present, the most common type of ANN in use and have been utilised in numerous practical applications (Demuth and Beale, 1994). The widespread use of such a network is due to its ability to model complex functional relationships between the given input and output data sets by learning from examples.

In these types of networks, the signals only flow in one direction, from the input to the output (final) layer. There are no feedback connections between the individual layers and no association between the neurones (simple processing elements) in each layer and therefore the MLP network is only capable of statically mapping the input vectors to their corresponding targets. However, they are still widely used in dynamic system identification by feeding the past input and output values of the system to be modelled as inputs to the network. This can be achieved by employing the tap-delay-line technique and using the next time step output of the system as the target output (Narendra and Parthasarathy, 1990), hence converting temporal modelling problem into a spatial modelling problem.

A 'black-box' model approach is beneficial in situations where the relationships between the input and output data carry heavier emphasis than an in-depth understanding of the process under study (Ljung, 1987; Premier *et al.*, 1999). The obstacles in formulating mathematical models based on the underlying dynamics of biological treatment processes are well known, and much literature has been reported with respect to this issue (Section 2.9). Figure 6.1 depicts the architecture of the MLP network employed for the modelling work.

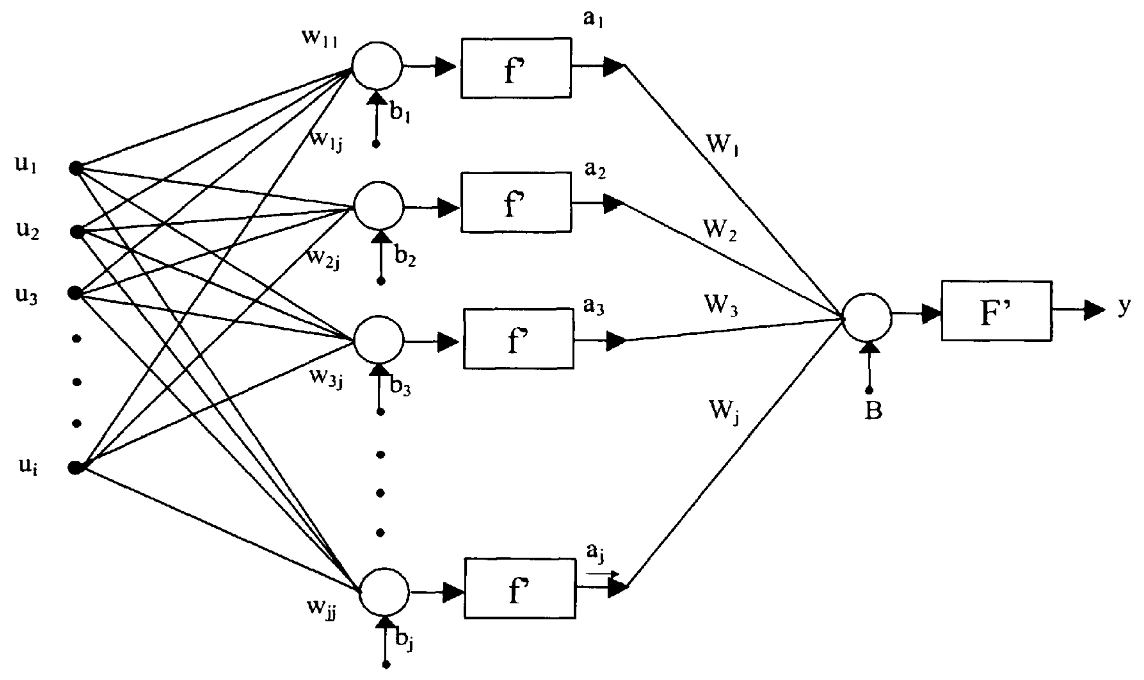


Figure 6.1 -The FF MLP network architecture for the NNARX model structure

From Figure 6.1 the following can be established:

$$y(u,w,b,W,B) = F'\left(\sum_{j=1}^{m} a_j W_j + B\right) = F'\left(\sum_{j=1}^{m} W_j f'\left(\sum_{i=1}^{n} u_i w_{ji} + b_j\right) + B\right)$$

Where:

n is the number of features in the input vector;

$y(u,w,b,W,B)$ is the model prediction, as a function of the network inputs, weights and biases;

F' is the linear activation function of the output layer;

W_j are the weights of the connections between the hidden and the output layer;

B is the bias of the output layer;

f' is the activation function of the hidden layer (hyperbolic tangent);

w_{ji} are the weights through which u_i is connected to the hidden neurone j and b_j is the bias of the hidden layer;

m represents the number of hidden neurones;

u_i represents the feature input vector of length n, presented as the input to the FFN.

The structure of the fully connected network consisted of a hidden layer with m neurones of hyperbolic tangent transfer function and a linear output layer with a single linear neurone, as depicted in Figure 6.1. The type of model considered in this work consisted of a MISO. A Gauss-Newton based Levenberg-Marquardt method was employed to minimise the mean square error criterion, due to its fast convergence property. The method adopted by Nørgaard in his function *marq* can be found in Fletcher (1987).

6.4. Development and Training of the UASB Reactor Models

Three models of the UASB reactor were constructed namely on the effluent TOD, average OD and also the biogas CO_2 with starch and dye loading as the inputs to these models. The three network architectures were based on the NNARX structure built using the Nørgaard (1995) ANN system identification toolbox, and they were of the MISO type. These models were developed, trained and tested using MATLAB®. The program written in MATLAB® to train the TOD NNARX model is shown in Section B.4 – Appendix B.

6.4.1. Data Selected for the NNARX Models Training

The data used to train the NNARX models was gathered from Experiments 3.1, 3.2, 3.3, and 3.4 over a period of 25 days of continuous operation (Section 3.6.3). These experiments were geared towards the generation of information regarding the behaviour of the UASB reactor when subjected to various starch and dye loading conditions. Continuous on-line monitoring of CO_2 was achieved from the experimental runs, apart from short periods, which were neglected (Chapter 3). However, no on-line measurements of TOD and average OD were available at all times due to sensor failure or loss of calibration, therefore a comparison with off-line measurements was carried-out. In the case of TOD, a comparison with off-line measurements of COD revealed a ratio of 1.4 during the 4 experiments and was subsequently used for conversion (*sd* = 0.23, n = 25). The average OD (which was available on-line) exhibited an identical trend to the true colour measurements (performed off-line). Although 0.83 absorbance units were taken from the average OD to be referred as true colour measurements expressed in TCU as the filter mesh was the 185 μm. In the following Figures, the average OD is abbreviated as OD. These experiments were felt to give a large enough spread of operating conditions to be used in the development of the models.

Chapter 4 described that for an increase in starch load there was no decrease in BA and pH values within the UASB reactor. Actually, the opposite happened due to the starch being hydrolysed with NaOH, which made those parameters rise within the reactor. This situation may not happen in real life as the starch may be hydrolysed with another chemical or substance such as amylase and therefore to model these relationships at this point would not be of any benefit. Therefore, it was decided to model the TOD, average OD and CO_2.

From the continuously gathered data, the sampling rate was decreased to 1 hour from an initial value of 2 minutes. The reason was to reduce the size of the training data and also to enable one-step ahead predictions further in the future (of 1 hour instead of 2 minutes). A 1 h sampling rate was found to be suitable and that at least two past samples were required by the regressor structure (as inputs to the models) to deliver its one-step ahead prediction. Premier *et al.* (1999) used a similar approach when modelling a fluidised bed anaerobic digester in which a 30 minutes sampling rate was adopted. The three response curves (TOD, average OD and CO_2) were passed through a 4th order Bessel low-pass filter, which was empirically found to provide suitable high frequency noise attenuation (National Instruments, 1998). Figures 6.2 and 6.3 show the responses of the reactor effluent TOD, average OD and the reactor biogas CO_2 (%) for the different loading concentrations of starch and dye (600 data points). The upper and lower limits of the input and output data set used to train the three MISO models are presented in Table 6.1. This range of limits adopted during training was the working boundary when validating the performance of the models.

Table 6.1 - Upper and lower limits of the training data

Parameters	**Maximum**	**Minimum**
Starch (g l^{-1})	3.8	1.9
Dye (g l^{-1})	0.75	0.15
UASB reactor effluent TOD (mg l^{-1})	2352	979
UASB reactor effluent average OD	3.11	0.33
UASB reactor biogas % CO_2	32.3	24.9

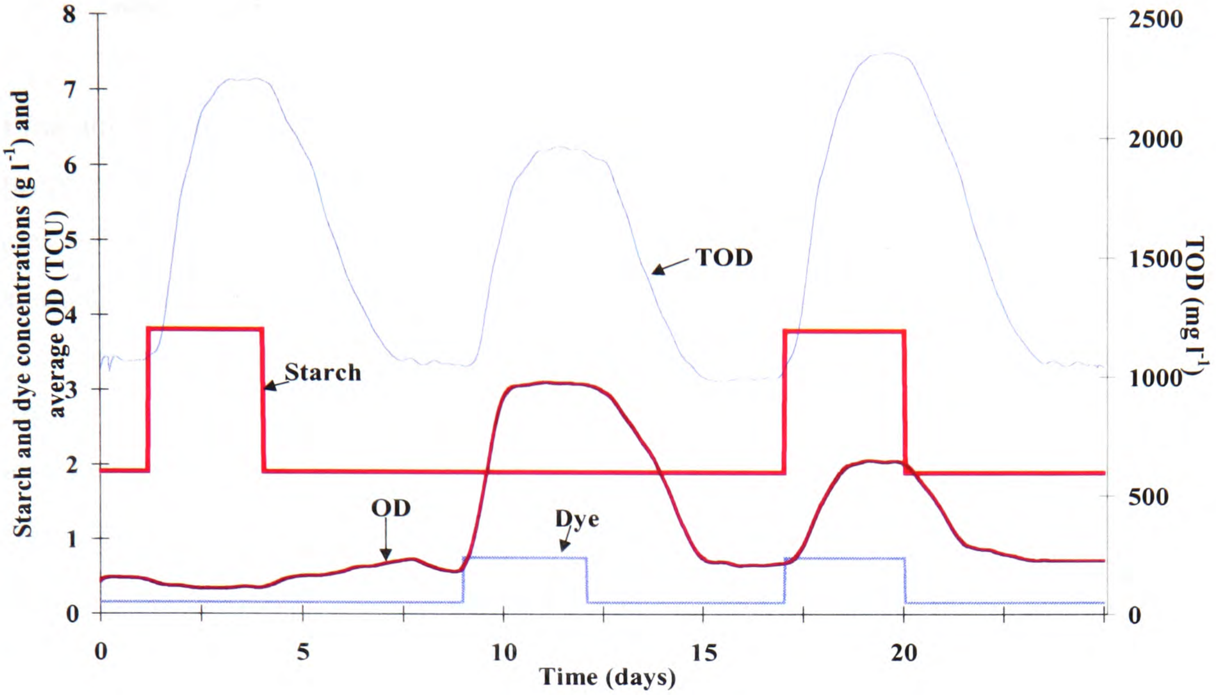


Figure 6.2 - Training data set - UASB reactor effluent TOD and average OD for the different organic and dye loads

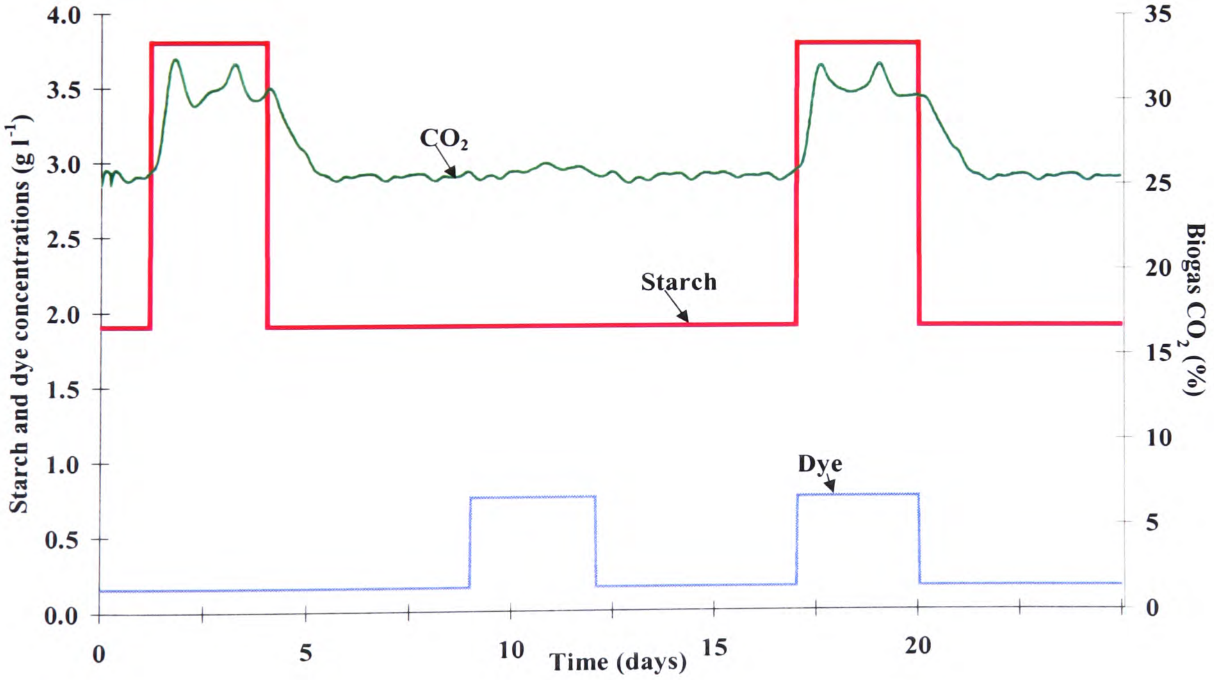


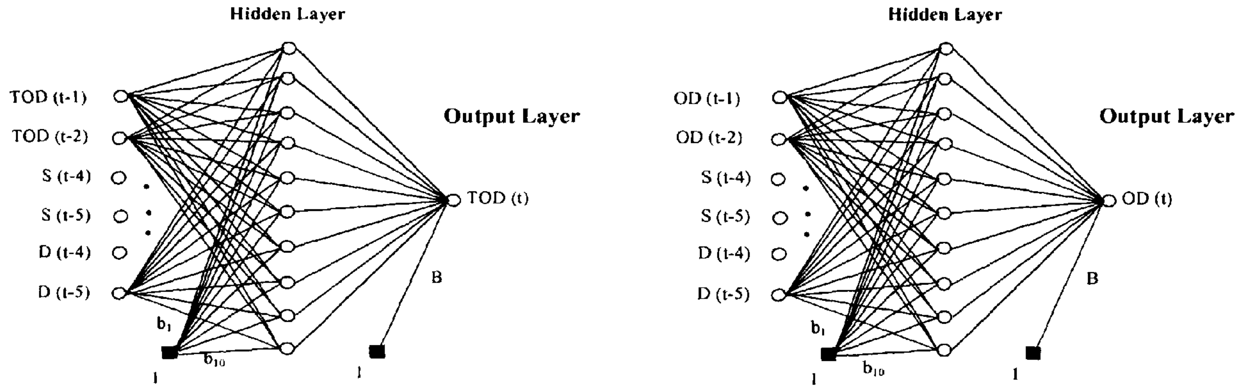
Figure 6.3 - Training data set - CO_2 in % in the UASB reactor biogas for the different organic and dye loads

6.4.2. NNARX Models' Structure and Training

An insight into the process to be modelled can prove to be extremely useful in selecting the appropriate parameters such as the delays and orders of the parameters when forming the regressor vector to be fed as inputs to the neural model. The NNARX models were, as previously noted, of the MISO type. Hence, each model represented one output parameter of the reactor. The delay time was observed (Figures 6.2 and 6.3) to be of one sampling period (1 h) for CO_2, (4 h) for the effluent TOD and average OD (Sections 4.2.1, 4.2.2 and 4.2.5). The response of the three output parameters was found heuristically to be two (i.e. order of the output) (Premier *et al.* 1999). Before training, both inputs and targets (600 data points) were normalised to a mean of 0 and variance of 1 in order to improve the network training and also to reduce training time using the function *dscale* (Nørgaard, 1995). The following parameters were carefully selected prior to training: the number of inputs (i.e. starch and dye), neurones in the hidden layer and the error goal (i.e. a measure of the desired predictive accuracy from the model).

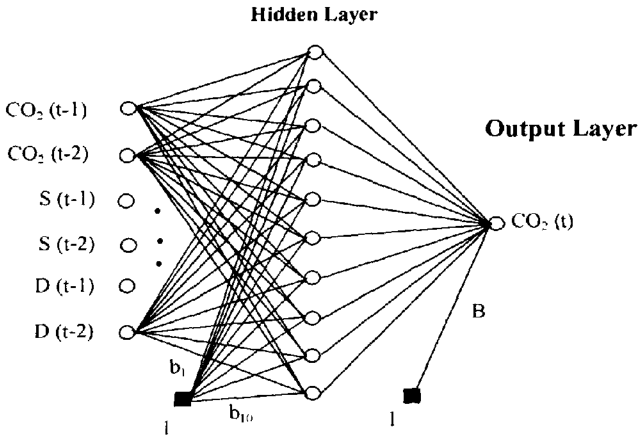
The input parameters of the three NNARX models were the starch and dye concentration in the STE (600 data sets). Each model had only one output (i.e. TOD, average OD or CO_2) and the structure is as depicted in Figure 6.4. The type of MLP network structure adopted for the identification tasks here was confined to one hidden layer of hyperbolic tangent activation function (*tanh*) neurones and a single neurone in the output layer with a linear activation function (Nørgaard, 1995). The following settings were used: n_a was 2 (order or number of past outputs), n_b was [2 2] (past inputs of both starch and dye) for the 3 models whilst the n_k (delay) was [4 4] for the TOD and average OD models and [1 1] for the CO_2 model. These parameters were set based on past working experience and also through trial and error. The same empirical derivation procedure was used to decide on the number of hidden neurones and the normalised sum squared error (NSSE) goal. Increasing the order could result in lower error goal during training, but to a large extent cause over-fitting of the data (Premier *et al.*, 1999). It is worth stating that the function *lipschit* in the Neural Network Based System Identification Toolbox can be used to calculate the order (lag space) for SISO models. However, Nørgaard (1995) emphasised that insight into the process being modelled is still the best means of synthesising a good model.

It was found that 10 neurones in the hidden layer were sufficient to represent the characteristic of the three models. As defined in the function *nnarx* by Nørgaard (1995), a NSSE goal of 0.0003 was set although a lower error was found for the average OD prediction model only after a few training iterations as depicted in Figures 6.5 to 6.7 (where S – starch and D – dye concentrations). Although, the predefined error goal was probably not the global minimum it was found to be sufficiently low to provide good results, and was used for all the three models. A maximum of 7 iterations were needed for the CO_2 model to achieve the specified NSSE while the other models required a smaller number of iterations. This demonstrates the rapid convergence property of the training algorithm adopted by Nørgaard in his function *marq* based on the Leverberg-Marquardt optimisation technique to reach the predefined NSSE goal.



