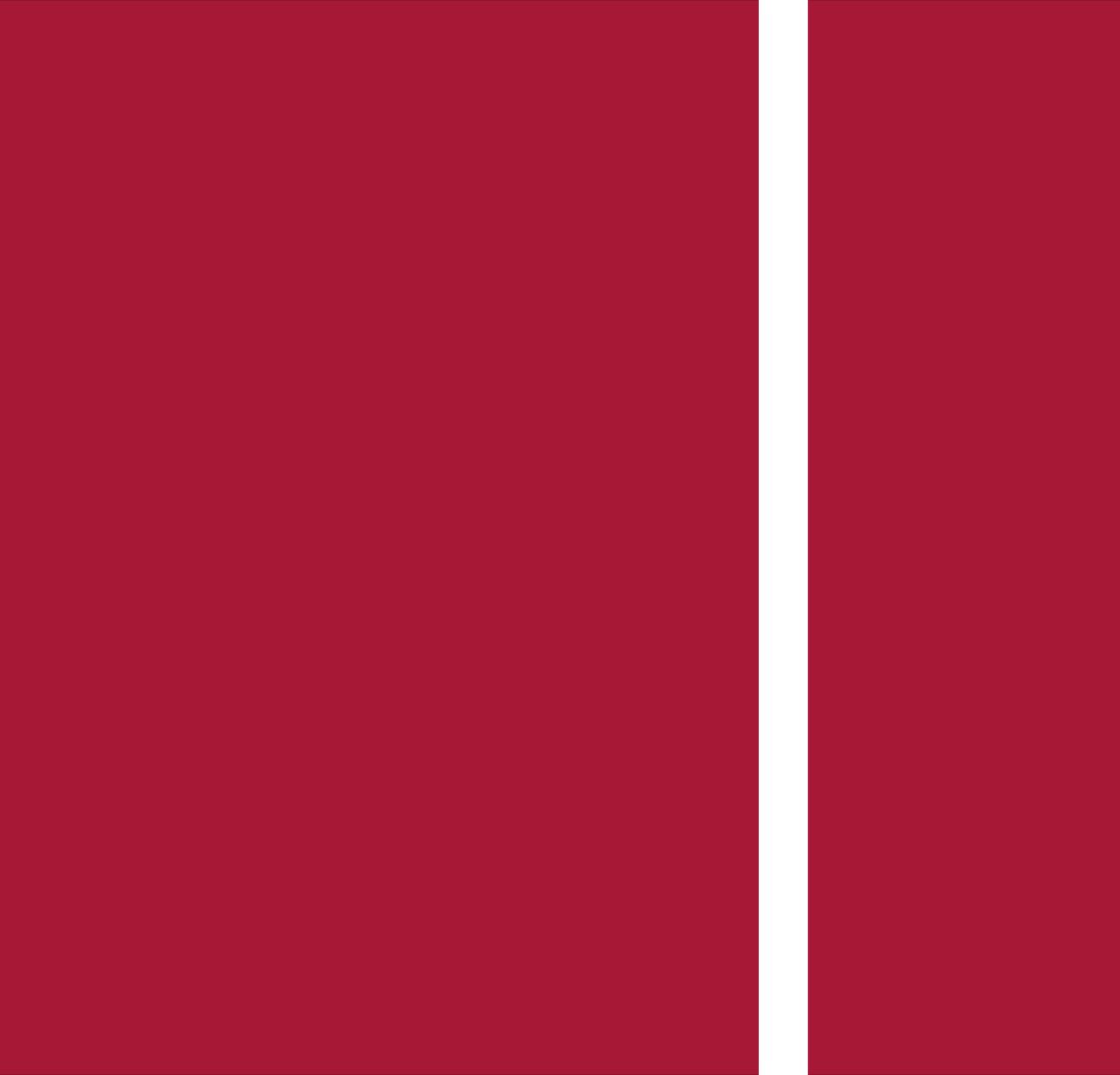


Escola de Engenharia

Merijn Amilcare Picave

Development of the Inverted Anaerobic Sludge Blanket Reactor A novel technology for the treatment of industrial wastewater containing fat





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Tese de Doutoramento Doutoramento em Engenharia Quimica e Biologica

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#### Título da tese

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Desenvolvimento do Reactor Anaeróbio de Manto de Lamas Invertido — Uma nova tecnologia para o tratamento de efluentes industriais com gordura

#### Orientadora

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Universidade do Minho, 21 de Junho de 2011



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Escola de Engenharia

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# **Summary**

Lipids are ubiquitous in industrial wastewater produced in the food industry, yet practically no biological treatment systems are available on the market that are capable of directly treating wastewater containing lipids. In general, lipids are considered a nuisance and are normally removed prior to biological treatment. Lipids are however compounds with a high calorific value and therefore highly interesting for conversion into biogas under anaerobic conditions. This thesis reports on the development of a high-rate anaerobic reactor capable of directly treating wastewater containing fat.

The reactor's technological basis for includes floatation as main sludge retention technique. Next to that, the specific contact surface between sludge and fat is maximised to prevent mass transfer limitation. Because fat degradation leads again to settlable sludge, a sedimentation step is incorporated in the form of a tilted plate separator (TPS). The settled sludge is then recycled and mixed up with raw wastewater to stimulate fat adsorption and sludge floatation. The sludge/raw wastewater mixture is subsequently injected into downers to induce reactor mixing and the break-up of floating sludge. Finally, to counteract foam formation, treated wastewater is sprayed in the head space. These considerations formed the basis of the Inverted Anaerobic Sludge Blanket (IASB) reactor.

The IASB concept was tested using several lab-scale configurations and lead to an optimised 1.2 m³ pilot scale reactor. Its robustness and capacity for treating wastewater strongly fluctuating in quality while maintaining treatment efficiency was demonstrated during a one year pilot trial on slaughterhouse wastewater. Until an organic loading rate of 12 kg COD/m³/day, no problems were encountered in terms of COD removal. The TPS showed to be efficient in retaining solids inside the reactor. The spray flow rate, however, should not go through the TPS. The IASB technology is currently being scaled-up to industrial scale taking the pilot findings into account. A modular approach was chosen as to reduce investment costs associated to the technology. The IASB technological development further resulted in a novel separation technology called Sludge Lift Separator. Separator design was optimised using computational fluid dynamics. The technology is currently being tested at pilot scale.

## Sumário

Os lipídos estão amplamente presentes em águas residuais industriais produzidas na indústria alimentar, mas praticamente nenhum sistemas de tratamento biológico existente no mercado permite o tratamento directo de águas residuais contendo teores de lípidos significativos. Em geral, os lipídios são considerados um problema e normalmente são retirados antes do tratamento biológico. Contudo, estes compostos têm um alto poder calorífico e, portanto, são altamente interessantes para a conversão em biogás em condições anaeróbias. Esta tese reporta o desenvolvimento de um reator anaeróbico de alta carga capaz de tratar diretamente efluentes com gordura.

A base tecnológica do reator inclui a flutuação como principal método de retenção das lamas. Além disso, a superfície específica de contato entre lamas e a gordura é maximizada para evitar limitação de transferência de massa. A acumulação de gordura nos flocos biológicos por mecanismos de adsorção, precipitação e oclusão provoca a flutuação da biomassa. No entanto, após criadas as condições para a degradação, a biomassa tende a sedimentar eficientemente. Assim, o reactor incorpora uma etapa de sedimentação na forma de um separador de placa inclinada (TPS). A lama sedimentada é então reciclada e misturada com o esgoto bruto e realimentada no topo do reactor para estimular a adsorção e flotação da lama. A mistura de lama e efluente bruto é injetada em downers para induzir mistura no reator e o desmembramento de lama flutuante. Finalmente, para contrariar a formação de espuma, águas residuais tratadas são pulverizadas no *head space* do reactor. Estas considerações formaram a base do reactor anaeróbio de manto de lamas invertido (IASB).