TOD Model

Average OD Model



CO_2 Model

Figure 6.4 – Structure of the NNARX models for TOD, average OD and CO_2

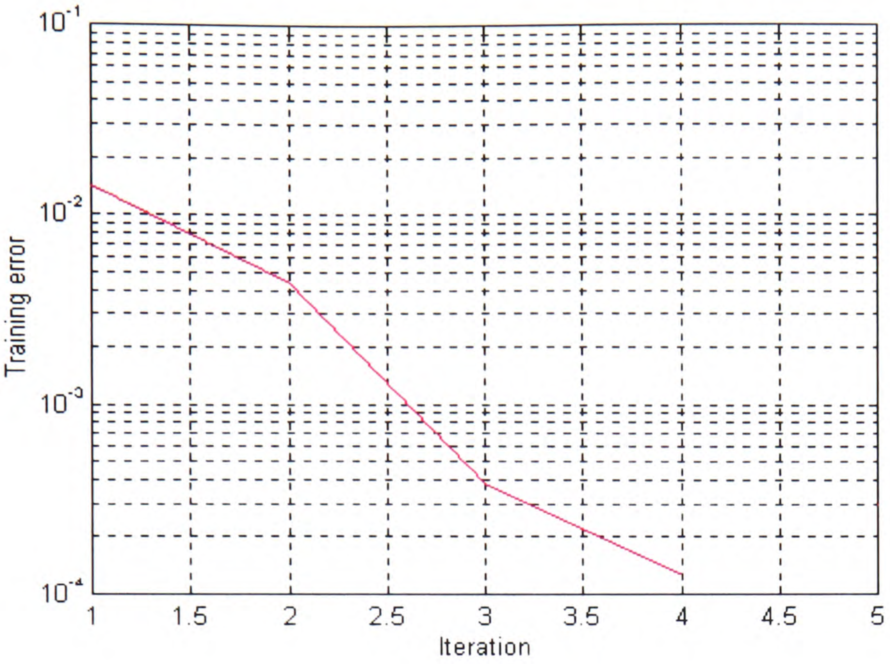


Figure 6.5 - Training error vs. number of iteration for the TOD model

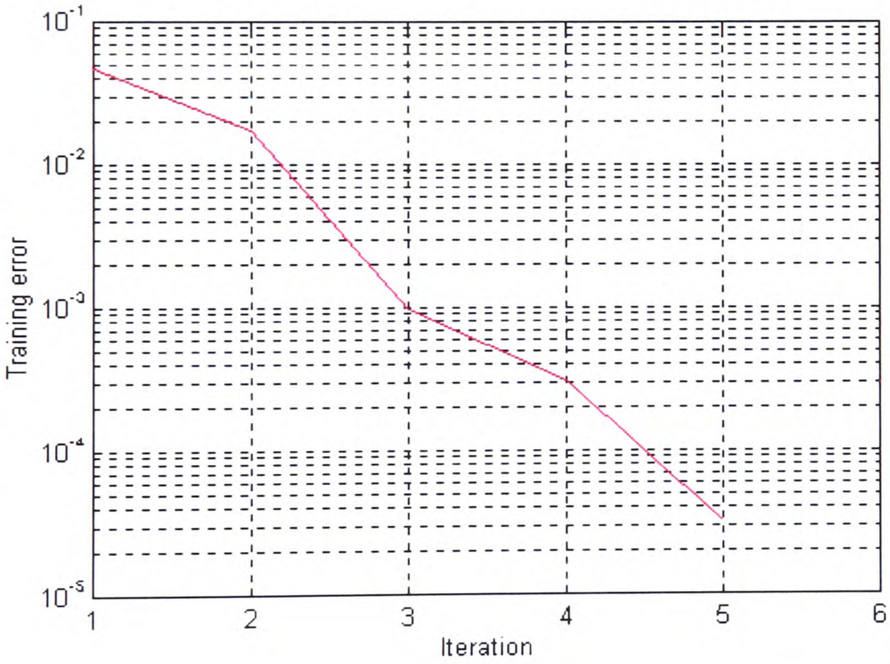


Figure 6.6 - Training error vs. number of iteration for the average OD model

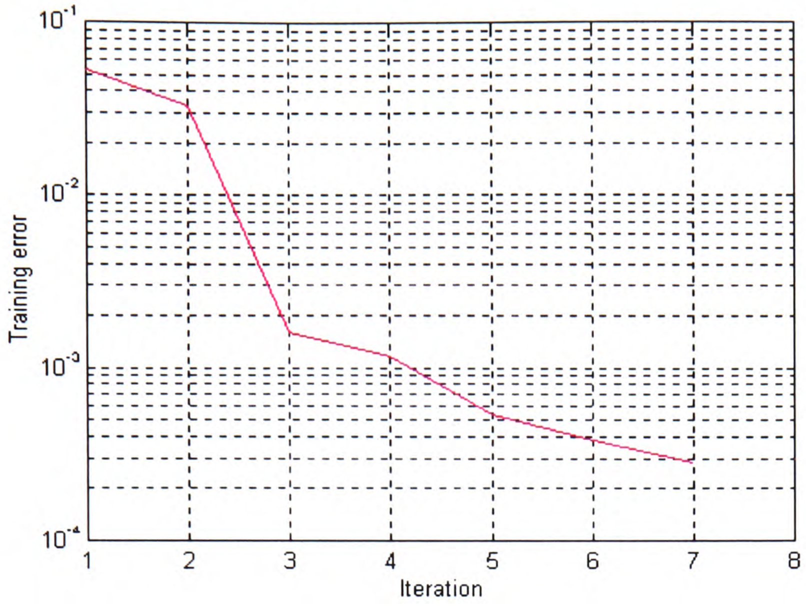


Figure 6.7 - Training error vs. number of iterations for the CO_2 model

The objective of this work was to use the three models to represent the physical UASB reactor, in order to test the proposed ANNBCS. As they were going to be employed in a computer simulation with an unlimited predictive horizon i.e. pure simulation, there was a need for their optimised performance. Nørgaard (1995) proposed a solution to reduce the dimensionality of the NNARX models by employing the so-called Optimal Brain Surgeon algorithm to 'prune' the network (*nnprune*) in order to optimise its performance. This involved re-training the networks for a number of iterations (in this case 50) in order to optimise the performance of the three models with respect to the training data sets after some weights of the network were removed (i.e. 5 %). This algorithm ran until a global minima was reached and the network weights that delivered the best performance were adopted (Nørgaard, 1995). The length of time taken to prune was 5 h for the TOD and average OD models and almost 6 h for the CO_2 model with a P120MHz PC with 48 MB of memory. The program written in MATLAB® to prune the NNARX models is shown in Section B.5 – Appendix B.

The testing and validation of the pruned models' one step ahead predictions will be shown in the next two sections (6.5 and 6.6) whilst the testing and validation for pure simulation predictions will be shown in Sections 6.7 and 6.8.

6.5. One Step Ahead Prediction Testing of the UASB Reactor NNARX Models

Figures 6.8 and 6.9 show the comparison between the models' one-step ahead predictions with the targets for TOD, average OD and CO_2 production, respectively. As one would expect they are in good agreement, since a good-fit model has been derived in response to the performance of an objective function through the learning process from the example data set. The ASSE found during the testing of the 3 models were as follows: 84 for the TOD, 3.63×10^{-5} for the average OD and 0.2 for the CO_2. These ASSE(s) will be the indices by which to compare the one-step ahead validation results.

Figure 6.8 shows that the predictions for the TOD and average OD models were extremely good as the two lines (targets and predictions) actually overlapped. The target and prediction lines for the CO_2 model did not overlap although they were very close as shown in Figure 6.9.

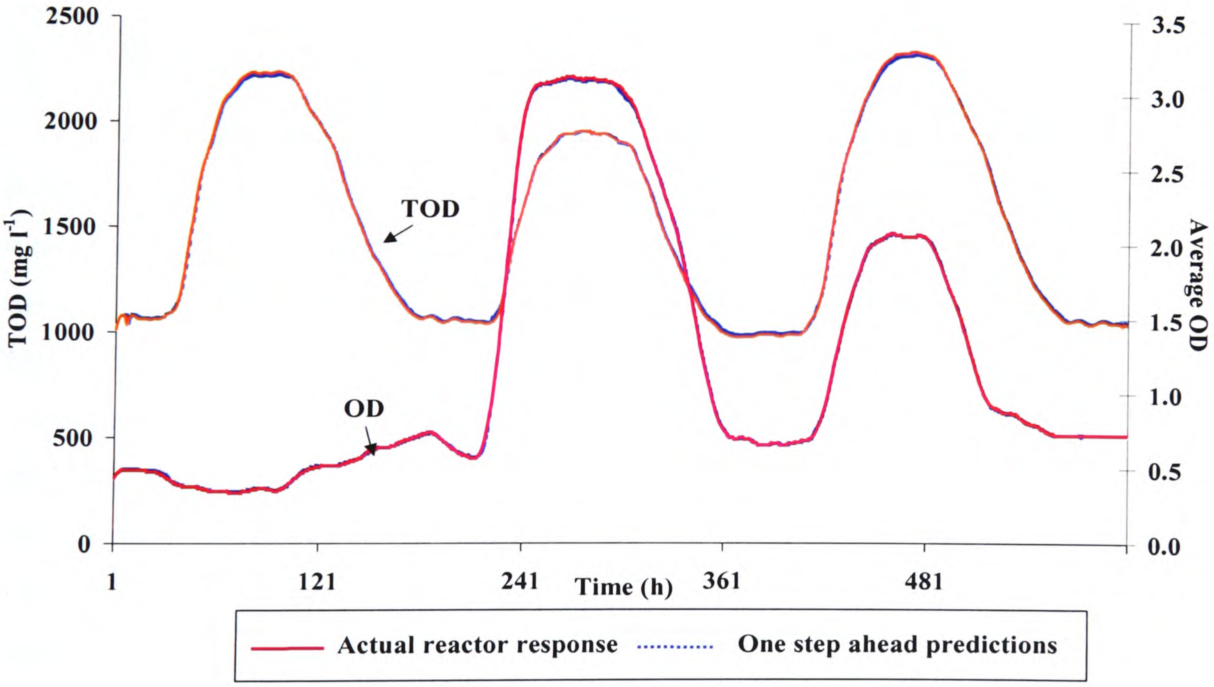


Figure 6.8 – Testing of the TOD and average OD models using one-step ahead predictions

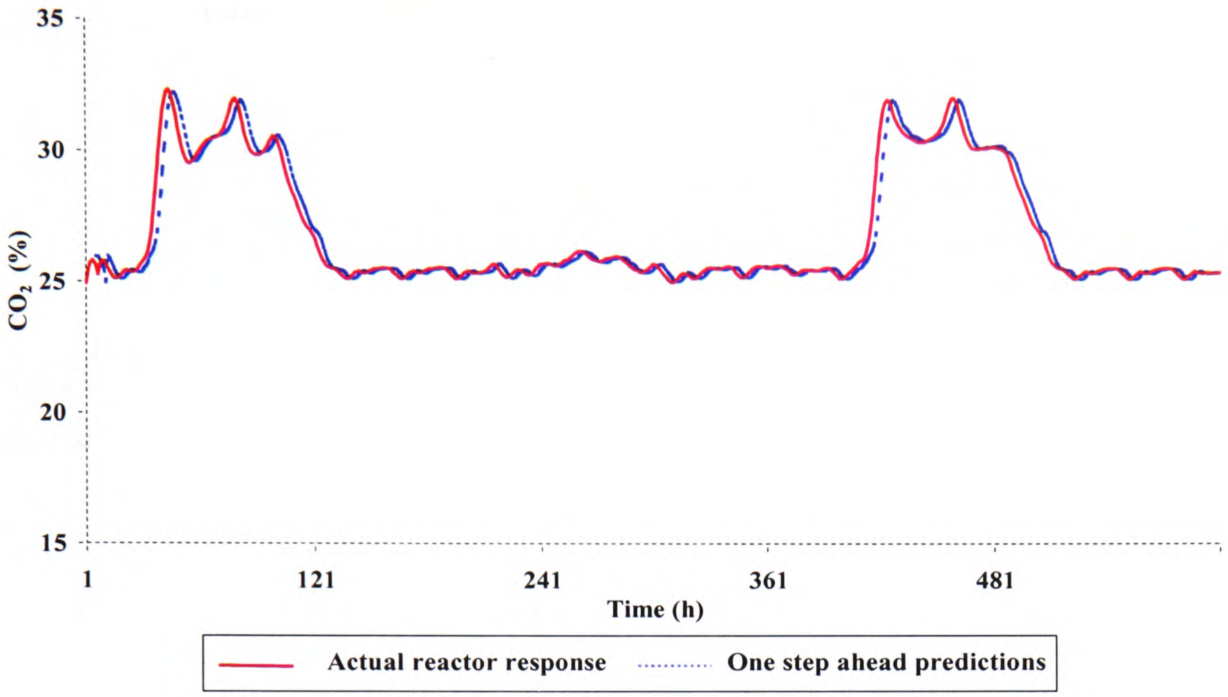


Figure 6.9 – Testing of the biogas CO_2 model using one step ahead predictions

6.6. One Step Ahead Prediction Validation of the UASB Reactor NNARX Models

The data used to validate the models was gathered from Experiment 3.7. This information was not presented to the models during training and was gathered over a period of 9 days of continuous operation with the same sampling rate (1 h). This data set (217 vectors) was collected from the UASB reactor operation 23 days after the training data sets from which the feed to UASB reactor was stopped and the reactor was just heated. This time period represents a significant temporal separation between the examples and the unseen data and would impose a stiffer test on the performance of the models. It is important to bear in mind that the model can only be tested using similar inputs (i.e. the inputs are starch and dye only no other input as for example an inhibiting factor can be considered). The upper and lower limits of the validation data set are presented in Table 6.2 and validation data sets were filtered using the same 4th order Bessel low-pass filter as before, prior to the one step ahead validation. The validation data sets are presented in Figures 6.10 and 6.11, for TOD, average OD and CO_2.

Table 6.2 - Upper and lower limits for the validation data set

Parameters	Maximum	Minimum
Starch (g l^{-1})	2.9	1.9
Dye (g l^{-1})	0.45	0.15
UASB reactor effluent TOD (mg l^{-1})	1859	895
UASB reactor effluent average OD	1.16	0.44
UASB reactor biogas % CO_2	29.83	23.8

When comparing the ASSE of the prediction and validation exercise for each model, the expected results were achieved. An ASSE of 8 times greater (i.e. ASSE = 686) was achieved when validating the TOD model, for the average OD and CO_2 models higher ASSEs were also achieved as 28 (i.e. ASSE = 0.001) and 1.1 (i.e. ASSE = 0.22) times, respectively. These show clearly that the validation results were very good when comparing them with the targets. However, the best way of assessing the model predictions is by looking at the predicting plots and analysing whether they are a good representation of the physical UASB reactor.

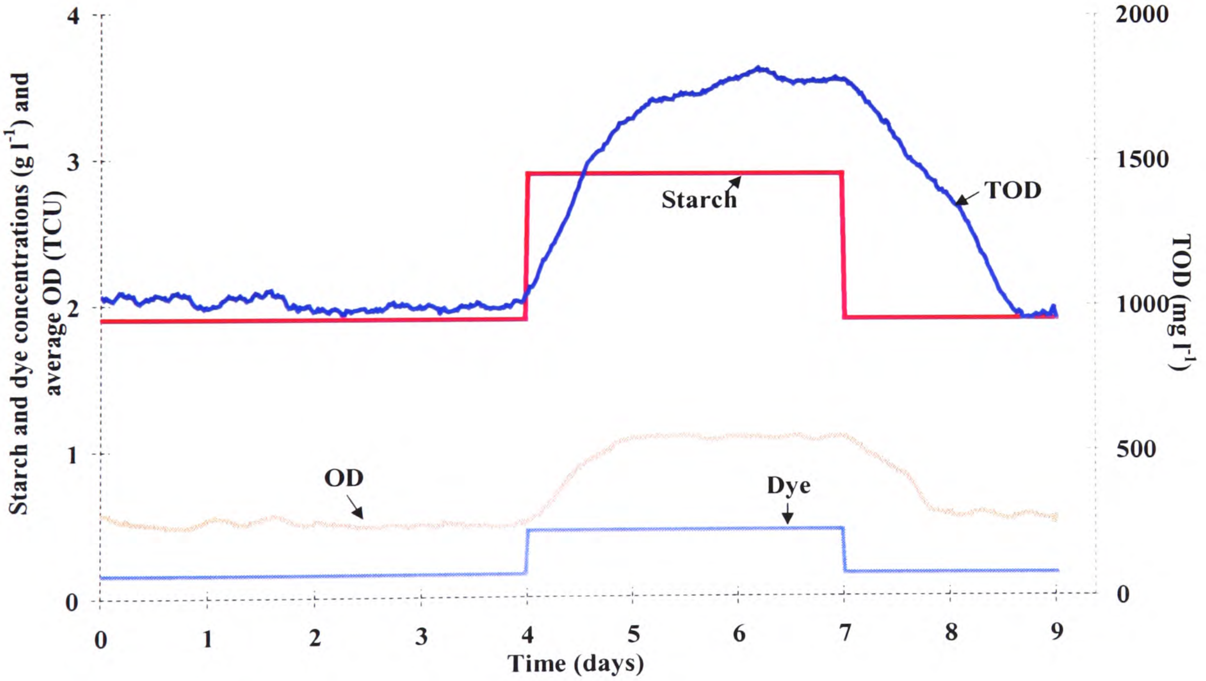


Figure 6.10 - Validation data set – responses of TOD and average OD resulting from step changes in colour and organic strength

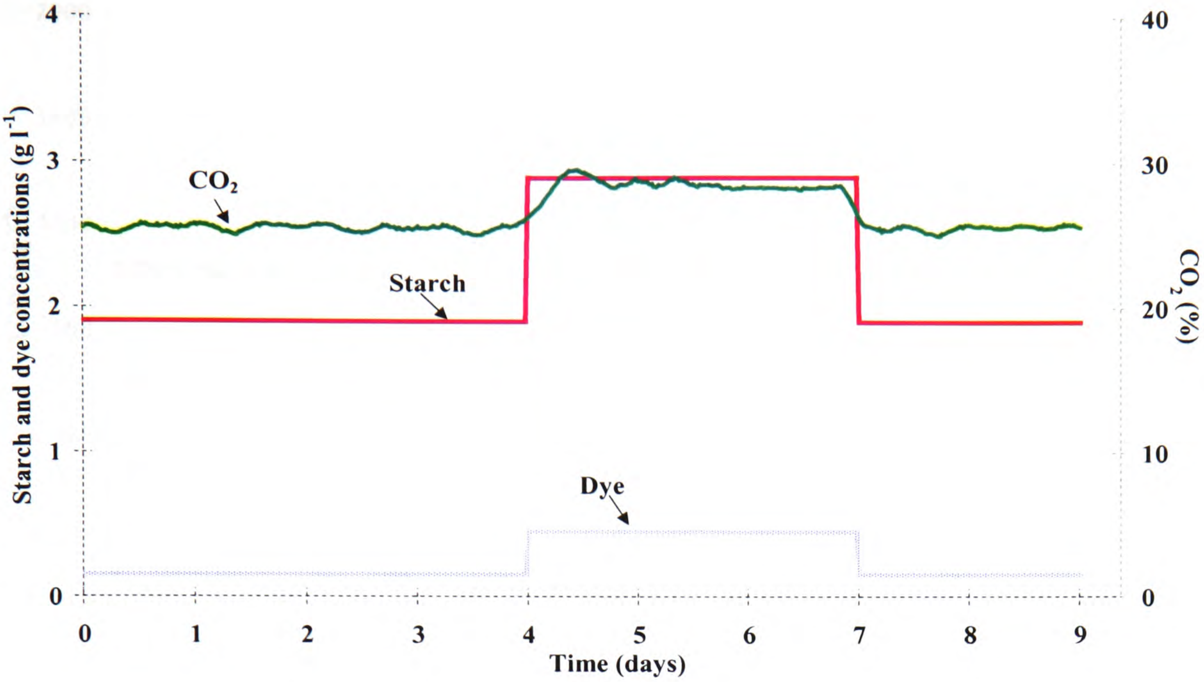


Figure 6.11 - Validation data set – Response for CO$_2$ (%) from step changes in colour and organic strength

At this point the designer can take a closer look at the delays and orders of the input parameters and intuitively fine tune these parameters and re-train the networks to investigate for possible improvements. But visual investigation of Figures 6.12 and 6.13 clearly demonstrates that the models are able to represent the dynamics during process operating conditions. It can therefore be concluded that the delays and orders of the inputs and output have been adequately chosen and an appropriate network architecture has been adopted as the structure for the non-linear NNARX model. The results suggested that 'black-box' modelling technique using the NNARX regressor structure was successful in modelling the non-linear and time variant behaviour of the UASB reactor, and is reflected in the satisfactory one step ahead (1 h) predictions. Sections 6.7 and 6.8 present the testing and validation of the models' predictions for pure simulation, respectively, and discuss the results.