O conceito IASB foi testado usando várias configurações à escala laboratórial o que resultou num reactor optimizado à escala piloto de 1,2 m³. Posteriormente, a sua robustez e capacidade de tratamento de águas residuais, provenientes de um matadouro e altamente variáveis, foi demonstrada nesse reactor piloto durante um ano. Não se detectou nenhum problema na remoção de CQO até uma carga orgânica de 12 kg CQO/m³/dia. O TPS provou ser eficiente na retenção de sólidos dentro do reator, no entanto, o fluxo de spray não deve passar pelo TPS. Na sequencia dos resultados obtidos à escala piloto a tecnologia IASB está atualmente em fase de *scale-up*. De forma a reduzir os custos de investimento associados à tecnologia escolheu-se uma abordagem modular. Uma nova tecnologia de separação de sólidos denominada "*Sludge Lift Separator*" resultou também do desenvolvimento da tecnologia IASB. O design do separador foi otimizado usando dinâmica de fluidos computacional. Esta tecnologia está atualmente em fase de teste à escala piloto.

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## List of symbols & abbreviations

ABR - Anaerobic Baffled Reactor

AD - Anaerobic Digestion

AF - Anaerobic Filter

AFFR - Anaerobic Fixed Film Reactor

Anammox - Anaerobic Ammonium Oxidation

AnMBR – Anaerobic Membrane Bioreactor

ASBR – Anaerobic Sequencing Batch Reactor

 $A_{TPS}$  – Total TPS plate surface area (m<sup>2</sup>)

BAT – Barroso e Alto Tâmega

BFBR - Buoyant Filter Bioreactor

BREF – Reference documento on best available techniques

 $Ca_3(PO_4)_2$  – Calcium phosphate

CANON - Completely Autotrophic Nitrogen removal Over Nitrite

CFD - Computational Fluid Dynamics

 $CH_{1.8}O_{0.8}N_{0.2}P_{0.05}$  – Mean biomass composition

-CH<sub>2</sub>- - Methyl group

CH<sub>4</sub> - Methane

CO<sub>2</sub> – Carbon dioxide

COD - Chemical Oxygen Demand

COOH - Carboxylic group

CSM – Centrale Suiker Maatschappij

CSTR - Continuously Stirred tank reactor

d – TPS plate distance (m)

DAEB - Downflow Anaerobic Expanded Bed

DAF – Dissolved Air Floatation

DCPD - Dicyclopentadiene

EGSB – Expanded Granular Sludge Blanket

FePO<sub>4</sub> – Ferric phosphate

FOG - Fat, Oil & Grease

Fr – Froude number (-)

GRABBR - Granular Bed Anaerobic Baffled Reactor

GWE - Global Water Engineering

HAc - Acetic acid

HRT – Hydraulic Retention Time (h)

HS - Hydrogen sulphide

IASB – Inverted Anaerobic Sludge Blanket

i-But - iso-butyric acid

IC - Internal Circulation

IFB - Inverse Fluidised Bed

ITB - Inverse Turbulent Bed

L – TPS plate length (m)

LCFA – Long Chain Fatty Acid

LEB - Laboratory for Environmental Biotechnology

MABR - Modified Anaerobic Baffled Reactor

meq – Milli-equivalent

MgNH<sub>4</sub>PO<sub>4</sub> – Magnesium ammonium phosphate

N - nitrogen

n-But - n-butyric acid

NH<sub>4</sub><sup>+</sup> - Ammonium

n-Val -n-valeric acid

OLR – Organic Loading Rate (kg COD/m³/day)

P - Phosphorus

 ${\sf P\&ID-Piping~\&~Instrumentation~Diagram}$ 

PBR - Packed Bed Reactor

PE - Polyethylene

PFD - Process Flow Diagram

PLC – Programmable Logic Controller

POME - Palm Oil Mill Effluent

PP - Polypropylene

Prop - Propionic acid

PVC – Polyvinyledene chloride

 $Q_f$  – Fluid flow rate through TPS (m<sup>3</sup>/h)

RC – Reactor Compartment

Re - Reynolds number (-)

SD – Standard Deviation

SLS - Sludge Lift Separator

SO<sub>4</sub><sup>2</sup> - Sulphate

SRT – Solids Retention Time (h)

SVI - Sludge Volume Index (mL/L)

TCS – Tilted Conical Separator

 $t_{\text{et}}$  – Time to effluent tank (h)

t<sub>m</sub> – Mixing time (h)

TPS – Tilted Plate Separator

t<sub>s</sub> – Settling time (h)