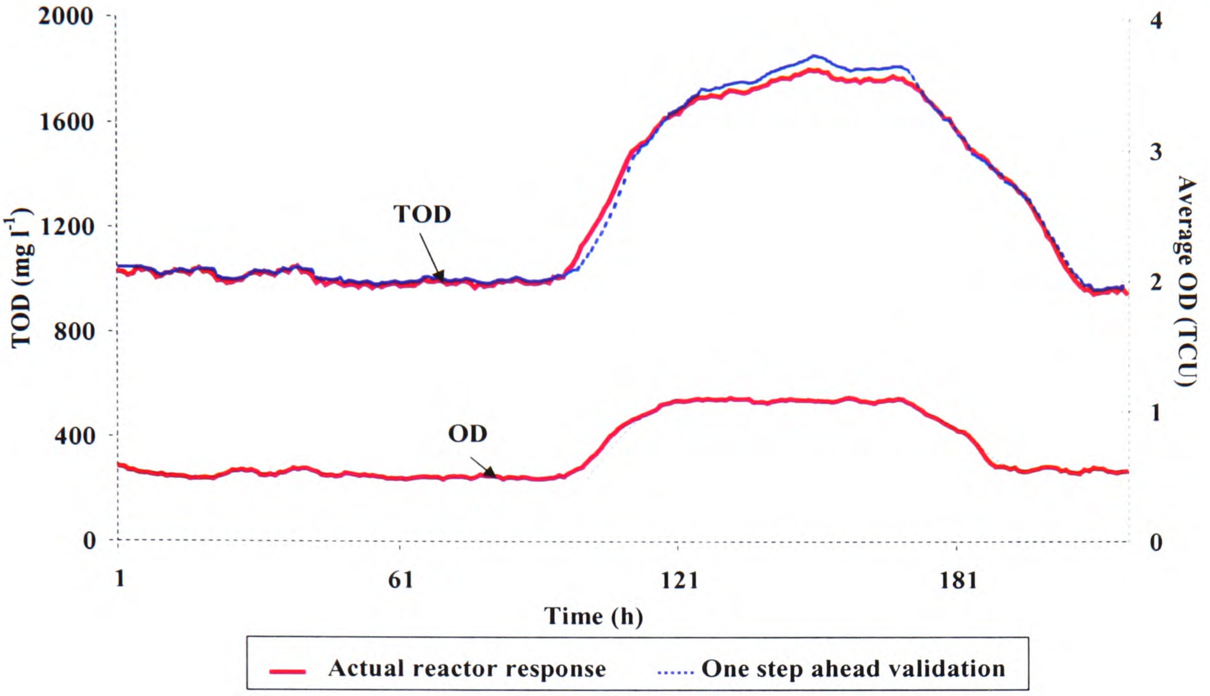


Figure 6.12 - Actual response vs. one step ahead validation for the UASB reactor effluent TOD and average OD

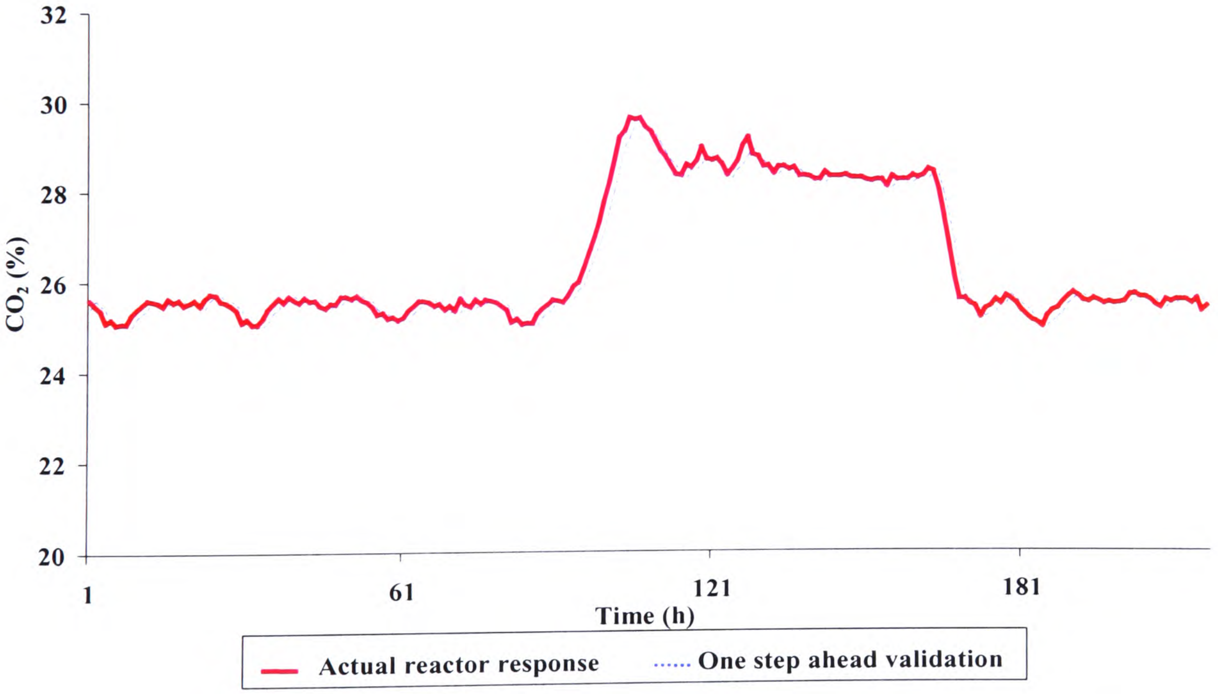


Figure 6.13 - Actual response vs. one-step ahead validation for the UASB reactor biogas CO$_2$

6.7. Testing of the NNARX Models Using Pure Simulation

In Sections 6.5 and 6.6, the performance of the three NNARX models to deliver one-step ahead predictions was tested with the training data, already seen by the networks and then validated with unseen data. It is important to emphasise that the one-step ahead predictions represent a future prediction, 1 hour in this case (i.e. sampling rate). It is very common to obtain good one step ahead predicting results with NNARX models (Premier *et al.*, 1999; Chong, 1999; Chong *et al.*, 2001). As for such prediction there are no accumulation of errors, meaning that for a second prediction the network takes the target values and not the previous model predictions.

A real test to the models was performed when the models' inputs would be its previous predictions until the end of the prediction process i.e. the actual targets were never shown to the models during the prediction. For these cases an increase of the ASSE would be expected, as would be an accumulation of the errors. This section deals with this pure simulation technique or futuristic prediction where the prediction is performed with the training data. Figures 6.14 and 6.15 show both the actual UASB reactor responses to data already seen by the models for pure simulation predictions.

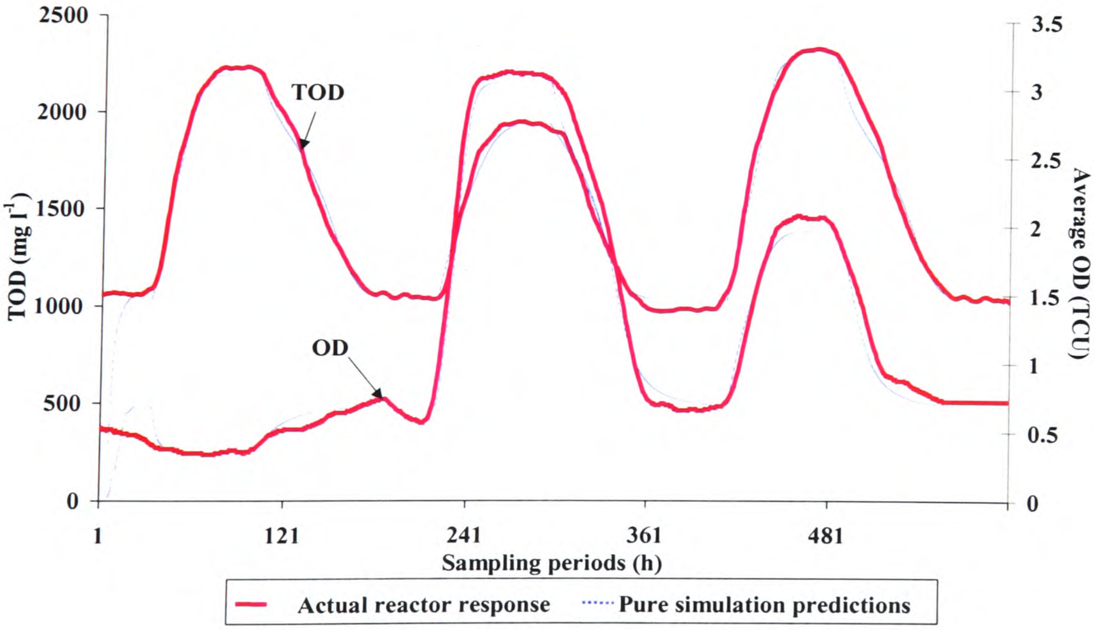


Figure 6.14 – Testing of the TOD and average OD models using pure simulation

As can be seen in Figure 6.14 the predictions for the first 24 sampling periods rapidly converged with the actual values, as the regression matrix had to be constructed first. From this point onwards the pure simulation predictions appeared to follow the trends very well. Similarly to the one step ahead predictions, the ASSEs were calculated here not accounting for the first 24 samples. As expected they have increased compared to those obtained in Section 6.6. They were as follows: 2253 for the TOD model, 0.011 for the average OD model and 0.42 for the biogas CO_2 model. These ASSEs will be used for comparison with the pure simulation predictions with validation data in the next section. This ASSE of 2253 actually means that an average error of ± 48 mg l^{-1} of TOD could be found when comparing the predictions with the actual targets. An average prediction error of 2-5 % defined a very good pure simulation prediction potential for the TOD model, as it is known that 5 % monitoring precision range is very common in organic strength monitors. For the average OD and CO_2 models the following average percentage error were found 3.8 – 15 % and 1.8 – 2.4 %, respectively. The model of average OD presented the least pure simulation prediction capacity for the testing data.

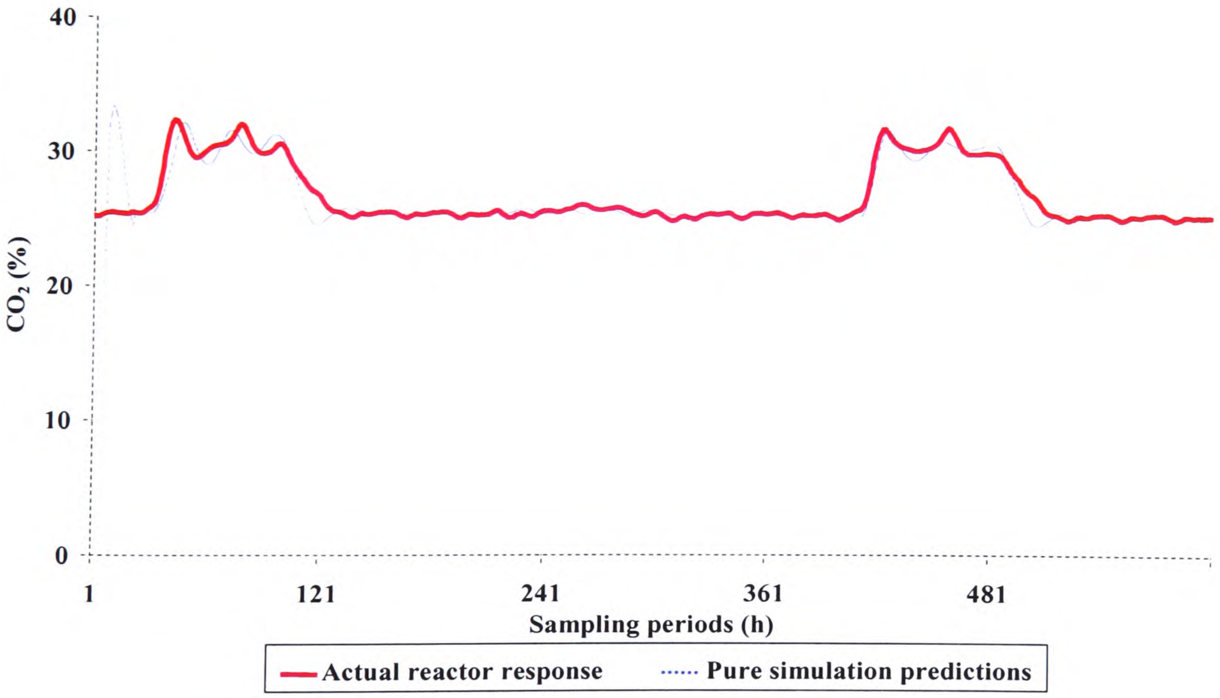


Figure 6.15 - Testing of the CO_2 model using pure simulation

6.8. Validation of the NNARX Models Using Pure Simulation

This section tests the full capability of the models when analysing its futuristic prediction with data unseen during training. It can be observed in Figures 6.16 and 6.17 that the predictions broadly follow the actual UASB reactor behaviour for TOD and average OD parameters, however the CO_2 model seemed to have some difficulties.

As undertaken previously in Sections 6.5 and 6.6, the pure simulation validation results were compared to the pure simulation testing results using the ASSE. These were 10696, 0.013 and 3.03 for the TOD, average OD and CO_2 models, respectively. In terms of % average prediction error for the TOD model was 6 – 10 % for the average OD model was 10 – 22 % and 6 – 7 % for the CO_2 model. As for testing, this validation exercise revealed that the least accurate model was the average OD model with up to 22 % average prediction error.

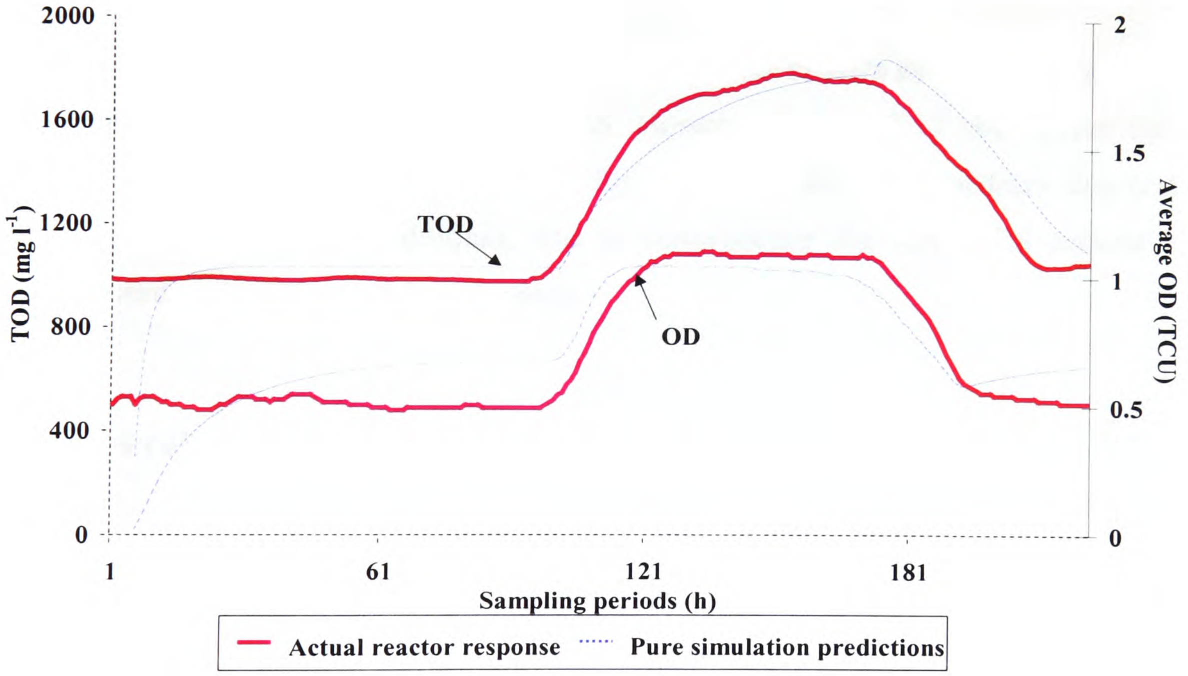


Figure 6.16 – Validation of the TOD and average OD models using pure simulation

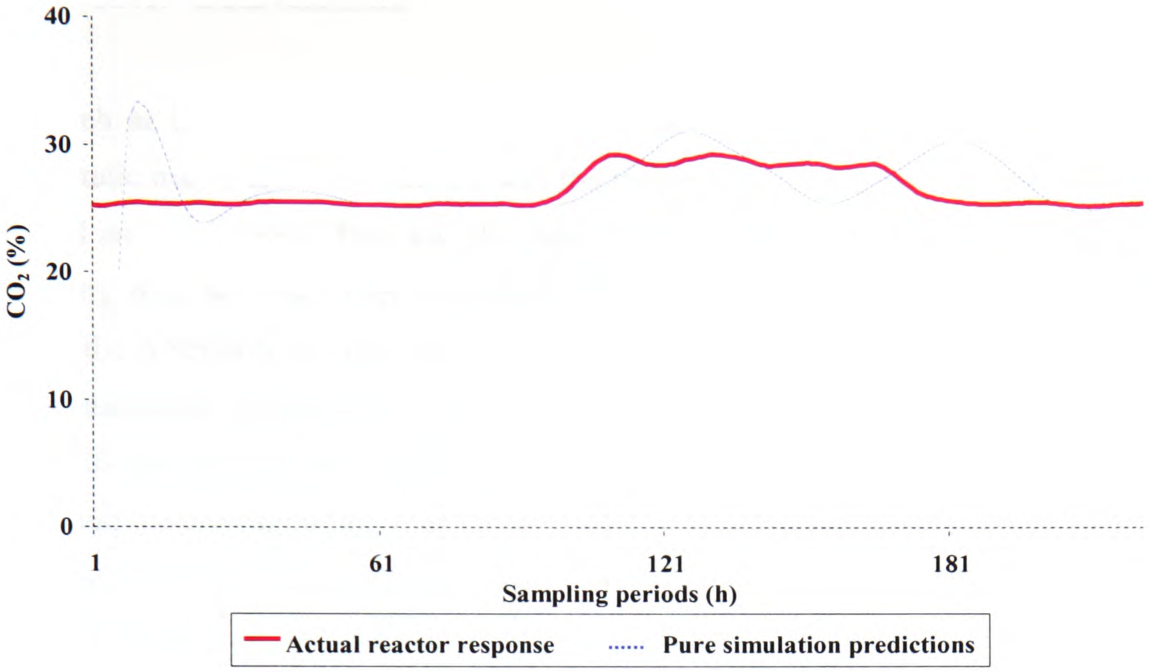


Figure 6.17 - Validation of the biogas CO$_2$ model using pure simulation

Considering the results, it was thought possible to use these models as a representation of the physical reactor in order to test the ANNBCS. However, Figure 6.17 shows that the CO$_2$ model predictions did not follow the trend for medium starch and medium dye (i.e. the considered good operating conditions), and in consequence the ANNBCS actions could fluctuate according to CO$_2$ model predictions.

6.9. Development and Training of the ANNBCS

The ANNBCS is based on a hybrid ANN structure (Chapter 5 – Control Scheme 4 (i.e. LVQ + BPs)), which was re-trained to be able to map the models' outputs to an adjustment factor of the starch and dye loading. In reality the textile industrial WW contains both starch and dye that cannot be separated although, an addition of a carbon source would compensate for any low organic textile effluent in the case where only dye concentration would have to be reduced. The program written in MATLAB® to train the ANNBCS is shown in Section B.6 – Appendix B.

6.9.1. Data Selection

ANNs such as LVQ and BP networks have no inherent temporal characteristics (i.e. they work as static maps) and therefore the way the training data is presented does not have to be sequential and continuous. This way the data gathered from Experiment 3.7 was included in the training data set since they represent the good operating conditions worthy of being taught to the ANNBCS to improve its control actions. In order to maintain the training data sets to a reasonable amount, data from Experiment 3.3 (i.e. high starch and low dye) was not included in the training set. Figures 6.18 and 6.19 show the training data for the LVQ network and the corresponding four BP networks. All the unshaded parts in Figures 6.18 and 6.19 included the training data for Class 1. These Figures show how the data was sub-divided for training of the LVQ network (with 4 classes) and the four BP networks. In this case, the classification network was used to sub-divide the data into smaller and similar data sets to improve the predictions of the BP networks and not for recognition of sensor failure as in Chapter 5. Testing of the ANNBCS for sensor failure conditions was not performed during this computer simulation as it was already tested on-line in Chapter 5 and it would be difficult to incorporate these failure conditions in the NNARX models as they have inherent temporal characteristics.

The ANNBCS outputs (i.e. adjustment factors for the starch and dye concentrations) were defined in order to maintain acceptable UASB reactor effluent quality and to maintain its health. This definition of constraints was based on previously gathered knowledge and by bearing in mind the following optimum conditions TOD ~ 1850 mg l^{-1}, average OD ~ 1.10 mg l^{-1}, and biogas CO_2 ~ 28.5 %. These being the average behaviour of the reactor for medium starch and medium dye loading conditions. During these operating conditions the UASB reactor was stable and degrading organic matter and dye to a good extent. At the same time biomass in the aerobic stage could be maintained so that the degradation of the dye breakdown products should take place. The output data for training of the ANNBCS was defined by the expert user as in Table 6.3.