TSS – Total Suspended Solids

 $t_{TPS}$  – TPS residence time (h)

u – Fluid velocity between TPS plates (m/h)

UAF – Upflow Anaerobic Filter

UASB - Upflow Anaerobic Sludge Blanket

```
UASFF - Upflow Anaerobic Sludge Fixed Film
u<sub>s</sub> – Particle settling velocity (m/h)
u<sub>sx</sub> – Particle settling velocity parallel to the TPS plate (m/h)
u_{\text{sy}} - Particle settling velocity perpendicular to the TPS plate (m/h)
UV – Ultraviolet
VFA - Volatile Fatty Acid
VLCFA – Very Long Chain Fatty Acid
V<sub>R</sub> – Reactor volume (m<sup>3</sup>)
V<sub>RC</sub> - Volume of the reaction compartment (m<sup>3</sup>)
VSS – Volatile Suspended Solids
W – TPS plate width (m)
wt. - weight
WWTP - Wastewater Treatment Plant
\alpha – TPS plate angle with horizontal (^{\circ})
\Phi_{\text{feed}} - Feed flow rate (m<sup>3</sup>/h)
\Phi_{spray} - Spray flow rate (m<sup>3</sup>/h)
```

Φ<sub>underflow</sub> - Reactor underflow flow rate (m<sup>3</sup>/h)

## 1 Introduction

More and more people are becoming aware of the need for a sustainable society. Using renewable products and controlling pollution are ways to achieve sustainability. Both are combined when wastewater is biologically treated in the absence of oxygen: through the action of anaerobic bacteria organic pollutants present in wastewater are effectively converted into biogas, a gas that mainly consists of methane (CH<sub>4</sub>) and carbon dioxide (CO<sub>2</sub>) and is a renewable energy source. In this way negative effects associated to industrialisation can be minimised (Rajeshwari et al., 2000; strangely enough the exact same review was published in 2004 by Saleh & Mahmood). The chemical energy stored in methane can be converted into other more useful forms of energy, such as electric energy or simply heat. Worldwide anaerobic technology is being successfully applied to treat effluents with high organic loading rates from *e.g.* breweries and the paper industry using compact wastewater treatment installations. An example of a widely applied anaerobic technology is the upflow anaerobic sludge blanket reactor or UASB reactor, of which more than 1100 have been installed worldwide (Van Lier, 2008).

A major group of organic pollutants which has shown very difficult to be converted into biogas are the lipids. Because of this, they are usually removed from wastewater prior to anaerobic treatment (Omil et al., 2003) using technologies that require energy instead of producing energy, dissolved air floatation (DAF) for example. Moreover, one could state that lipids are organic compounds that should be converted to biogas. For the specific case of slaughterhouse wastewater, Massé & Masse (2000b) actually state that it is particularly well suited for anaerobic treatment because of the high concentrations of organic pollutants and particularly fat. In this chapter, this will be explained. Furthermore, conventional anaerobic reactor technologies and their problems with lipids will be discussed. Finally, from this analysis and the identification of process problems the potential foundations for anaerobic reactor technology that is capable of efficiently treating industrial wastewater containing lipids will be forged.

## 1.1 Anaerobic degradation

Anaerobic degradation or digestion (AD) of complex organic compounds occurs in the absence of oxygen through several parallel and sequential conversion steps, all catalysed by several groups of micro-organisms. The main conversion steps are hydrolysis, acidogenesis, acetogenesis and methanogenesis. Figure 1.1 summarises the process of anaerobic digestion of organic matter resulting in the production of methane and carbon dioxide. Sousa (2006) gives a thorough description of these four steps and it is beyond the scope of this thesis to give an in-depth explanation. All four steps will be briefly addressed below, however.

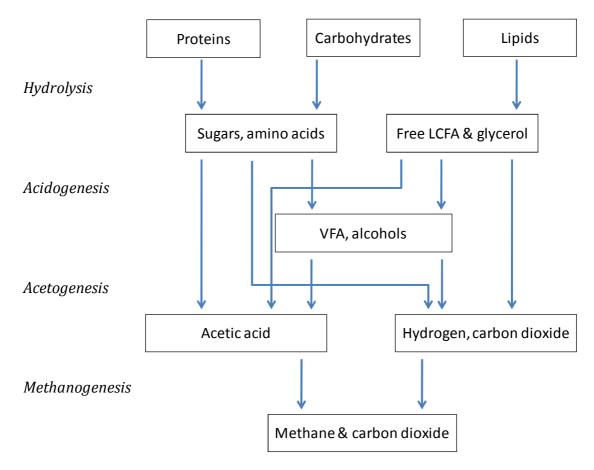


Figure 1.1. Simplified representation of the anaerobic digestion process (Adapted from Pereira, 2003)

Through the process of hydrolysis complex organic compounds are converted into their constituents, thus making them better available for further degradation. In this way starch, for example, is converted into its monomeric carbohydrate building blocks, while proteins are converted into amino acids. Lipids are converted into glycerol and free long chain fatty acids (LCFA).

Acidogenesis is an oxidative process during which the more complex monomers resulting from hydrolysis are converted into organic acids and alcohols. This is followed by acetogenesis which results in the formation of acetic acid. Acetic acid is in its turn is used by methanogenic micro-organisms for methane production. The main final products of complete anaerobic degradation of organic compounds are methane ( $CH_4$ ), carbon dioxide ( $CO_2$ ) and anaerobic biomass. Depending on the composition of the organic compounds being degraded and the presence of other compounds, other products are reduced inorganic compounds such as ammonium ( $NH_4^+$ ) and hydrogen sulphide ( $HS^-$ ). If these inorganic compounds need to be removed, other techniques than anaerobic technology should be applied.

In industrial practice, several tools are available to provide optimised conditions and properly control the four steps of anaerobic digestion. Main parameters normally monitored for proper control are temperature, pH, alkalinity, chemical oxygen demand (COD), volatile fatty acid (VFA) concentration, N and P. Which ones are actively controlled depends on the kind of wastewater or organic slurry that is being anaerobically treated and the processing risks one is willing to take. After all, more process control generally means a bigger capital investment and potentially higher operational costs.

Lipids are the kind of complex organic compounds that have to go through all four anaerobic degradation steps. Yet already during the first step of hydrolysis they may cause irreversible damage to industrial anaerobic treatment systems due to inhibition and induction of floatation. This being the case, there is a strong preference to reduce the risk of process upsets and remove lipids before anaerobic treatment. A question one could ask is why to even consider not removing lipids before anaerobic treatment. The answer is given below.

## 1.2 Why convert lipids to biogas

When looking at everyday life, it is obvious that lipids are ubiquitous. They form an essential part of life and in the form of oil & fat they serve for energy storage. Yet somehow, in many situations lipids, and specifically oils and fats, are considered a nuisance, be it because of aesthetic reasons or because they stick to things and are hard to remove. Thus, one could state that there is a love/hate relationship towards oils & fat and this is exactly the same for anaerobic treatment of wastewater. If this is the case, then why even consider converting oil & fat present in wastewater into biogas?

Oils & fat are tri-esters of glycerol and long chain fatty acids (LCFA). As previously indicated, they are readily converted into their constituents through the process of hydrolysis making them more susceptible for further degradation. Having three hydroxyl groups, glycerol is easily degraded under anaerobic or aerobic circumstances. Thus remain the LCFA and these are a bit tougher to degrade, due to their chemical and physical properties. Therefore, they are considered a nuisance in biological treatment systems and are normally removed prior to biological treatment (Hwu et al., 1996).

LCFA are aliphatic chains of mainly methyl groups (-CH<sub>2</sub>-) with a carboxylic group (-COOH) at one end of the chain. As such, they have a hydrophobic tail connected to a hydrophilic head. The length of the chain may vary and the longer the chain, the more hydrophobic the LCFA. Furthermore, the chains may by either saturated (no double bonds in the aliphatic chain), unsaturated (one double bond) or poly-unsaturated. Fats are composed of saturated LCFA, while oils are normally composed of (poly-) unsaturated LCFA. Table 1.1 shows several of the most commonly encountered LCFA with their trivial and systematic name, their length and the number of double bonds (CRC Handbook of Chemistry & Physics, 85<sup>th</sup> Edition).