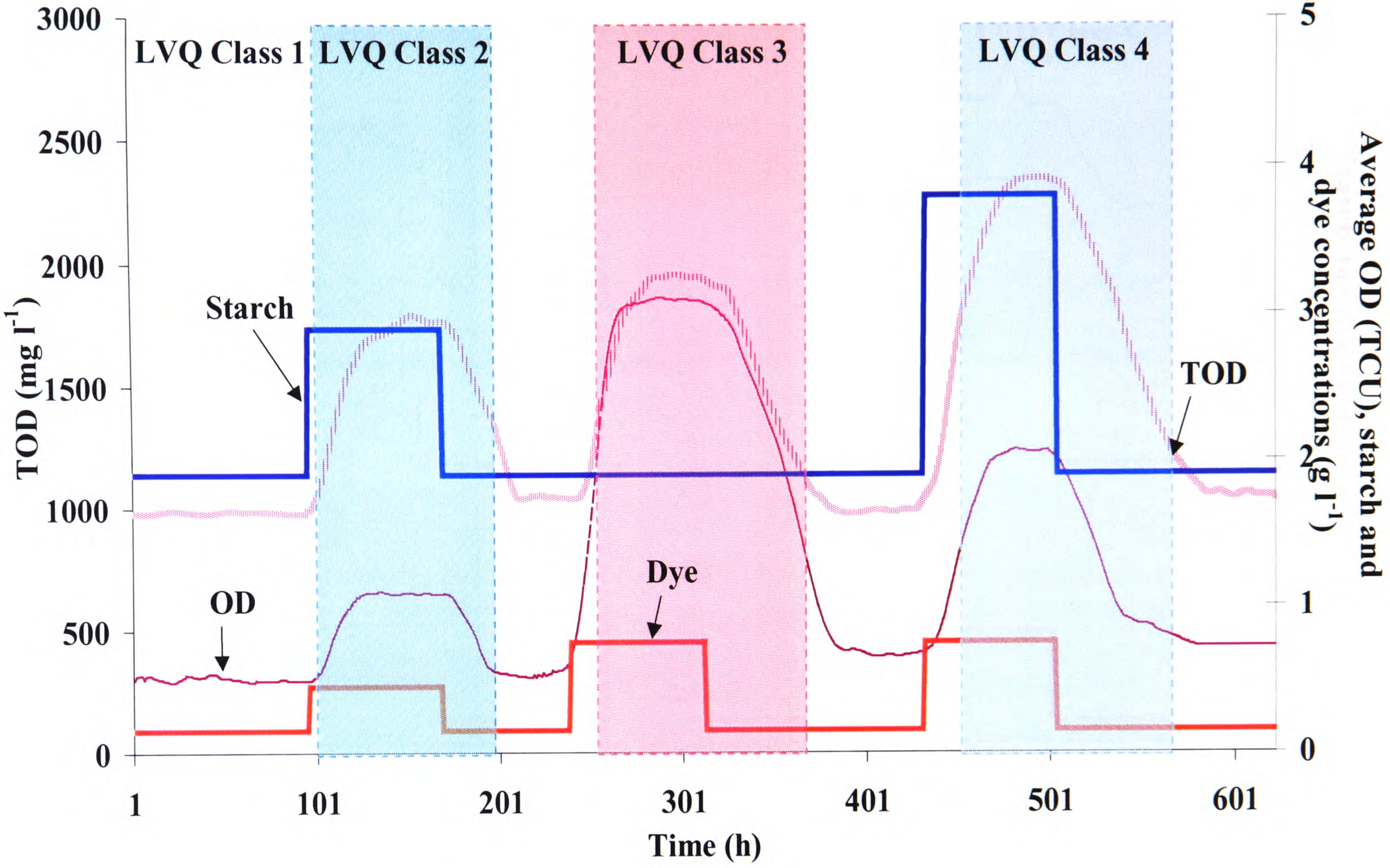


Figure 6.18 – Training data for the LVQ network and the four BP networks (starch, dye, TOD and average OD)

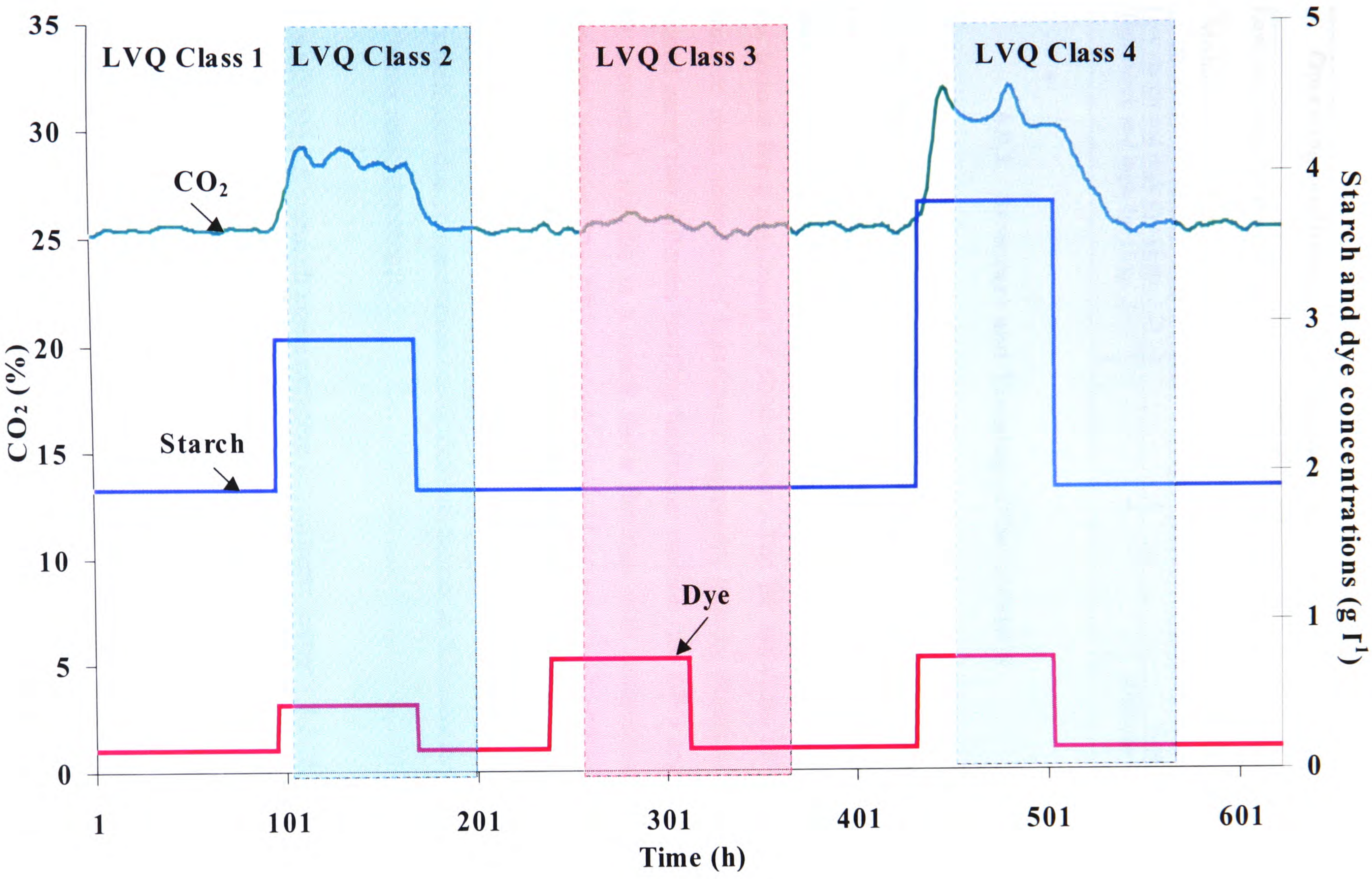


Figure 6.19 - Training data for the LVQ network and the four BP networks (starch, dye and CO_2)

Table 6.3 – Operating conditions and respective ANNBCS (LVQ + BPs) actions

Operating conditions	**LVQ network Class and respective BP network**	**ANNBCS actions**
Low starch and low dye (Exp. 3.1)	1	Increase in both starch and dye loads
Medium starch and medium dye (good operation) (Exp. 3.7)	2	Maintain both starch and dye loads
Low starch and high dye (Exp. 3.2)	3	Addition of starch
High starch and high dye (Exp. 3.4)	4	Reduction of both starch and dye loads

6.9.2. Structure and Training of the ANNBCS

The structure and training of the 5 ANN components of the ANNBCS are presented in Table 6.4. The LVQ network was trained to classify four classes of data as already mentioned. Each class had three subclasses (i.e. 12 competitive neurones) for better data distribution and it was trained for a maximum of 5000 epochs. The BP networks (1 to 4) were of a 3-layer structure with neurones of logarithmic sigmoidal transfer function in the three layers and trained using two different learning functions: *trainlm* (for BP1) and *trainbpx* (for the other BP networks). *trainlm* is a much faster function with momentum but required a lot of computer memory. The BP1 network achieved an error goal of 2 x 10^{-5} in only 7 iterations while BP2, BP3 and BP4 achieved an error goal of 0.04 with 2906, 9092, and 2704 iterations, respectively. The ANNBCS was trained to recognise the minimum and maximum of starch and dye concentration multiplication factors as follows (% adjustment is also given for better understanding):

- (-20 %) $0.8 <$ **starch concentration adjustment factor** < 1.5 (50 %);
- (-45 %) $0.55 <$ **dye concentration adjustment factor** < 3 (200 %);

It can be seen that dye concentration adjustments were more significant than the starch concentration adjustment in order to maintain the UASB reactor optimum operating conditions. The combination of the optimum values of TOD = 1850 mg l^{-1}, average OD = 1.10 mg l^{-1}, and biogas CO_2 = 28.5 % corresponded to the adjustment factor of 1 for both starch and dye loads i.e. maintaining the previous loads.

Table 6.4 – Structure and training parameters of the ANNBCS

ANN	Data sets	Network inputs (MISO model outputs)	Hidden neurones	Network outputs
LVQ	618	3 (TOD, average OD, CO_2)	N/A	[0 0 0 1] **Class 1**; [0 0 1 1] **Class 2**; [0 1 1 1] **Class 3**; [1 1 1 1] **Class 4**
BP1	278	as above	30	2 adjustment factors to be multiplied by the previous starch and dye conc.
BP2	102	as above	20	As above
BP3	122	as above	20	As above
BP4	116	as above	20	As above

6.10. Further Evaluation on the ANNBCS Performance in a Computer Simulation

The computer simulation was performed in MATLAB® programming environment, with the help of two ANN tools the ANN toolbox (Demuth and Beale, 1994), and the ANN system identification toolbox (Nørgaard, 1995). The first toolbox for the development and training of the ANNBCS and the second toolbox for the development and training of the NNARX models.

6.10.1. Concept and Architecture of the Computer Simulation

The general schematic of the organisation of the simulation program is depicted in Figure 6.20 and the utilised computer program can be found in Section B.7 - Appendix B. Figure 6.21 details the inputs and outputs of the three models. The basic idea behind the computer simulation was to utilise the predictions of the models by the ANNBCS. Should the treatment process be sub-optimal, the ANNBCS would suggest changes in the input parameters such as a certain percentage of adjustment in the starch and/or dye concentration being fed to the reactor. For example, if the biogas CO_2 and the reactor effluent TOD suggested that there was an organic overload then the ANNBCS would suggest a decrement in the starch load and so on as detailed in Table 6.3.

Part of the major work involved was to create the new 'regression matrix' (Nørgaard, 1995) by taking into account the new inputs as adjusted by the ANNBCS, and output (i.e.

predictions of the ANN models) matrices. This involved constructing the necessary regression vector at previous time steps (depending on the delays and orders of the inputs and output parameters) to deliver a prediction of the three parameters namely TOD, average OD and CO_2 at one-step ahead. For the computer simulation, appropriate initial conditions of TOD, average OD and CO_2 were provided in order to obtain a better representation of the actual process during the first few control iterations. The setting of the correct initial conditions was of paramount importance in the correct functioning of the ANNBCS.

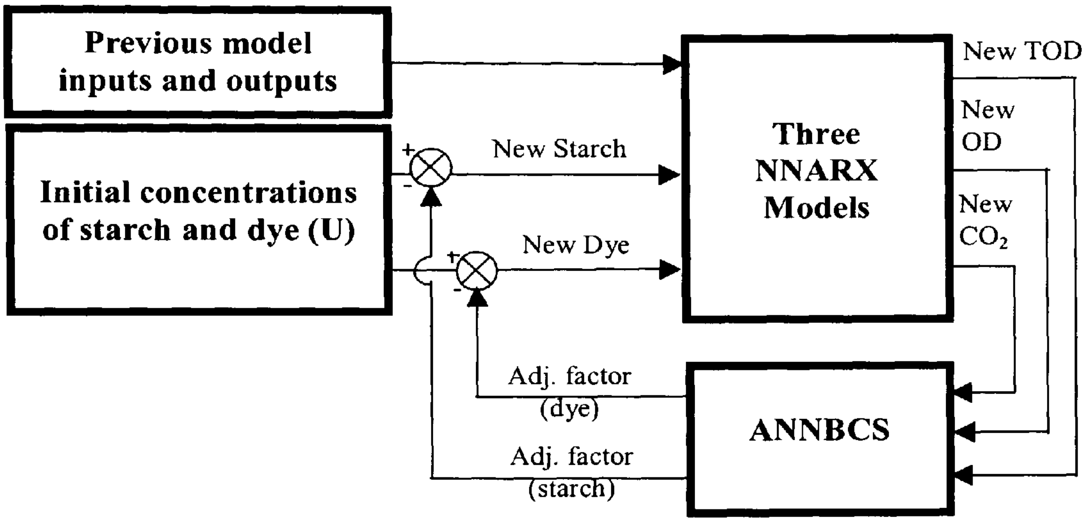
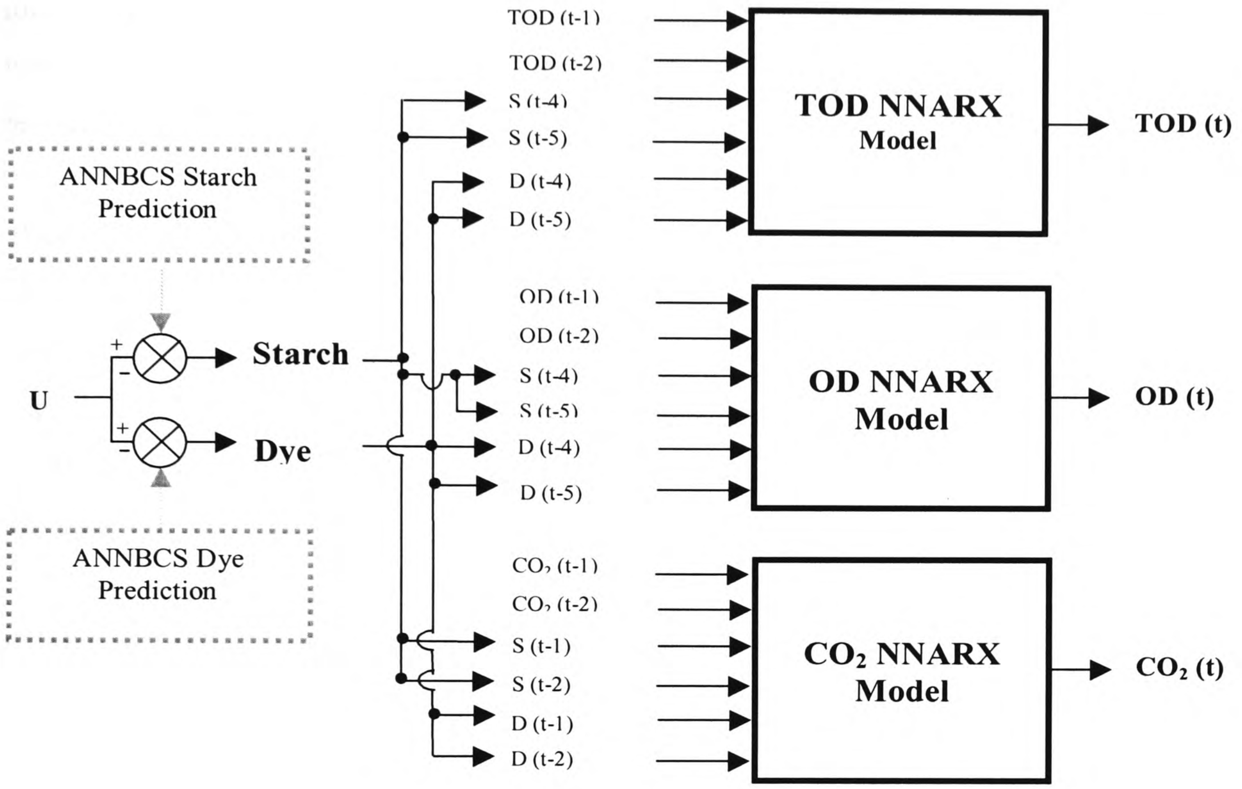


Figure 6.20 – Computer simulation architecture

An important factor that one must take into account is the aspect of disturbances to the system. As with computer simulation involving ANNs one would hope that the training data set would have incorporated if not all, some of the disturbances that are most likely to be encountered in the day-to-day operation of the reactor (e.g. biomass loss or growth). Therefore, the NNARX models would respond accordingly should these conditions be encountered again as there are no means of quantifying the relationship between for example the changes in the monitoring parameters with the bacterial population change even when starch and dye are constant over an extended period of time. Obviously, it would be more interesting and realistic to test the ANNBCS on the physical UASB reactor as it could after a period of time have improved or decreased in its treatment efficiency. Therefore, the starch and dye concentration predicted by the ANNBCS would vary more in order to maintain optimal values of TOD, average OD and biogas CO_2. The effect of integrating the CO_2

model within the computer simulation to further test the ANNBCS performance was analysed. The relatively faster CO_2 response to changes in the starch load, as it is a gas phase monitoring parameter compared for example with the TOD as a liquid phase monitoring parameter, became very valuable in terms of a control action.



Models:
TOD & OD → $n_a = 2$, $n_b = [2\ 2]$, $n_k = [4\ 4]$; CO_2 → $n_a = 2$, $n_b = [2\ 2]$, $n_k = [1\ 1]$

Figure 6.21 – Inputs and outputs from the three NNARX models within the computer simulation

6.10.2. Results and Discussion

This section describes the results obtained by carrying out the computer simulation exercise on the NNARX models and ANNBCS. The approach to further evaluate the ANNBCS was by carefully selecting examples of non-optimal operation of the reactor over a period of time (in the order of a few days) and feeding such information to the computer simulation with a view of comparing the ANNBCS effort with the reactor response. These examples are shown in Figures 6.22 to 6.30. Results of *Test A* are presented in the Figures 6.22 to 6.24, for the *Test B* in Figures 6.25 to 6.27 and finally, for the *Test C* in Figures 6.28 to 6.30. These Figures show the ANNBCS response (new starch and dye), being the starch and dye concentration, new predictions of the input parameters for the three NNARX models, which identified sensorial information, namely new TOD, average OD and CO_2. The three tests were designed to assess the predictions of the NNARX models in pure simulation and also to test the response of the ANNBCS.

Test A – Simulation of the ANNBCS response to low and medium starch and dye loads

The data sets of *Test A* were used to evaluate the proposed ANNBCS in a computer simulation environment, and consisted of information gathered from the UASB reactor when successfully treating a higher load intake. As can be seen in Figures 6.22 to 6.24, the step load changes were in the order of 50 % in starch and about 200 % in the dye loads. These step increases were carried manually in order to study the response of the reactor to such loading pattern upon the attainment of favourable conditions. The corresponding output parameters increased accordingly to such change with new values of TOD, average OD and CO_2 reaching a maximum of 1860 mg l^{-1}, 1.16 TCU and 29.83 %, respectively.

It can also be seen from these Figures, that the ANNBCS was able to make appropriate decisions upon detection of highly favourable conditions. These were detectable from modest values of the models' output parameters, which resulted in an increase in the load intake of a similar magnitude to those of manual operation. The models within the computer simulation were found to be able to 'replicate' the physical reactor in such working condition. Such satisfactory responses can be seen firstly in the response profile where the 'black-box' models were able to reproduce the actual trend to sufficient accuracy. The 'new TOD' from the TOD model prediction rose to a maximum of 1800 mg l^{-1} as compared to 1860 mg l^{-1} (actual data) following a very similar loading pattern in the starch intake. It is important to note that changes in the dye intake will inevitably affect the response of the TOD as dye itself contributes towards the organic level. Despite the changing level of dye intake after the step increase the average level was 0.38 g l^{-1}, as compared to the constant and maximum value of 0.45 g l^{-1} in the actual data. Following the first step increase in the dye intake during a time span of 10 h, the first noticeable change in the average OD model prediction was an increase from an average of 0.52 TCU to a maximum of 1.05 TCU. Such response was similar to that of the actual process where upon the step increase in the dye intake resulted in an increment of 25 % in the average OD level over a very similar time period. Hence, one can conclude that the ANNBCS performed well as it maintained the level of the average OD to below 1.10 TCU.

It is well known from the literature and from this study that the CO_2 level is more responsive to changes in the starch than dye intake. Therefore, the CO_2 level from its initial value rose by an additional 10 %, which was highly comparable to the actual reactor response. It must be mentioned here that the pure simulation predictive accuracy of the CO_2 model is the least satisfactory among the three 'black-box' models as described in Section 6.8. However, despite the additional 50 % increase in the starch intake the CO_2 fluctuated within a fairly tight band of between 25 and 32 %, such behaviour demonstrated by the CO_2 model can be very well observed in the actual reactor operation. Therefore, it can be concluded that even the CO_2 response behaved accordingly in the functioning of the ANNBCS (mimicking the behaviour of the actual reactor).

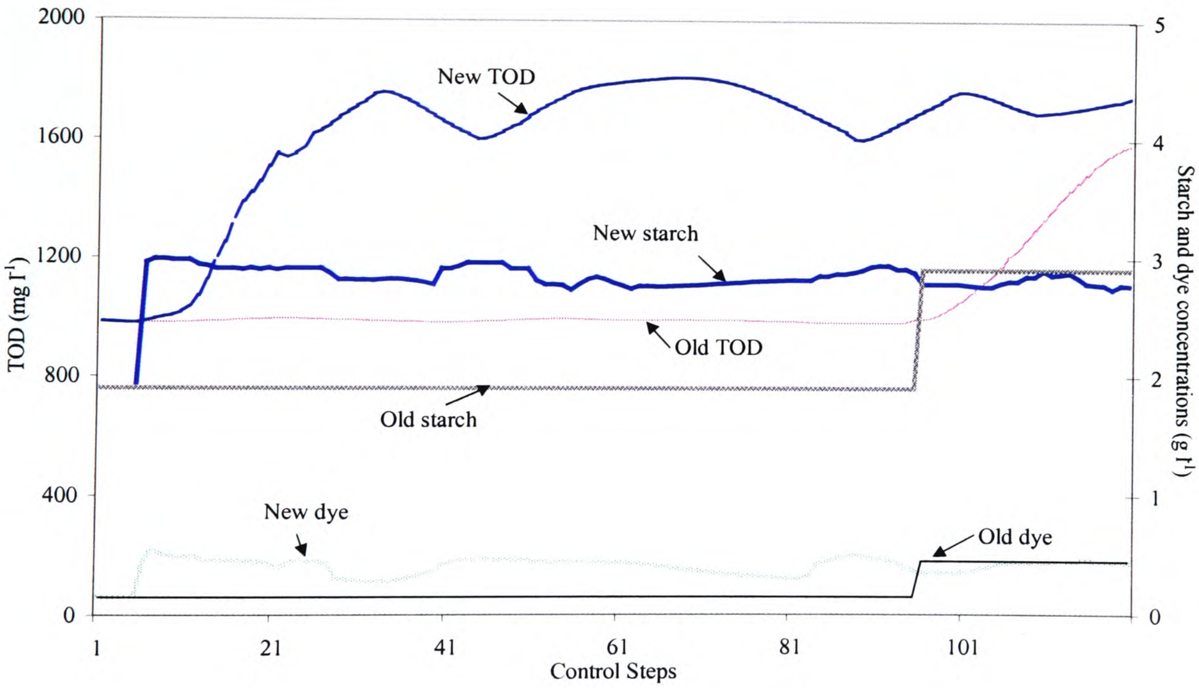


Figure 6.22 – Response of the ANNBCS to sensorial information for *Test A* – Changes to the input parameters (starch and dye) vs. TOD

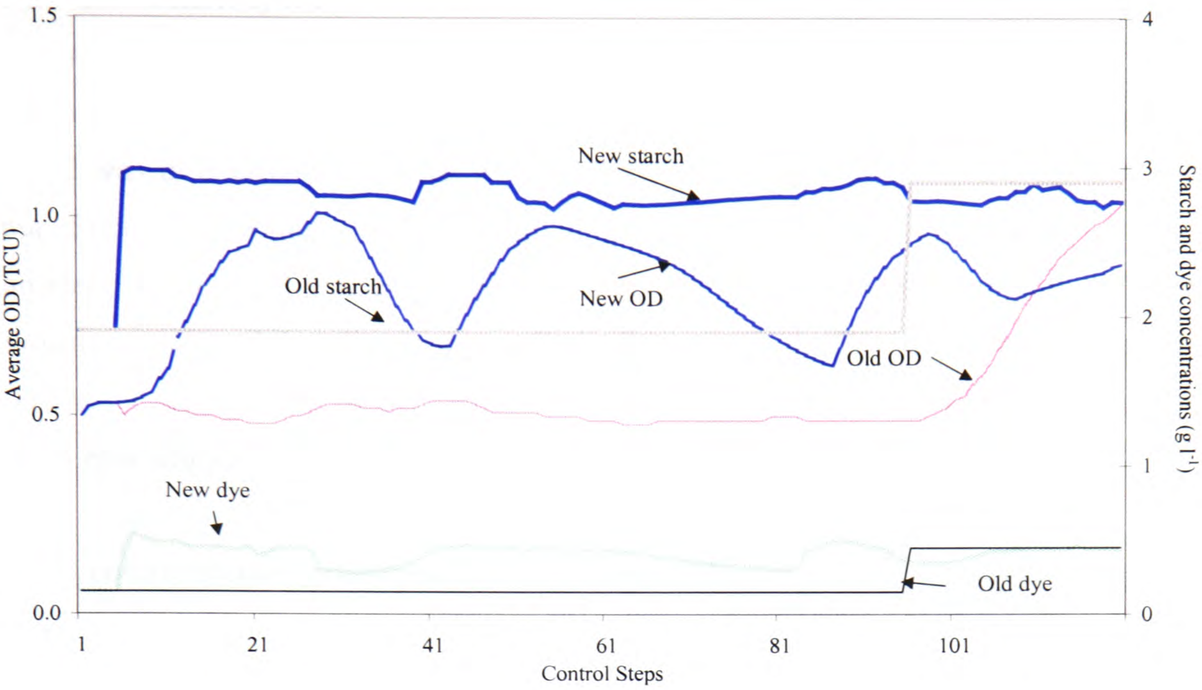


Figure 6.23 - Response of the ANNBCS to sensorial information for *Test A* – Changes to the input parameters (starch and dye) vs. average OD

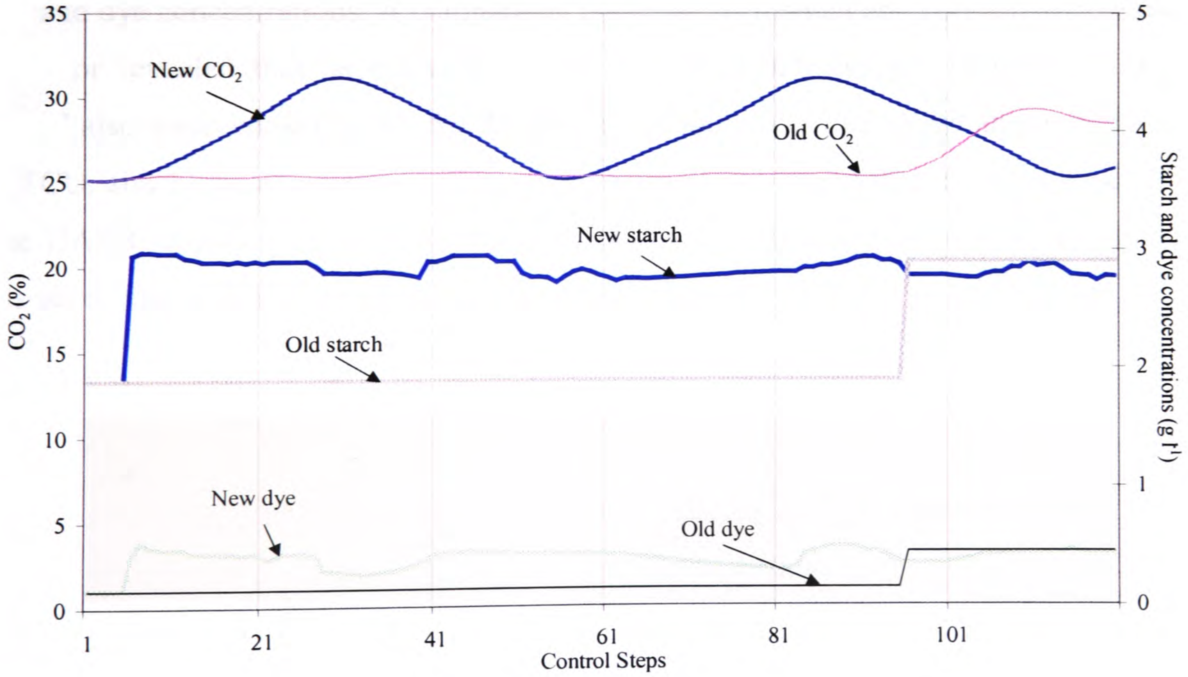


Figure 6.24 - Response of the ANNBCS to sensorial information for *Test A* – Changes to input parameters (starch and dye) vs. CO_2

Test B - Simulation of the ANNBCS response to high dye and low starch loads

Test B was mainly designed to assess the ANNBCS behaviour during high dye and low starch loading conditions (Figures 6.25 to 6.27). These operating conditions showed that the dye load was in excess and needed to be reduced to maintain a reasonable reactor effluent colour. Such an unwanted situation was controlled based on the adjustment of two main parameters: the overall dye concentration and also the low starch/dye ratio. It has been previously mentioned that the presence of a carbon source (in this case starch) is essential in the anaerobic degradation of dye. Its ratio has to be carefully selected for the best possible working efficiency.

After 18 control steps (corresponding to 18 h) from the start of the experiment only the dye load intake was manually increased by 400 %, the starch load concentration remained the same. The corresponding output parameters increased accordingly to such a change with new values of TOD, average OD and CO_2 reaching about 1950 mg l^{-1}, 3.1 TCU and 26.2 %, respectively. In this case the increase in the biogas CO_2 was almost negligible. It can also be seen from Figures 6.25 to 6.27, that the ANNBCS was also able to detect as previously shown in Figures 6.22 to 6.24, the initial favourable conditions and made an increase in starch and dye concentrations. A comparison between the manual and controlled operation of the reactor revealed that its response i.e. its effluent TOD, average OD and the CO_2 is favoured also when controlled by the ANNBCS. Those values did not go above 1800 mg l^{-1}, 1.05 TCU, and 31 %, respectively. These maximum values meant that the working condition of the UASB reactor was in good health and was within the organic and colour constraint limits set during training.

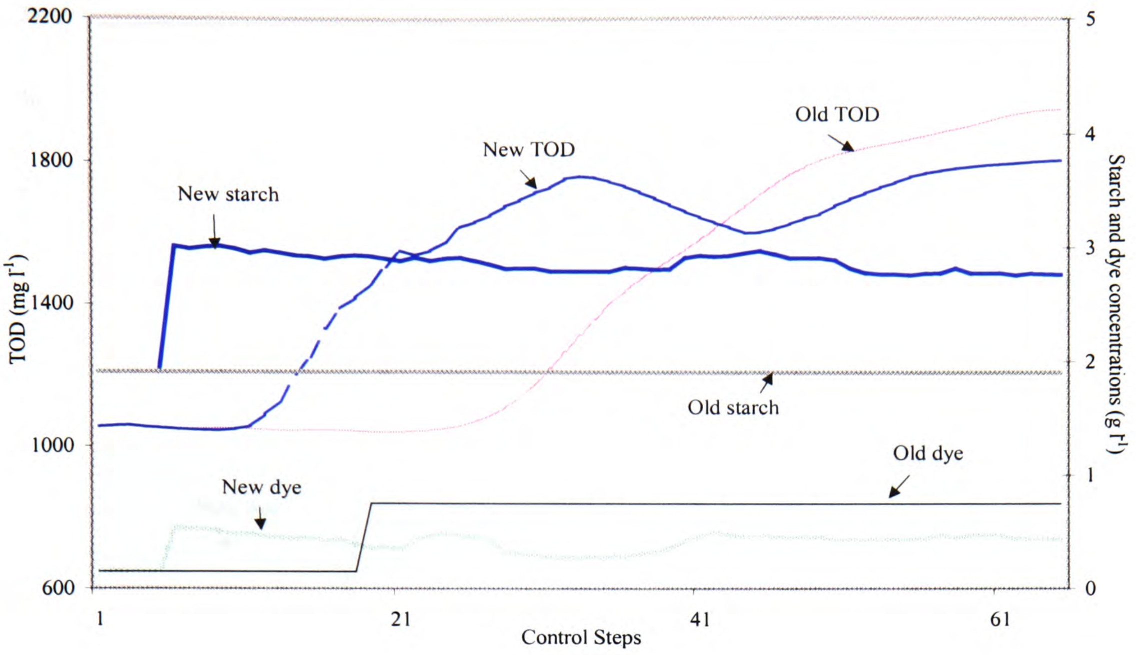


Figure 6.25 - Response of the ANNBCS to sensorial information for *Test B* – Changes to the input parameters (starch and dye) vs. TOD

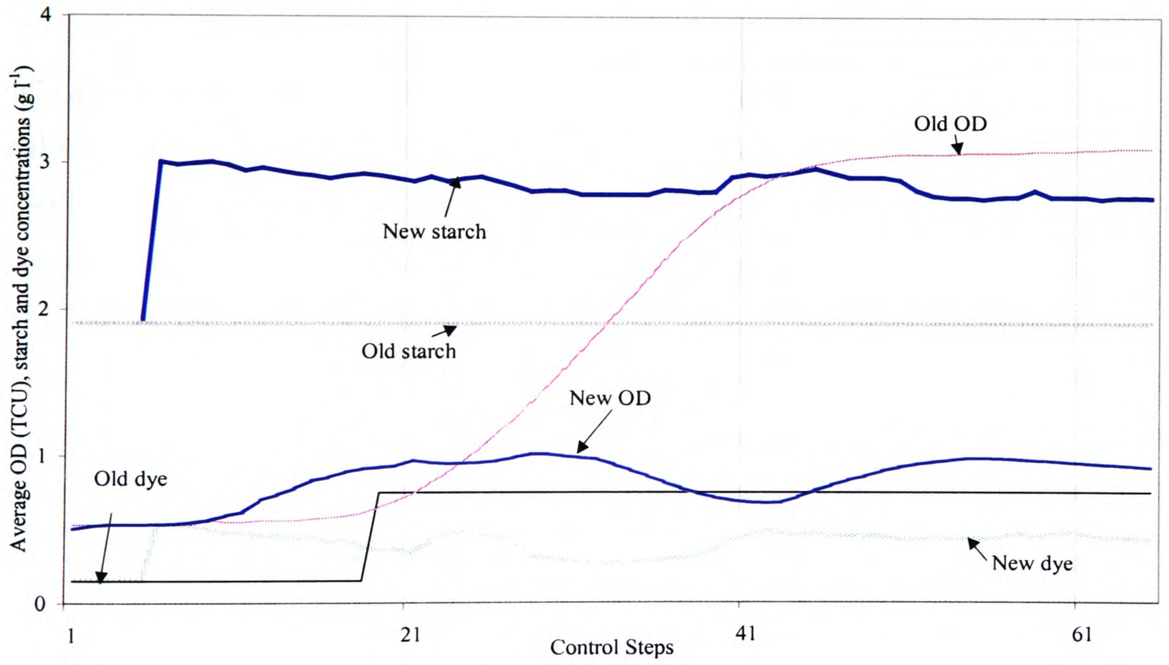


Figure 6.26 - Response of the ANNBCS to sensorial information for *Test B* – Changes to the input parameters (starch and dye) vs. average OD

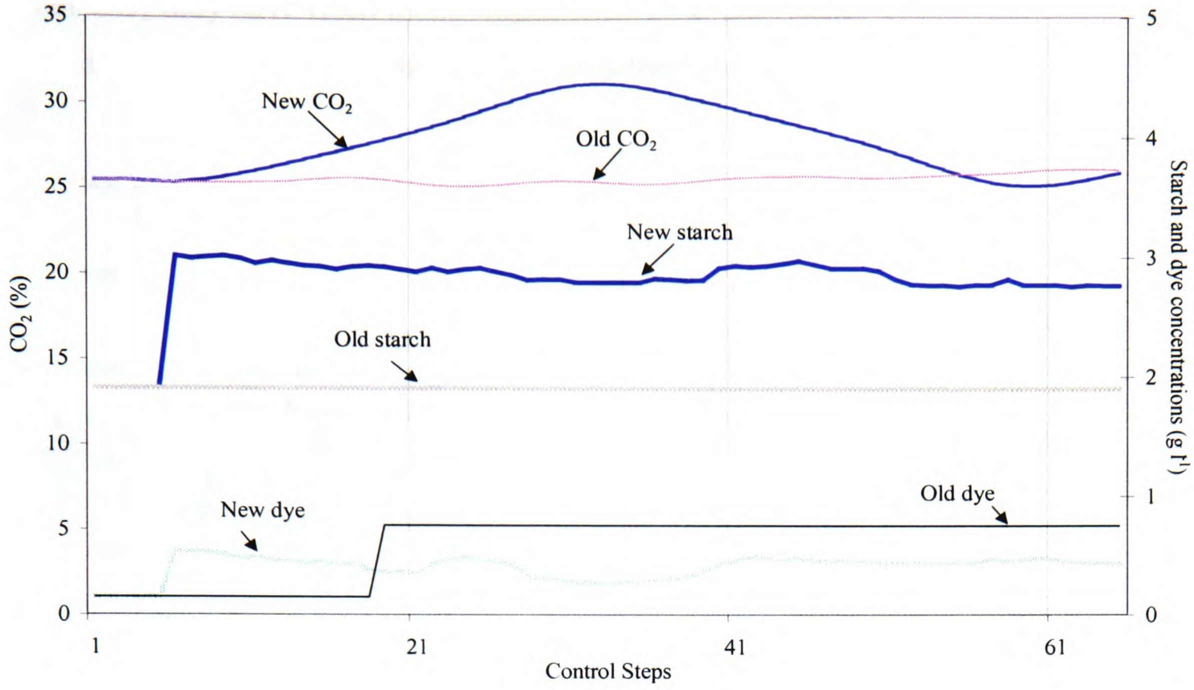


Figure 6.27 - Response of the ANNBCS to sensorial information for *Test B* – Changes to the input parameters (starch and dye) vs. CO_2

Test C - Simulation of the ANNBCS response to high dye and starch loads

A final test, *Test C* serves to confirm the feasibility of the ANNBCS at controlling the starch and dye loads when both concentrations were higher than the reactor could cope with. It can be seen in Figures 6.28 to 6.30, that the ANNBCS reduced both the starch and dye loads to roughly half of the loads used during manual operation. With these suggestions, the TOD values were reduced to below 1800 mg l^{-1}, the average OD below 1.05 TCU and the CO_2 remained below 31 %. It is important to point out that, the use of CO_2 as a monitoring parameter in which a control action could be based upon is of extreme importance as it responded faster than the other process variables, for example TOD to changes in the input parameter (i.e. variation of starch load). This phenomenon is clearly shown in Figures 6.28 and 6.30. After control step 28 the ANNBCS suggested a reduction in starch load to the reactor, from 2.9 g l^{-1} to 2.7 g l^{-1}. This need for reduction was taken based on the CO_2 values, which have increased to around 30 %, and not by the TOD values which were still below 1800 mg l^{-1}. If the CO_2 measurements were not considered, the decrement in the starch load would have come only when the values of TOD have risen above the limit. At this time the values of CO_2 may have risen much higher than what the reactor could cope with, meaning that the health of the methanogenic bacteria was then at risk.