Table 1.1 LCFA nomenclature, chain length and double bonds (CRC Handbook of Chemistry & Physics, 85<sup>th</sup> Edition)

Trivial name	Systematic name	Chain length	Nr. of double bonds
Lauric acid	Dodecanoic acid	12	0
Myristic acid	Tetradecanoic acid	14	0
Palimitic acid	Hexadecanoic acid	16	0
Palmitoleic acid	Cis-9-hexadecenoic acid	16	1
Oleic acid	Cis-9-octadecenoic acid	18	1
Linoleic acid	Cis,cis-9,12-octadecadienoic acid	18	2
Stearic acid	Octadecanoic acid	18	0

Within the LCFA chains a lot of chemical energy can be stored. Sousa (2006) reported a mean biogas production from three different classes of substrates commonly encountered in industrial wastewater: lipids, proteins and carbohydrates. The results are reproduced in Table 1.2. and these show why LCFA conversion into biogas is so interesting. Not only can more biogas be produced from lipids than from carbohydrates and protein, the biogas quality is also superior to the biogas produced from the other two substrate groups.

Table 1.2 Potential biogas production in different classes of substrates (Sousa, 2006)

Component	Methanogenic reaction	Biogas	CH4
		(L/g)	%
Lipids	$C_{15}H_{90}O_6 + 24.5H_2O \rightarrow 3 4.75CH_4 + 15.25CO_2$	1.425	69.5
Carbohydrates	$C_{16}H_{10}O_5 + H_2O \rightarrow 3CH_4 + 3CO_2$	0.830	50.5
Proteins	$C_{11}H_{24}O_5N_4+14.5H_2O \rightarrow 8.25CH_4+3.75CO_2+4NH_4^++4HCO_3^-$	0.921	68.8

Besides the fact that a high biomethane yield can be obtained on LCFA, direct conversion of LCFA to biogas has other advantages. First of all, separation of fat & oil before anaerobic treatment can be avoided, thus eliminating one wastewater treatment step. Additionally, by omitting a lipids separation step prior to anaerobic treatment, fatty sludge production is avoided as well. In this way, direct treatment of lipids may cut investment and operational costs.

As briefly indicated before, lipids are everywhere and also have a strong presence in industrial wastewaters. This makes direct lipid conversion into biogas quite interesting: the stored chemical energy becomes directly available as energy source for the productive process. This can be considered a good thing, since a big part of the industries with fat present in their wastewater is also an intensive energy user. Therefore, reducing energy costs and dependency on outside energy sources by producing biogas from organic pollutants present in the wastewater you produce might be an interesting consideration. Direct production of biogas from fat present in wastewater only makes this consideration more appealing. Besides this, the production and use of renewable energy sources linked to wastewater treatment improves the public image of any company. Nestlé, for example, has an excel sheet available on their website showing all their environmental performance indicators (Nestlé website). This includes, amongst others, their actual energy consumption, renewable energy consumption and water quantity and quality discharged. The direct production of biogas from lipidic compounds present in wastewater and use of the biogas in the productive process would have a direct influence on the four indicators previously mentioned.

The food processing industry, of which Nestlé currently is the biggest representative, is probably the most interesting industrial sector for direct anaerobic treatment of wastewater

containing fat. Most are energy intensive due to hygienic requirements (pasteurisation or sterilisation) or heat treatment of the food (baking, cooking, distillation, etc.); and most of them have to keep an appealing public image of environmental friendliness and sustainability.

Food processing includes the dairy industry, meat & fish processing industry, edible oil production, bakery products, etc.. All of them generate complex wastewaters with high organic loads and most have relatively high concentrations of lipids present in their wastewater. Tables 1.3, 1.4 and 1.5 list a selection of wastewater compositions reported in the scientific literature. Table 1.3 shows the quality of wastewater produced within the dairy industry. Table 1.4 lists wastewater quality for meat processing, such as slaughterhouses & meat rendering facilities, and fish processing industry. Table 1.5 includes data of wastewater generated by other selected industries where there is a strong presence of lipidic compounds, and has been included for comparative reasons only.