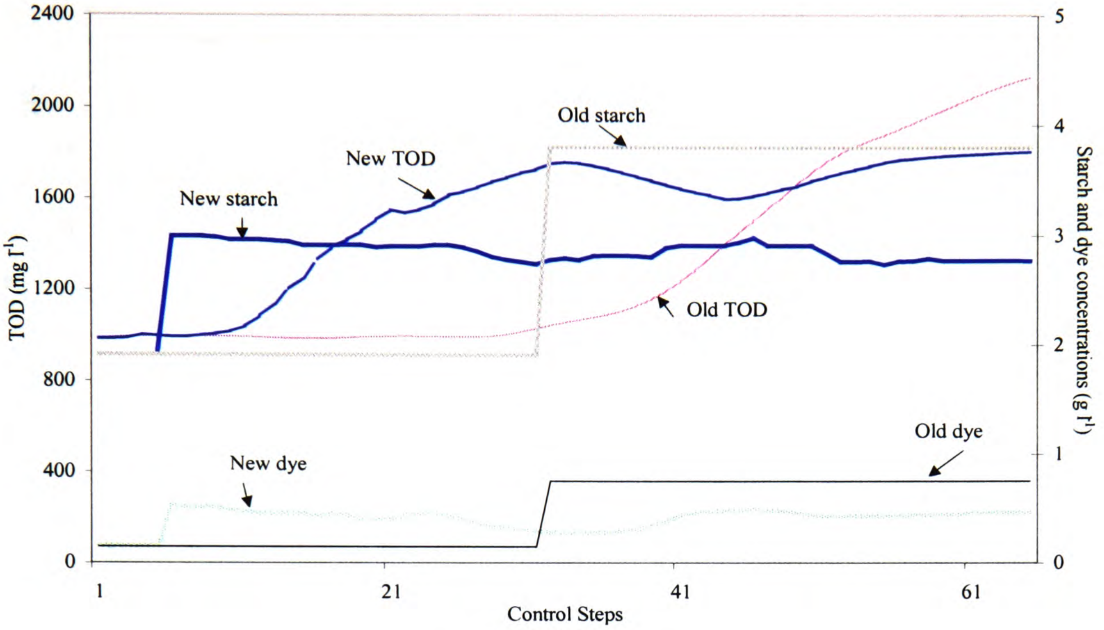


Figure 6.28 - Response of the ANNBCS to sensorial information for *Test C* – Changes to the input parameters (starch and dye) vs. TOD

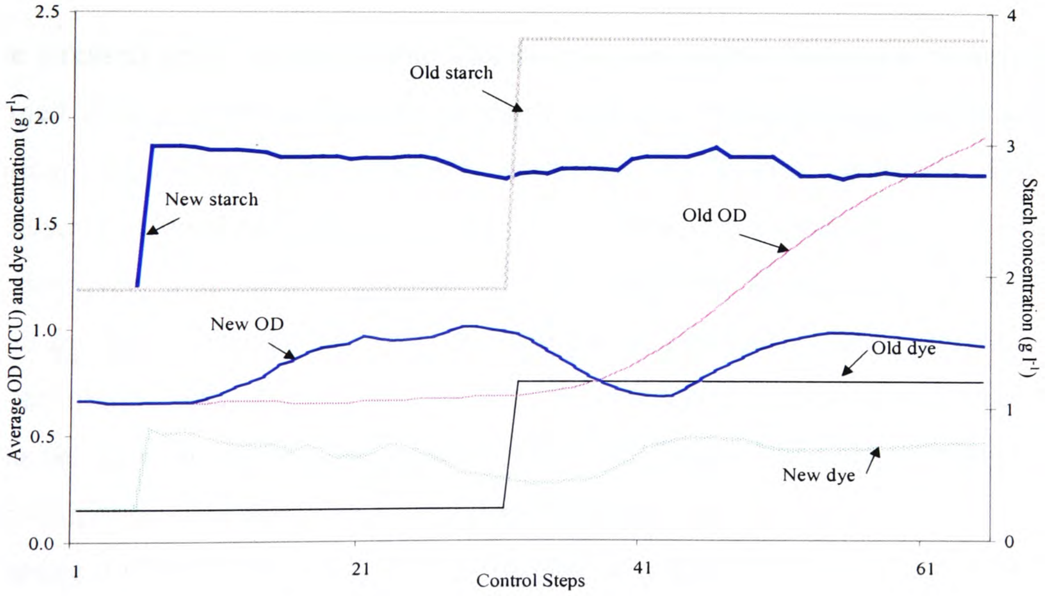


Figure 6.29 - Response of the ANNBCS to sensorial information for *Test C* – Changes to the input parameters (starch and dye) vs. average OD

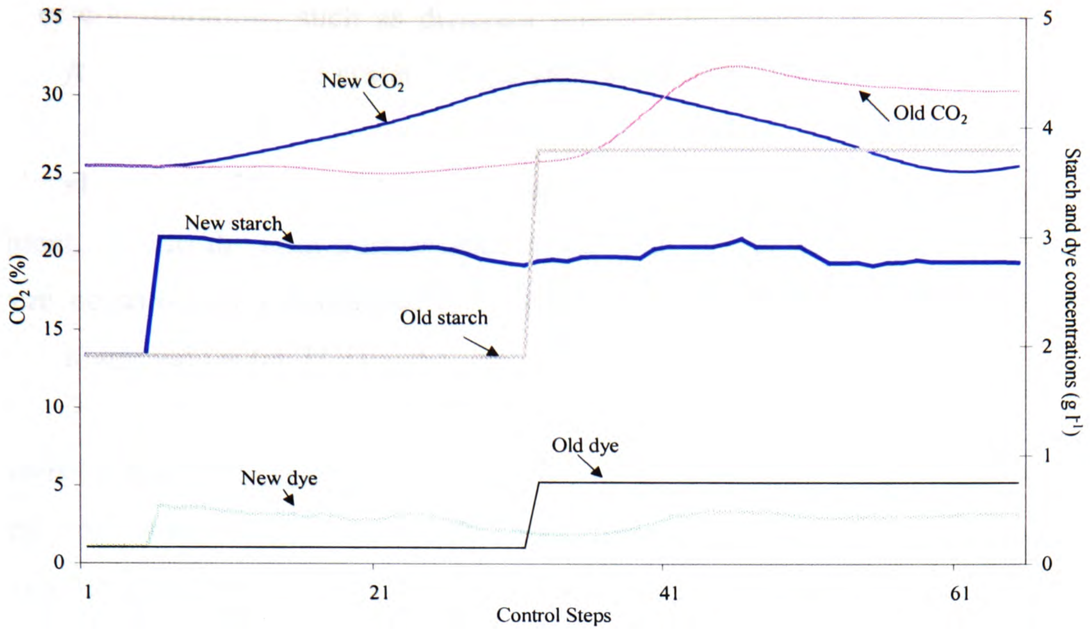


Figure 6.30 – Response of the ANNBCS to sensorial information for *Test C* – Changes to the input parameters (starch and dye) vs. CO_2

6.11. Conclusions from the UASB Reactor Modelling and Usage of the ANNBCS in a Computer Simulation

It must be stressed here that the system identification of such a biological treatment process as performed in this work can be seen in many ways as a simplistic approach compared to mathematical modelling. This relative simplistic modelling approach lacks model transparency, as it does not allow the user to understand the underlying dynamics of the process. However, it is able to produce accurate one step ahead (i.e. 1 h in the future) predictions for TOD, average OD and CO_2 both using the training and validation data sets. The pure simulation tests showed that the NNARX models were also able to make futuristic predictions for seen data over a typical time period of 25 days. The pure simulation test using unseen or validation data were not at all disastrous yielding a reasonable prediction from the TOD, average OD and CO_2 models, being the average OD model the least accurate. However, in terms of fluctuations in the predictions the CO_2 model may be more critical when used in conjunction with the ANNBCS. To overcome the difficulty of obtaining better pure simulation results with the validation data set, a more comprehensive data set could be used. Other configurations such as different numbers of hidden neurones, decreasing the NSSE, delays, and order of the system might yield some improvements.

Having developed the CO_2 model, it was used by the ANNBCS in a computer simulation environment. Due to the poor performance of the CO_2 model the values of starch and dye loads were occasionally sub-optimal (i.e. below 2.9 g l^{-1} starch and 0.45 g l^{-1} dye) with consequent lower values for TOD and average OD.

The ANNBCS has been found to be able to mimic the action of an expert operator by increasing or decreasing the starch and dye loads intake upon detection of satisfactory/unsatisfactory UASB reactor condition. Such changes in the load were performed without jeopardising the integrity of the reactor represented by the models, namely it maintained acceptable levels of the process parameters (i.e. TOD < 1800 mg l^{-1}; average OD < 1.10 TCU and $CO_2 < 32$ %). In addition, one can also conclude that the NNARX models of the reactor behaved very well in a pure simulation environment, although its use is strictly confined to a specific reactor operating under these specific conditions.

The ANNBCS actions were very similar throughout the three *Tests (A, B* and *C)*. These resulted as it was controlling based on the NNARX models' outputs and not the physical reactor where unknown disturbances (e.g. biomass growth or loss, inhibitory agent) could occur. Disturbances such as an increase in the treatment efficiency due to biomass growth or a decrease in case of biomass loss or intrusion of an inhibitory agent could happen in real life.

This ANNBCS would only work if the treatment efficiency of the UASB reactor would be close to the ones used here so its control actions would fall into the ones used to train it. As for any ANNBCS, any big changes to the learned relationships would have to be incorporated by re-training. Instead, an on-line trained ANNBCS could also be an alternative. A more sophisticated control scheme that takes into account the delays of the system could be more advantageous than just a static mapping approach.

There are considerable advantages in using models that can represent the UASB reactor. They could be used in a control strategy for example to substitute the small reactors used widely to indicate the effects of such a waste in the real and bigger reactor. Simulation could be used instead of experimental runs, which can be labour intensive, costly and could risk the reactor's health. However, it is important to note that the models must be built based on quite a wide range of operating conditions to better represent the physical process. This computer simulation could also be utilised to detect what would be the most important parameters to be fed into the ANNBCS. For example the same computer simulation could be used to detect the effect on the performance of not modelling one of the monitoring parameters and hence not using it by the ANNBCS. Extra investigations could also take place to study the use of other monitoring parameters within the ANNBCS such as biogas hydrogen, VFAs and so on. Other advantages come from the ability of using this computer simulation in order to evaluate the effect of possible control/remedial actions more intensely such as addition of nutrients or recycling biomass.

7. CONCLUSIONS AND RECOMMENDATIONS FOR FURTHER WORK

The main objective of this project was to develop and test on-line an original ANNBCS to improve the performance of an UASB reactor and an aerobic biotreatment stage for treatment of a STE. The control scheme needed to output remedial actions for different 'health' states of the biotreatment processes and quality of their effluents, namely in terms of organic and colour concentrations, reducing at the same time the operating costs (e.g. control of chemical addition and oxygen input) and be tolerant to sensor loss. Other objectives were also inherent to this project, namely the selection of: appropriate ANNs for use in the ANNBCS; the most useful on-line monitoring parameters (in terms of usefulness, response time, maintenance and reliability) used by the ANNBCS.

It can be concluded from the work presented here that the objectives were achieved. ANNBCSs were configured to control simultaneously multiple parameters of the biological processes during organic and/or colour step loads and to tolerate sensor loss with some success. The ANNBCSs were tested using a range of on-line instruments to measure BA, pH, colour, DO, TOD, TOC, temperature, biogas flowrate and H_2 and CO_2 in the UASB reactor gas space. For the UASB reactor a few remedial actions were used by the ANNBCSs, for testing them off-line (i.e. adjustment of BA, dye and starch loads, and increase in carbon source) and for testing them on-line (i.e. adjustments of BA and dye intakes). For the aerobic stage, remedial actions such as adjustments of starch, acid and oxygen intakes to the aerobic tank were performed by the ANNBCS.

In addition to the referred achievements other work was also accomplished. Detailed studies were performed on applying filtering systems and a biocide for use with the on-line colour analyser, which helped improve the accuracy of the readings and also prolonged the monitoring time before maintenance was required. Real time hardware and software links from the TOC, BA and colour monitors to the main computer were established. Also improvements to the intermittent BA monitor working mode were made. Interface of the monitoring and control software (LabVIEW™ and MATLAB®) and hardware was performed. This thesis also highlighted the improvements that could be gained on the controller's performance through the integration of NNARX models into a control scheme.

Specific conclusions from this work can be seen in Chapters 4, 5 and 6. However, a summary of the main conclusions is following presented:

- On-line colour measurements must be performed at various wavelengths using a well filtered sample with added biocide to counter bacterial fouling.
- The on-line TOD instrument described may not be used with industrial wastes containing significant mineral content because of blockages. Another instrument for monitoring organic strength both at the influent to the UASB reactor and at the effluent are a priority for RTC.
- It is recommended that all the on-line measurements (except for TOD) must be used by the ANNBCS for control of the textile WWT.
- The biomass catalase activity monitor may be used in a control scheme for the activated sludge process without the need for off-line measurements of MLSS or VSS.
- ANNs such as linear, BP, RBF, Elman, SOM and LVQ networks were tested and selected for integration within the ANNBCS, based on the accuracy of the network predictions, time required for the necessary training and the size of the training data. Results demonstrated that a hybrid structure containing a LVQ network followed by a series of BP networks was the most efficient at dealing with different colour, organic and BA load conditions whilst being least influenced by sensor failure.
- The two ANNBCSs used for on-line control performed successfully, as they suggested appropriate remedial actions and responded accordingly to the training examples.
- Accurate NNARX models of the UASB reactor were built to predict CO_2 concentrations in the biogas and TOD and average OD of the reactor's effluent, even during pure simulation.

- The ANNBCS tested on the NNARX models using a computer simulation performed well. Adjustments of the starch and dye intake loads to the UASB reactor were successfully determined by the ANNBCS using the NNARX models' predictions.

Using a similar laboratory scale plant the following recommendations are outlined for further work:

- Perform similar experiments as the ones presented here with a more complex STE or with a real waste. This effluent would then include for example other dyes mixture and surfactants.
- Test toxicity monitors such as a rapid anaerobic toxicity tester - RANTOX (Rozzi *et al.*, 1995) for the anaerobic stage and Activity and Nitrification Analyser - ANITA (Massone and Rozzi, 1997) and RODTOX (Vanrolleghem *et al.*, 1990) for the aerobic stage and possibly incorporate their output within the ANNBCS. Test also the use of a dissolved hydrogen monitor (e.g. Cord-Ruwisch *et al.*, 1997), correlate its results with other monitored parameters and possibly incorporate it within the ANNBCS.
- Use on-line instruments such as TOC and colour monitors with multi-channel capabilities, that is, before the entrance in the UASB reactor, before going to the aerobic stage and in the final effluent quality. Duplicate measurements of such parameters as pH, DO and temperature so they could be used by the ANNBCS to identify sensor failure.
- Integrate the biomass catalase activity instrument within an ANNBCS to control the RAS flowrate and possibly the addition of nutrients and other supplements.
- A more sophisticated ANNBCS that takes into account the delays of the system could be more advantageous than just a static mapping approach.
- Use a measurement of aromatic amines if possible on-line for determination of process performance and integrate it within the ANNBCS.
- Use if possible an on-line monitor for measuring anaerobic biomass activity or for the loss rate or at least an off-line technique that could provide some 'early' indication of the deterioration of granules for which no remedial actions were successful in tackling the event after they started to float (e.g. Dubourguier *et al.* (1988) used: direct examination by light microscopy for observing bacterial conglomerates; staining with toluidine blue

for identification of active bacteria; X-ray analysis for observation of mineral precipitates; transmission electron microscopy to study the ultrastructure of the granules; SEM for surface examination and location of each bacterium inside the granule).

- In RTC, incorporate NNARX model(s) of the UASB reactor together with the ANNBCS developed. The model(s) would predict for example 3 hours ahead and rapid remedial actions could then take place before the reactor's efficiency would have deteoriorated drastically.
- A computer simulation such as the one performed here could be used to detect other important parameters to be used by the ANNBCS such as the concentration of biogas H_2, and of VFAs.
- A similar computer simulation study could be performed for the aerobic stage.
- Other remedial actions to be used by the ANNBCSs for both biotreatment stages could be tested. These could be: adjustment of nutrients (different types of nutrient analysers have been used for close loop control of activated sludge plants – Lynggaard-Jensen *et al.*, 1996) and trace elements flow for both stages, recycle anaerobic biomass to the reactor; recycle the effluents from both stages for re-treatment; use GAC (Walker and Weatherlay, 1999) or PAC (Lin, 1993) in a separate module, for example after the UASB reactor; use of a H_2O_2 unit after the UASB reactor in case of a colour overload (McCurdy *et al.*, 1992); and control the RAS and WAS in the aerobic stage.

It would be also useful to apply a similar control scheme to a real industrial plant using real textile effluent after re-training of the networks involved.

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APPENDICES

Appendix A - Monitoring and control hardware

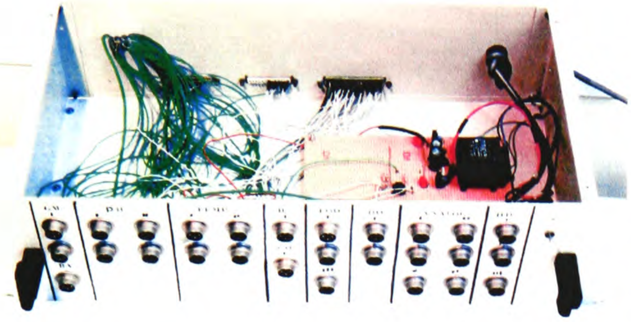

Figure A.1 – Interface Box 1

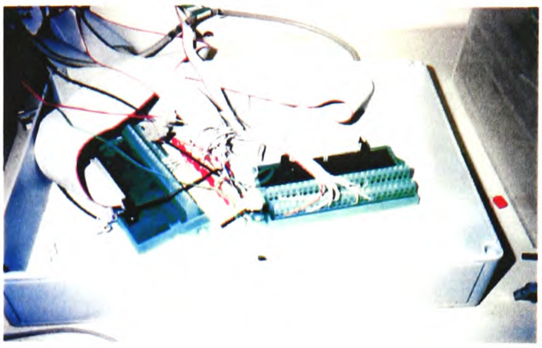

Figure A.2 – Interface Box 2

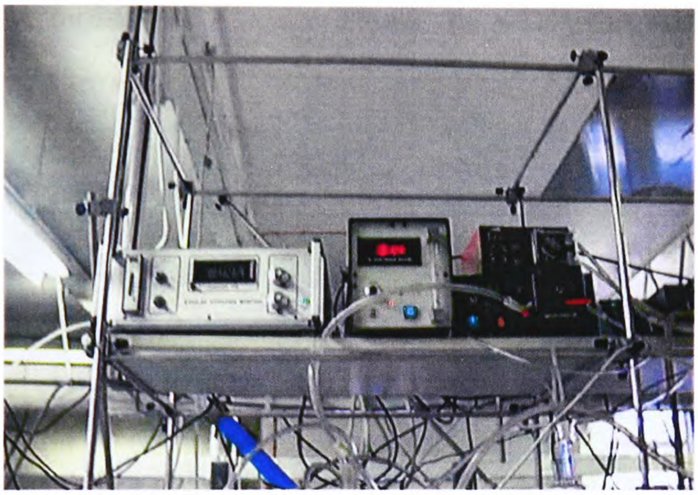

Figure A.3 – From the left: H_2 monitor, CO_2 analyser and respective pumps. Also part of the H_2S scrubber at the bottom

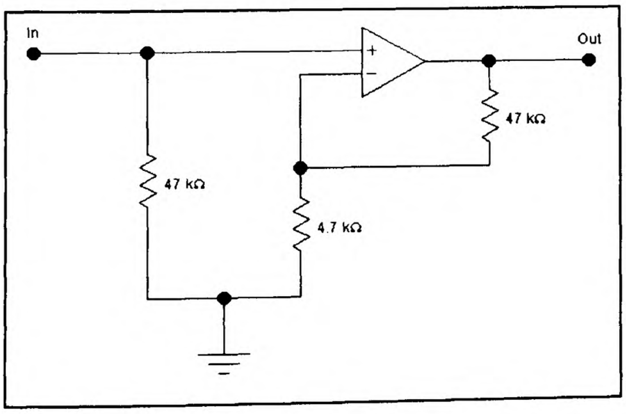

Figure A.4 - Electronic circuit built for conversion from current to voltage signal with 10-fold amplification

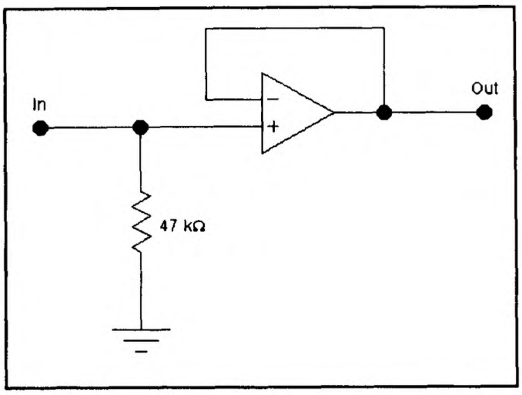

Figure A.5 - Electronic circuit built for stability of the CO_2 analyser signal

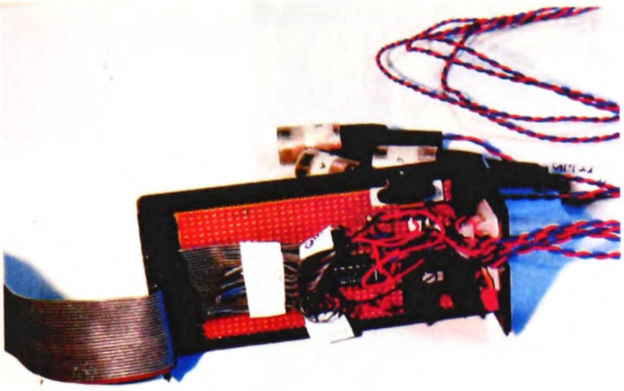

Figure A.6 – Interface Box 4

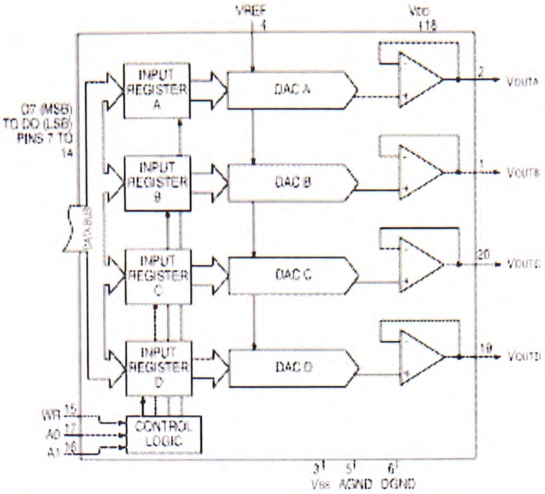

Figure A.7 – D/A Converter 8-Bit Quad

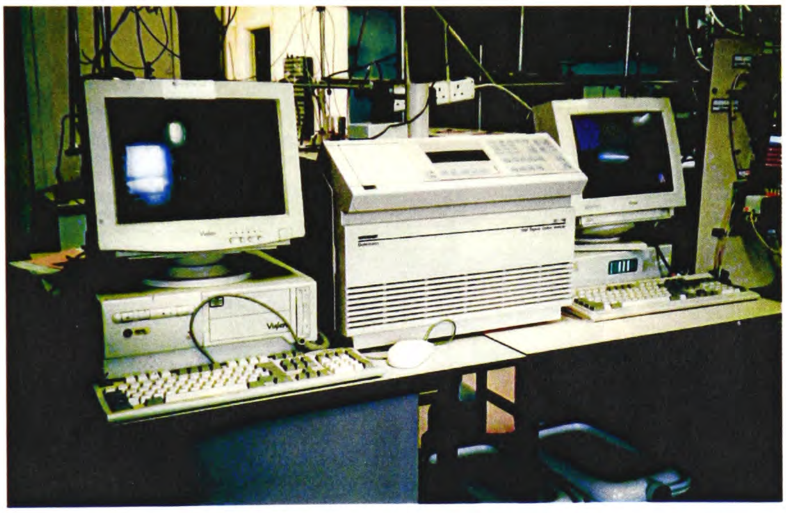

Figure A.8 - From the left: UV/Visible Spect. dedicated PC, TOC analyser, and intermittent BA analyser dedicated PC

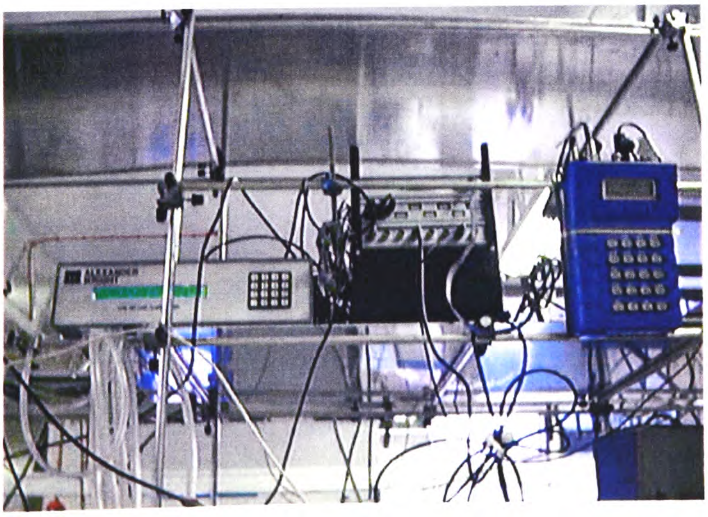

Figure A.9 – From the left: LFM 300 gas meter, MX8000 and WP4007

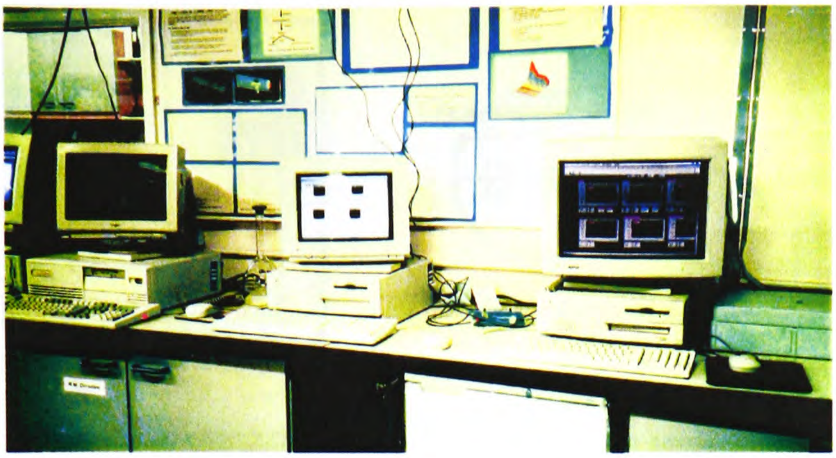

Figure A.10 – From the left: PC for the TOC analyser (DC190 Terminal Software), Macintosh for the TOC analyser and CB50, central computer

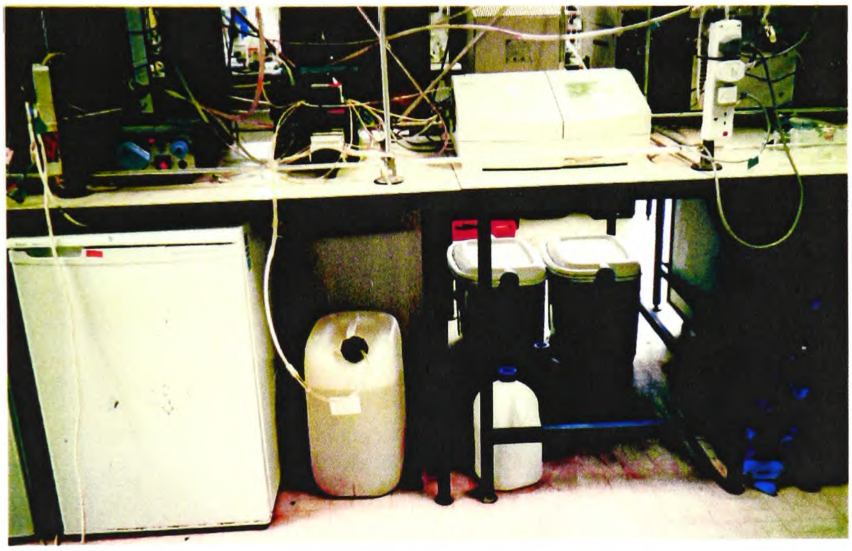

Figure A.11 – UV/Visible Spect. (top right) and DI + biocide tank under the bench

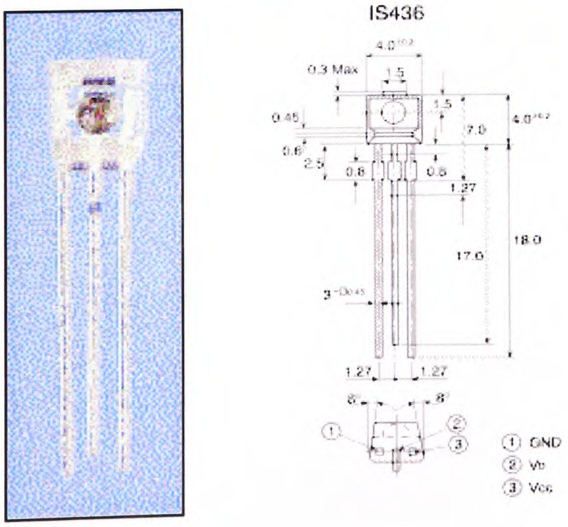

Figure A.12 - Opto Schmitt Trigger Detectors (IS436)

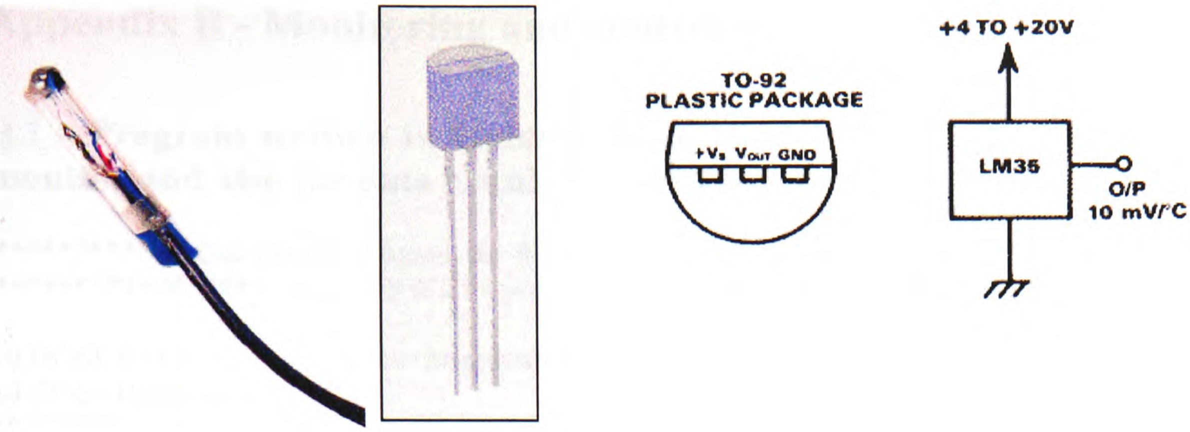

Figure A.13 - Temperature sensor IC LM35 and temperature probe

Appendix B - Monitoring and control software

B.1 - Program written in QuickBasic for instructing the intermittent BA monitor and also for data acquisition and output data

```
'************ QuickBasic Program for the Intermittent BA Analyser *********
'***************** Textile WWT Project *****************

10 GOSUB start            '(main program).
20 GOSUB control1
30 GOSUB measurement
40 GOSUB CONV
50 GOSUB SENDAC
60 GOSUB control2

start:                '(subroutine).

ba% = &H300
COLOR (8), (15)
OUT ba% + 11, 255         '(all valves and pumps de-energised).
CLS
PRINT "             Welcome to the Bicarbonate Analysis"
PRINT "             ******************************"
PRINT "If you want to start the operation program for the analyser please first make"
PRINT "sure that the electrical circuit of the analyser is switched on!"
again:
PRINT
PRINT "Do you want to start the program now? (yes = 1, cancel = any other number)"
PRINT
INPUT "---> ", answ1
IF answ1 = 1 THEN
PRINT
PRINT "Is the electrical circuit switched on? (yes = 1, no = any other number)"
ELSE
GOTO stopping
END IF
PRINT
INPUT "---> ", answ2
IF answ2 = 1 THEN
GOTO continue
ELSE
GOTO again

continue:
END IF
RETURN

cycles:               '(subroutine).

CLS
COLOR (9)
PRINT "    Purging Cycle"
```

```
PRINT "    Discharging Cycle"
PRINT "    Sample Filling Cycle"
PRINT "    Saturation Cycle"
PRINT "    Acidification Cycle"
PRINT "    Measurement Cycle"
PRINT "    Discharging Cycle"
PRINT "    Cleaning Cycle"
PRINT "    Discharging Cycle"
PRINT "    Waiting Cycle"
PRINT
PRINT
COLOR (5)
PRINT "    Temperature:"; temp%; "Degree Centigrade"
PRINT "    Pressure:   "; press%; "mbar"
PRINT "    BA:         "; CARBO%; "mg CaCO3/l"
RETURN

control1:               '(subroutine).

'****************************** Purging ******************************
GOSUB cycles
LOCATE 1, 1
COLOR (12)
PRINT ">>>>"
COLOR (8)
OUT ba% + 11, 190       '(peristaltic pump on/outlet gas v. on).
OUT ba% + 11, 58        '(sample valve on).
SLEEP (6)

'****************************** Discharging **************************
GOSUB cycles
LOCATE 2, I
COLOR (12)
PRINT ">>>>"
COLOR (8)
OUT ba% + 11, 63        '(peristaltic p. off/d. fluid v. off).
SLEEP (5)
OUT ba% + 11, 191       '(drain valve off).
SLEEP (2)

'****************************** Filling ******************************
OUT ba% + 11, 190       '(peristaltic pump on).
SLEEP (2)
GOSUB cycles
LOCATE 3, 1
COLOR (12)
PRINT ">>>>"
COLOR (8)
OUT ba% + 11, 186       '(digester fluid valve on).
SLEEP (94)            '(sample in/filling time).
OUT ba% + 11, 190       '(digester fluid valve off).
OUT ba% + 11, 191       '(peristaltic pump off).
OUT ba% + 11, 189       '(circulating pump on).
```

```
SLEEP (2)

'****************************** Saturation ****************************
GOSUB cycles
LOCATE 4, 1
COLOR (12)
PRINT ">>>>"
COLOR (8)
OUT ba% + 11, 173          '(CO2 valve on).
SLEEP (180)              '(CO2 saturation time).
OUT ba% + 11, 189          '(CO2 valve off).
SLEEP (2)

'****************************** Acidification **************************
OUT ba% + 11, 253          '(outlet gas valve off).
SLEEP (2)
OUT ba% + 11, 252          '(peristaltic pump on).
SLEEP (2)
GOSUB cycles
LOCATE 5, 1
COLOR (12)
PRINT ">>>>"
COLOR (8)
OUT ba% + 11, 244          '(acid valve on).
SLEEP (76)               '(acidification time).
OUT ba% + 11, 252          '(acid valve off).
OUT ba% + 11, 253          '(peristaltic pump off).
SLEEP (20)               '(CO2 gas evolution).
OUT ba% + 11, 255          '(circulating pump off).
SLEEP (5)
RETURN

measurement:             '(subroutine).

'****************************** Measurement *************************
GOSUB cycles
LOCATE 6, 1
COLOR (12)
PRINT ">>>>"
COLOR (8)

t = 0                  '(temperature measurement).
FOR a% = 1 TO 50
OUT ba% + 1, 0
OUT ba% + 2, 0
l = INP(ba% + 3)
h = INP(ba% + 4)
t = (l + h * 256) / 4.095 + t
FOR b% = 1 TO 2000: NEXT b%
NEXT a%
temp% = t / 50

p = 0                  '(pressure measurement).
```

```
FOR c% = 1 TO 50
OUT ba% + 1, 2
OUT ba% + 2, 2
l = INP(ba% + 3)
h = INP(ba% + 4)
p = (l + h * 256) / .4095 + p
FOR d% = 1 TO 2000: NEXT d%
NEXT c%
IF ((p / 50) - 40) < 0 THEN
press% = 0
ELSE
press% = ((p / 50) - 40) * 1.06369280723# * ((p / 50) - 40) ^ .043260703617#
END IF
                    '(carbonate equation).
CARBO% = (((press% / 1000 * (.0775 / (.082057 * (273.15 + temp%)) + .06406 * .973857 ^ temp%
* .0499)) / (.0499 * .00002)) * 1.2) * 1.1667 * 1.25

                    '(saving data in file ba.dat).
OPEN "ba.dat" FOR APPEND AS #1
WRITE #1, DATE$, TIME$, temp%, press%, CARBO%
CLOSE #1
RETURN

'******************** Conversion ******************************

CONV:  '(Conversion from digital to analogue)
BB% = CARBO%
msb11% = 0: msb10% = 0: msb9% = 0: msb8% = 0
IF BB% >= 2048 THEN msb11% = 1: BB% = BB% - 2048
IF BB% >= 1024 THEN msb10% = 1: BB% = BB% - 1024
IF BB% >= 512 THEN msb9% = 1: BB% = BB% - 512
IF BB% >= 256 THEN msb8% = 1: BB% = BB% - 256
lsb% = BB%
msb% = (msb11% * 8) + (msb10% * 4) + (msb9% * 2) + (msb8% * 1)
'PRINT "lsb="; lsb%
'PRINT "msb="; msb%
RETURN

SENDAC:
OUT ba% + 5, lsb%
OUT ba% + 6, msb%
RETURN

control2:             '(subroutine).

'****************************** Discharging *************************
GOSUB cycles
LOCATE 7, 1
COLOR (12)
PRINT ">>>>"
COLOR (8)
OUT ba% + 11, 127         '(drain valve on).
SLEEP 2
```

```
OUT ba% + 11, 63          '(outlet gas valve on).
SLEEP (10)              '(sample out/discharging time).
OUT ba% + 11, 191          '(drain valve off).

'****************************** 1. Cleaning **************************
LOCATE 8, 1
COLOR (12)
PRINT ">>>>"
COLOR (8)
OUT ba% + 11, 159          '(water valve on).
SLEEP (12)              '(cleaning water in/filling time).
OUT ba% + 11, 191          '(water valve off).
OUT ba% + 11, 189          '(circulating pump on).
SLEEP (12)              '(1. cleaning time).
OUT ba% + 11, 191          '(circulating pump off).

'****************************** Discharging **************************
GOSUB cycles
LOCATE 9, 1
COLOR (12)
PRINT ">>>>"
COLOR (8)
OUT ba% + 11, 63          '(drain valve on).
SLEEP (15)              '(cleaning water out/discharging time).

'****************************** 2. Cleaning **************************
GOSUB cycles
LOCATE 8, 1
COLOR (12)
PRINT ">>>>"
COLOR (8)
OUT ba% + 11, 159          '(drain valve off/water valve on).
SLEEP (18)              '(cleaning water in/filling time).
OUT ba% + 11, 191          '(water valve off).
OUT ba% + 11, 189          '(circulating pump on).
SLEEP (20)              '(2. cleaning time).
OUT ba% + 11, 191          '(circulating pump off).

'****************************** Discharging **************************
GOSUB cycles
LOCATE 9, 1
COLOR (12)
PRINT ">>>>"
COLOR (8)
OUT ba% + 11, 63          '(drain valve on).
SLEEP (15)              '(cleaning water out/discharging time).

'****************************** 3. Cleaning **************************
GOSUB cycles
LOCATE 8, 1
COLOR (12)
PRINT ">>>>"
COLOR (8)
```

```
OUT ba% + 11, 159          '(drain valve off/water valve on).
SLEEP (19)               '(cleaning water in/filling time).
OUT ba% + 11, 191          '(water valve off).
OUT ba% + 11, 189          '(circulating pump on).
SLEEP (20)               '(2. cleaning time).
OUT ba% + 11, 191          '(circulating pump off).

'****************************** Discharging **************************
GOSUB cycles
LOCATE 9, 1
COLOR (12)
PRINT ">>>>"
COLOR (8)
OUT ba% + 11, 63          '(drain valve on).
SLEEP (15)              '(cleaning water out/discharging time).

'****************************** 4. Cleaning **************************
GOSUB cycles
LOCATE 8, 1
COLOR (12)
PRINT ">>>>"
COLOR (8)
OUT ba% + 11, 159          '(drain valve off/water valve on).
SLEEP (19)               '(cleaning water in/filling time).
OUT ba% + 11, 191          '(water valve off).
OUT ba% + 11, 189          '(circulating pump on).
SLEEP (20)               '(2. cleaning time).
OUT ba% + 11, 191          '(circulating pump off).

'****************************** Discharging **************************
GOSUB cycles
LOCATE 9, 1
COLOR (12)
PRINT ">>>>"
COLOR (8)
OUT ba% + 11, 63          '(drain valve on).
SLEEP (15)              '(cleaning water out/discharging time).

'****************************** 5. Cleaning **************************
GOSUB cycles
LOCATE 8, 1
COLOR (12)
PRINT ">>>>"
COLOR (8)
OUT ba% + 11, 159          '(drain valve off/water valve on).
SLEEP (12)               '(cleaning water in/filling time).
OUT ba% + 11, 191          '(water valve off).
OUT ba% + 11, 189          '(circulating pump on).
SLEEP (20)               '(2. cleaning time).
OUT ba% + 11, 191          '(circulating pump off).

'****************************** Discharging **************************
GOSUB cycles
```

```
LOCATE 9, 1
COLOR (12)
PRINT ">>>>"
COLOR (8)
OUT ba% + 11, 63          '(drain valve on).
SLEEP (15)               '(cleaning water out/discharging time).

'****************************** Waiting ******************************
GOSUB cycles
LOCATE 10, 1
COLOR (12)
PRINT ">>>>"
COLOR (8)
SLEEP (4200)              '(waiting time).
GOTO 20                  '(start of a new analysis procedure).
stopping: END
```

B.2 - Program written in QuickBasic for instructing the UV/Visible Spectrophotometer and also to output data

```
'GOTO dummy
test:
OPEN "com1:9600,n,8,1" FOR RANDOM AS #1
PRINT #1, "TER"; CHR$(13); CHR$(10);
'PRINT #1, "LIS"; CHR$(13); CHR$(10);
'PRINT #1, "BAS"; CHR$(13); CHR$(10);
SLEEP 30
PRINT #1, "MOD ABS"; CHR$(13); CHR$(10);
SLEEP 10
PRINT #1, "WDR 436"; CHR$(13); CHR$(10);
SLEEP 40
'PRINT #1, "FIX 1"; CHR$(13); CHR$(10);
SLEEP 10
PRINT #1, "RUN"; CHR$(13); CHR$(10);
SLEEP 10
PRINT #1, "WDR 525"; CHR$(13); CHR$(10);
SLEEP 40
PRINT #1, "RUN"; CHR$(13); CHR$(10);
SLEEP 10
PRINT #1, "WDR 620"; CHR$(13); CHR$(10);
SLEEP 40
PRINT #1, "RUN"; CHR$(13); CHR$(10);
SLEEP 20
'PRINT #1, "GET"; CHR$(13); CHR$(10);
s = 0
b$ = ""
c$(1) = ""
c$(2) = ""
c$(3) = ""
FOR v = 1 TO 11
INPUT #1, a$
PRINT a$
```

```
b$ = b$ + a$
NEXT
CLOSE #1

'dummy:
's = 0
'b$ = "--1.1234-3.5234-3.1234--"
PRINT "len= "; LEN(b$);
FOR n = 1 TO LEN(b$)
IF MID$(b$, n, 1) = "." THEN GOSUB this
NEXT
PRINT c$(1), c$(2), c$(3)
'Dilution factor
c(0) = VAL(c$(1)) * 6.533
c(1) = VAL(c$(2)) * 6.533
c(2) = VAL(c$(3)) * 6.533
c(3) = (c(0) + c(1) + c(2)) / 3
PRINT c(0), c(1), c(2), c(3)
GOSUB that
IF INKEY$ = "e" THEN END
GOTO test
'GOTO dummy

this:
s = s + 1
PRINT "this"
FOR m = n - 1 TO n + 4
c$(s) = c$(s) + (MID$(b$, m, 1))
NEXT
RETURN

that:
PRINT "that"
FOR s = 0 TO 3
OUT &H37A, s + 4
'rem IF c(s) > INT(c(s)) + .5 THEN h = INT(c(s)) + 1: GOTO other
h = c(s)
other:
OUT &H378, (h * 18.21)

FOR m = 1 TO 10: NEXT
OUT &H37A, s
PRINT , h * 18.21
NEXT
RETURN
```

B.3 - Program written in MATLAB® for controlling the UASB reactor on-line

```
echo on
t = clock;
```

```
for k=1:99999
    fid=-1;
    while fid==-1
        fid = fopen('Macintosh HD:labVIEW 3.1:data:FEB DATA:11FEB99:3.dat','r');
%(read the excel file)
        pause(1) %(if file is already opened pause 1 s and try again)
    end; %(end the while loop)
    fbv = fscanf(fid,'%f\r\n'); %(matrix fbv is equal the fid file where the values will be displayed %
separatly so use carriage return and line feed)
    fclose(fid); %(close the file)
    [m,n]=size(fbv);
    c=m/11;
    other=reshape(fbv,11,c);
    use=other';
    [a,b]=size(use) %(x is the no. of rows and y the no. of columns)
    BA=use(a,1); pH=use(a,7); avcol=use(a,6);
    Input1=[pH BA]';
    Input2=pH;
    Input3=avcol;
    AA=preanaecontlvq1(Input1);
    if AA==1
        CC=preanaecont1(Input1); % output for extra BA addition
    else AA==2
        CC=preanaecont2(Input2); % output for extra BA addition
    end
    BB=preanaecont3(Input3); % output for dye pump
    Output=[BB CC AA]
    while etime (clock, t) < (k*120); end; %(send output every 1 min)
    disp('waiting cycle')
    fid1=-1;
    while fid1==-1
        fid1 = fopen('Macintosh HD:labVIEW 3.1:data:FEB DATA:11FEB99:4.dat','w')
% (write to a file)
        pause(1)
    end;
    fprintf(fid1,'%10.5f\n',Output);
    fclose(fid1);
end;
```

B.4 - Program written in MATLAB® to train the TOD NNARX model

```
%_______________________________________________________________________
% NNARX Dynamic Model (MISO) for the UASB reactor effluent TOD
%_______________________________________________________________________

% 2 Inputs: concentration of starch and dye
% 1 Output: TOD of the UASB effluent
% _______________________Load inputs and output _______________________
load training.mat
input=input'; % the inputs must be presented raw wise and not column wise
tod=tod'; % the same with the output, when using to predict OD use OD=OD';
% or for CO2 use CO2=CO2';
```

```
input=[starch;dye];
% First thing to do is to scale down the inputs and target. Remember
% with DSCALE, the matrix using DSCALE must be row wise
% [X_sc, X_m&std]=dscale, where the X_sc are the scaled matrix of data and
% X_m&std is the mean and standard deviation of each elements

% ________________________Normalisation of inputs and outputs_______________
[P,Pscale]=dscale(input); [T,Tscale]=dscale(tod);

%_______________Defining the training parameters of the ANN Model_____________
% 2-LAYER 'FF' structure (1 hidden (tansig function)
% and 1 output (linear function) layer)
NetDef=['HHHHHHHHHH'; % no. of neurones in the hidden layer (e.g. 10)
      'L---------']; % no. of neurones in the output layer (1)
na=[2]; % no. of past outputs
nb=[2 2]; % 2nd order of the system, no. of past inputs
nk=[4 4]; % delay for each element in P
NN=[na nb nk]; % combine parameters which define the temporal patterns of the
% system (ARX type regressor as inputs to the model)
max_iter=1000; % maximum iterations
eg=0.0001;    % error goal
lambda=1; D=0; % other training parameters (default)
trparms=[max_iter eg lambda D]; % combine the training parameters

[W1,W2,PI_vector,iteration,lambda]=nnarx(NetDef,NN,W1,W2,trparms,T,P);

% Automatic initialisation of the Network weights and biases and training
% with the function nnarx

%_____________________Plot learning curve____________________________
semilogy(PI_vector); grid; % (PI_vector is the error that the network reaches after
% training)

hold on
semilogy(iteration,ones(size(iteration))*eg,'r-')
xlabel('Iteration'); % x-axis legend
ylabel('Training error'); % y-axis legend

% Saving the weights and associated parameters of the trained network
save tparatod.mat NetDef NN W1 W2 Pscale Tscale

% To rescale back
[w1,w2]=wrescale(W1,W2,Pscale,Tscale,NN);
[prdtod,totalerr]=nnvalid('nnarx',NetDef,NN,w1,w2,tod,input)
```

B.5 - Program written in MATLAB® to prune the NNARX models

```
% ___________________________________________________________
%  Pruning of the developed NNARX Models (Function file)
%____________________________________________________________

function [W1_opt,W2_opt,input_scale,target_scale]=pruning(NetDef,W1,W2,...
```

```
NN,trparms,tr_input,tr_target,vld_input,vld_output)
% W1_opt (optimised weights after pruning)

% Scaling training data set to zero mean and variance 1 and then scale the
% validation set with the same constants.

[tr_input_s, input_scale]=dscale(tr_input);%input_scale=mean and std dev.
                                            %tr_input_s=scaled tr_input.
[tr_target_s,target_scale]=dscale(tr_target);%target_scale=mean and std dev.
                                       %tr_target_s=scaled tr_target.

[m,n]=size(vld_input);
for k=1:m
vld_input_s(k,:)=(vld_input(k,:)-input_scale(k,1))/input_scale(k,2);
end;
vld_output_s=(vld_output-target_scale(1,1))/target_scale(1,2) % Only 1 row.

% Training parameters for the Optimal Brain Surgeon
prparms=[50 5]; % max. iter of 50, 5% elimination of weights - see page 1-16.

% Note that W1 & W2 are the unscaled weights, obtained after training.
% trparms is the training parameters adopted for the function 'nnarx'.
% e.g. trparms=[500 0.015 1 0] refers to the epochs, eg, lambda & D.

[thd,tr_error,FPE,te_error,d_eff,pvec]=nnprune('nnarx',NetDef,W1,W2,...
tr_input_s,tr_target_s,NN,trparms,prparms,vld_input_s,vld_output_s);

% Retrieving the optimal weight matrix.
[minte_error,index]=min(te_error(pvec));
index=pvec(index);
[W1_obs,W2_obs]=netstruc(NetDef,thd,index); % Weights determine by the Optimal Brain
% Surgeon.

% Retraining the network with the Optimum weights without weight decay.
[W1_opt,W2_opt,NSSEvec]=nnarx(NetDef,NN,W1_obs,W2_obs,trparms,tr_target_s,...
tr_input_s);
save d:\MACD1\MOD_CONT\model\prune\odprn_w.mat W1_opt W2_opt ....
input_scale target_scale
```

B.6 - Program written in MATLAB® to train the ANNBCS for testing in B.7

Training the subBP1 (the other 3 SubBPs were similarly trained)

```
% SUB_BP1 Network
echo on
% chan=ddeinit('excel','BP1_data.xls')
% data=ddereq(chan,'r1c1:r279c5');
% save c:\Sandra\Mod_Cont\Control\Cont_tr\Bp1_tr\dataBP1.mat data
cd c:\Sandra\Mod_Cont\Control\Cont_tr\Bp1_tr
load dataBP1.mat
```

```
% Desiccating data into inputs and outputs
Input1=data(:,1:3);
Target1=data(:,4:5);

% Choosing the data
Pm=Input1;
Tm=Target1;
Pm=Pm';
Tm=Tm';

% Find the maximum values of each row in input and target matrix
Pmax=nnmaxr(Pm); Tmax=nnmaxr(Tm);
p1=Pm(1,:)./Pmax(1,1); t1=Tm(1,:)./Tmax(1,1);
p2=Pm(2,:)./Pmax(2,1); t2=Tm(2,:)./Tmax(2,1);
p3=Pm(3,:)./Pmax(3,1);

% Regroup the reformed input and target matrix
P=[p1;p2;p3]; T=[t1;t2];

% Number of input, hidden, and output neurones, respectively
S1=3; S2=20; S3=2;

% Matrix Prange only takes min and max values of each row of matrix P
Prange=[nnminr(P(1,:)) nnmaxr(P(1,:));
        nnminr(P(2,:)) nnmaxr(P(2,:));
        nnminr(P(3,:)) nnmaxr(P(3,:))];

% Initialising the weights and the biases
[W1,b1,W2,b2,W3,b3]=initff(Prange,S1,'logsig',S2,'logsig',S3,'logsig');

% Setting the training parameters
TP(1)=10;%....frequency of progress display in epochs....
TP(2)=10000; %....maximum number of epochs to train...
TP(3)=0.001; %...sum-squared error goal...
TP(4)=0.0001; %...minimum gradient...
TP(5)=0.001; %...initial value for MU...
TP(6)=10; %...Multiplier for increasing MU...
TP(7)=0.1; %...multiplier for decreasing MU....
TP(8)=1e10; %....maximum value for MU....

tp=[TP(1) TP(2) TP(3) TP(4) TP(5) TP(6) TP(7) TP(8)];
[W1,b1,W2,b2,W3,b3,te,tr]=trainlm(W1,b1,'logsig',W2,b2,'logsig',W3,b3,'logsig',P,T,tp);

save subbp1.mat W1 b1 W2 b2 W3 b3

% Simulation of network outputs
a1=simuff(P,W1,b1,'logsig',W2,b2,'logsig',W3,b3,'logsig');

% Multiplying the scale down output of the network by their % respective maximum value
a1a=a1(1,:).*Tmax(1,1);
a2a=a1(2,:).*Tmax(2,1);
```

```
% Regroup the reformed output matrix
A1=[a1a;a2a]

Pmax1=max(data(:,1))
Pmax2=max(data(:,2))
Pmax3=max(data(:,3))

Tmax1=max(data(:,4))
Tmax2=max(data(:,5))

end
```

Training the LVQ network

```
% Control simulation (4 classes)
% three inputs (tod, OD, co2) and 4 classes (low/low, normal, high dye/low starch and high/high)
%.....................Training with LVQ.....................
load lvqtrain.mat
P=[tod;OD;co2];
T=[target1;target2;target3;target4];
[W1,W2]=initlvq(P,12,T); % having 3 subclasses (competitive layer) in each class
tp=[20 5000 0.1];
[W1,W2]=trainlvq(W1,W2,P,T,tp);
save Lvq_we.mat W1 W2
A=simulvq(P,W1,W2)
A=full(A)
end
```

B.7 - Program written in MATLAB® to further evaluate the ANNBCS performance in a computer simulation

```
function [U_new,Y_new]=simul1b1(NetDef_tod,NN_tod,W1opt_tod,...
  W2opt_tod,NetDef_od,NN_od,W1opt_od,W2opt_od,NetDef_co2,...
  NN_co2,W1opt_co2,W2opt_co2,U,Y_all)

% Function File of the Control Simulation

[nu,Ndat]=size(U);
outputs=1; % one linear output neurone
[ny,Ndat]=size(Y_all);
nmax_tod=max([NN_tod(1),NN_tod(2:1+nu)+NN_tod(2+nu:1+2*nu)-1]);
nmax_od=max([NN_od(1),NN_od(2:1+nu)+NN_od(2+nu:1+2*nu)-1]);
nmax_co2=max([NN_co2(1),NN_co2(2:1+nu)+NN_co2(2+nu:1+2*nu)-1]);
nmax=max([nmax_tod nmax_od nmax_co2]);
N=Ndat-nmax; % no. of column vectors in the regression matrix

jj=nmax+1:Ndat;
U_change=zeros(nu,N); % starch and dye
delta_Yall=zeros(ny,N); % TOD, OD and CO2

% Parameters for the tuning block (maximising the performance of the UASB reactor Model)
```

```
stch_fac_inc=1.05;
dye_fac_inc=1.05;
tune_t=zeros(1,N); % Dummy matrix for the recording of tuning instances.

% Setting of the Parameters Required for the TOD Model.
%Y_tod=Y_all(1,:); % Y_all=[1 to Ndat for tod, od and co2]
na_tod=NN_tod(1); nb_tod=NN_tod(2:1+nu); nk_tod=NN_tod(2+nu:1+2*nu);
nab_tod=na_tod+sum(nb_tod);
% Network Initialisation for the TOD Model.
H_hidden_tod=find(NetDef_tod(1,:)=='H')';
L_output_tod=find(NetDef_tod(2,:)=='L')';
[hidden_tod,inputs_tod]=size(W1opt_tod);
y1_tod=[zeros(hidden_tod,N);ones(1,N)];
Yhat_tod=zeros(1,N);
Regr_tod=[zeros(nab_tod,N);ones(1,N)];

% Setting of the Parameters Required for the OD Model.
%Y_od=Y_all(2,:); % Y_all=[1 to Ndat for tod, od and co2]
na_od=NN_od(1); nb_od=NN_od(2:1+nu); nk_od=NN_od(2+nu:1+2*nu);
nab_od=na_od+sum(nb_od);
% Network Initialisation for the OD Model.
H_hidden_od=find(NetDef_od(1,:)=='H')';
L_output_od=find(NetDef_od(2,:)=='L')';
[hidden_od,inputs_od]=size(W1opt_od);
y1_od=[zeros(hidden_od,N);ones(1,N)];
Yhat_od=zeros(1,N);
Regr_od=[zeros(nab_od,N);ones(1,N)];

% Setting of the Parameters Required for the CO2 Model.
%Y_co2=Y_all(3,:); % Y_all=[1 to Ndat for tod, od and co2]
na_co2=NN_co2(1); nb_co2=NN_co2(2:1+nu); nk_co2=NN_co2(2+nu:1+2*nu);
nab_co2=na_co2+sum(nb_co2);
% Network Initialisation for the CO2 Model.
H_hidden_co2=find(NetDef_co2(1,:)=='H')';
L_output_co2=find(NetDef_co2(2,:)=='L')';
[hidden_co2,inputs_co2]=size(W1opt_co2);
y1_co2=[zeros(hidden_co2,N);ones(1,N)];
Yhat_co2=zeros(1,N);
Regr_co2=[zeros(nab_co2,N);ones(1,N)];
for t=1:N, % Number of Control iterations = N

  % Construction of the Regression Matrix for the TOD Model.
  index_tod=0;
  for k_tod=1:na_tod,
    Regr_tod(k_tod+index_tod,:)=Y_all(1,jj-k_tod);
    % For loop to set the first 2 (na) rows of the reg. matrix.
  end
  index_tod=index_tod+na_tod;
  for kk_tod=1:nu,
    for m_tod=1:nb_tod(kk_tod),
      Regr_tod(m_tod+index_tod,:)=U(kk_tod,jj-m_tod-nk_tod(kk_tod)+1);
    end
    index_tod=index_tod+nb_tod(kk_tod);
```

```
end;
% Calculating the TOD Model's One Step Ahead Prediction.
h1_tod=W1opt_tod*Regr_tod(:,t);
y1_tod(H_hidden_tod,t)=pmntanh(h1_tod(H_hidden_tod));
h2_tod=W2opt_tod*y1_tod(:,t);
Yhat_tod(L_output_tod,t)=h2_tod(L_output_tod,:);
tod_prd=Yhat_tod(L_output_tod,t); % TOD Model's Prediction at t.
tod_mtrx(:,t)=tod_prd; % Building the TOD Models prediction matrix.
% Construction of the Regression Matrix for the OD Model.
index_od=0;
for k_od=1:na_od,
  Regr_od(k_od+index_od,:)=Y_all(2,jj-k_od);
  % For loop to set the first 2 (na) rows of the reg. matrix.
end
index_od=index_od+na_od;
for kk_od=1:nu,
  for m_od=1:nb_od(kk_od),
    Regr_od(m_od+index_od,:)=U(kk_od,jj-m_od-nk_od(kk_od)+1);
  end
  index_od=index_od+nb_od(kk_od);
end;
% Calculating the OD Model's One Step Ahead Prediction.
h1_od=W1opt_od*Regr_od(:,t);
y1_od(H_hidden_od,t)=pmntanh(h1_od(H_hidden_od));
h2_od=W2opt_od*y1_od(:,t);
Yhat_od(L_output_od,t)=h2_od(L_output_od,:);
od_prd=Yhat_od(L_output_od,t); % OD Model's Prediction at t.
od_mtrx(:,t)=od_prd;
% Construction of the Regression Matrix for the CO2 Model.
index_co2=0;
for k_co2=1:na_co2,
  Regr_co2(k_co2+index_co2,:)=Y_all(3,jj-k_co2);
  % For loop to set the first 2 (na) rows of the reg. matrix.
end
index_co2=index_co2+na_co2;
for kk_co2=1:nu,
  for m_co2=1:nb_co2(kk_co2),
    Regr_co2(m_co2+index_co2,:)=U(kk_co2,jj-m_co2-nk_co2(kk_co2)+1);
  end
  index_co2=index_co2+nb_co2(kk_co2);
end;
% Calculating the CO2 Model's One Step Ahead Prediction.
h1_co2=W1opt_co2*Regr_co2(:,t);
y1_co2(H_hidden_co2,t)=pmntanh(h1_co2(H_hidden_co2));
h2_co2=W2opt_co2*y1_co2(:,t);
Yhat_co2(L_output_co2,t)=h2_co2(L_output_co2,:);
co2_prd=Yhat_co2(L_output_co2,t); % CO2 Model's Prediction at t.
co2_mtrx(:,t)=co2_prd;

% Feeding tod_prd, od_prd and co2_prd to the controller.
[new_stch,new_dye,AA]=controller(tod_prd,od_prd,co2_prd);

% Tuning Block (Its purpose is to maximise the treatment process
```

```
% in terms of starch and dye intake loads.
if t>=10 & rem(t,10)==0,
  tod_diff=tod_prd-tod_mtrx(t-9);
  od_diff=od_prd-od_mtrx(t-9);
  co2_diff=co2_prd-co2_mtrx(t-9);
  if tod_diff<=75 & tod_prd<=1500 & od_diff<=0.3 & od_prd<=1 & co2_diff<=3 & co2_prd<=30,
    disp('everything OK'),
    disp(t),
    tune_t(1,t)=t; % marking of the instances when tuning took place.
    new_stch=new_stch*stch_fac_inc;
    new_dye=new_dye*dye_fac_inc;
  end;
end;

% Changes to the Original Input and Target Matrix as specified by the
% Neural Network Controller/tuning block.
U_change(:,t)=[new_stch;new_dye];
U(:,nmax+t)=U_change(:,t);
U_new=U;

delta_Yall(:,t)=[tod_prd;od_prd;co2_prd];
Y_all(:,nmax+t)=delta_Yall(:,t);
Y_new=Y_all;
end;
save d:\MacD1\Mod_Cont\Strateg2\Simul1b1\results.mat U_new U_change...
Y_new delta_Yall tune_t AA
```

Appendix C – Organic loading rates and COD and colour removals for the combined bio-treatment system for Experimental Phases 2 and 3

Table C.1 – Organic loading rates and COD removed by the combine bio-treatment system for Experimental Phases 2 and 3

Exp. Phase 2	**B_v (g COD l^{-1} reactor d^{-1})**	**COD removed by the UASB reactor (%)**	**COD removed by the aerobic stage (%)**	**COD removed in total (%)**
2.1	2.67	49	24	73
2.2	1.66	58	9	67
2.3	2.73	71	3	74
2.4	1.81	66	3	69
Exp. Phase 3				
Initial	2.29	62	17	79
3.1	2.22	66	14	80
3.2	2.71	52	14	66
3.3	3.73	61	27	88
3.4	3.90	60	19	79
3.5	3.14	62	15	77

Note – All the results are average values calculated after 3 anaerobic HRTs

Table C.2 – Colour removed by the combine bio-treatment system for Experimental Phases 2 and 3

Exp. Phase 2	**Colour removed by the UASB reactor (%)**	**Colour removed by the aerobic stage (%)**	**Colour removed in total (%)**
2.1	48	19	67
2.2	58	15	73
2.3	66	7	73
2.4	69	8	77
Exp. Phase 3			
Initial	58	15	73
3.1	41	15	56
3.2	38	7	45
3.3	59	18	77
3.4	56	12	68
3.5	56	11	67

Note – All the results are average values calculated after 3 anaerobic HRTs

Appendix D – Papers Published to Date

S. R. R. Esteves, S. J. Wilcox, D. L. Hawkes, C. O'Neill and F. R. Hawkes (2001) The development of a neural network based monitoring and control system for biological wastewater treatment systems. *International Journal of Condition Monitoring and Diagnostic Engineering Management* **4**(3): 22-28.

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S.R.R. Esteves, D.L. Hawkes, F.R. Hawkes, A.J. Guwy, C. O'Neill, R.M. Dinsdale and S.J. Wilcox (1998) Prediction of Remedial Actions During the Biological Degradation of Textile Effluents by Neural Networks. *Proceedings of the 11th International Congress on Condition Monitoring and Diagnostic Engineering Management*, COMADEM '98, Tasmania, Australia, December 1998.

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C. O'Neill, A. Lopez, **S. R. R. Esteves,** F. R. Hawkes, D. L. Hawkes, S. Wilcox (2000) Azo-dye degradation in an anaerobic-aerobic treatment system operating on simulated textile effluent – Short Contribution. *Applied Microbiology and Biotechnology* **53:** 249-254.

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