 Table 1.3 COD, volatile suspended solids (VSS) and fat, oil & grease (FOG) concentrations of dairy wastewater.

Type of industry	COD	TSS	VSS	FOG	Reference
	(g/L)	(g/L)	(g/L)	(g/L)	
Cheese processing	2.45	0.59	0.42	nd	Danalewich et al. (1998)
	7.62	1.53	1.48	nd	
	2.03	0.39	0.23	nd	
	2.31	0.70	0.57	nd	
	3.56	0.73	0.66	nd	
	0.79	0.47	0.31	nd	
	2.91	1.91	1.01	nd	
	2.29	3.56	1.94	nd	
	4.90	0.89	0.68	nd	
	1.64	0.37	0.33	nd	
	3.09	0.76	0.70	nd	
	2.82	0.85	0.77	nd	
	3.23	0.92	0.89	nd	
	1.57	0.33	0.28	nd	
Milk processing	10.0-12.0	ND	ND	0.2-1.2	Cammarota et al. (2001)
Dairy laboratory	10.5	ND	ND	1.8	Omil et al. (2003)
Dairy	18.05	7.18	6.51	4.89	Arbeli et al. (2006)
Whey production	68.6	1.35	ND	9.4	Saddoud et al. (2007)
Whey production	60	1.5	ND	0.9 % <sup>1)</sup>	Gannoun et al. (2008)
Mixed dairy products <sup>2)</sup>	4.44	1.07	ND	0.57	Janczukowicz et al. (2008)

Fat expressed as a percentage. It is not indicated what the basis is.
 Analysis of samples taken at the pumping station.

**Table 1.4** COD, volatile suspended solids (VSS) and fat, oil & grease (FOG) concentrations of meat & fish processing wastewater.

Type of wastewater	COD	TSS	VSS	FOG	Reference
	(g/L)	(g/L)	(g/L)	(mg/L)	
Slaughtering (cattle) <sup>1)</sup>	6.7	1.9	1.6	1.2	Martinez et al
					(1995)
	21.0	12.0	10.0	1.7	
Slaughtering	7.54	1.21	1.1 <sup>2)</sup>	0.42	Ruiz et al. (1997)
Slaughtering (cattle/hog)	10.41	ND	ND	ND	Borja et al. (1998)
Slaughtering	1.4-4.2	0.37-0.84	ND	0.05-	Nuñez & Martinez
				0.28	(1999)
Slaughtering (hog)	2.94	0.96	0.77	ND	Massé & Masse
					(2000a)
	3.59	0.74	0.58	ND	
	4.98	1.35	1.19	ND	
	2.33	0.88	0.59	ND	
	8.63	2.10	1.89	ND	
	3.42	1.43	1.15	ND	
Slaughtering (hog)	6.9-11.5	2.1-2.9	1.9-2.5	ND	Massé & Masse
					(2000b)
Slaughtering (sheep/goat)	1.10-7.25	0.3-2.3	ND	0.13-	Manjunath et al.
				0.40	(2000)
Slaughtering (hog )	7.5	1.71	1.52	0.44	Massé et al (2001)
	12.6	7.00	4.9		
Slaughtering (poultry)	5.25 <sup>3)</sup>	1.1	ND	0.57	Del Nery et al.
					(2001)
Slaughtering (hog) <sup>4)</sup>	3.23	ND	ND	0.16	Massé et al (2002)
Meat processing	4.5	3.6	1.9	0.5	Reimann et al.
					(2002)
Slaughtering (cattle/hog)	2.0-6.2	0.85-6.3	0.66-5.3	0.04-	Caixeta et al. (2002)
				0.60	
Slaughtering	6.04	ND	ND	ND	Torkian et al. (2003)
Pet food/meat rendering	96.7	36.8	34.4	38.8	Nakhla et al. (2003)
Slaughtering (poultry)	5.8-20.2	2.4-4.7	ND	ND	Fuchs et al. (2003)
Slaughtering (sheep/cattle)	7.23	0.91	0.85	0.25	Del Pozo et al.
					(2003)
Slaughtering (poultry)	1.13	0.44	ND	ND	Del Pozo et al.
					(2005)
	2.03	0.43	ND	ND	

Type of wastewater	COD	TSS	VSS	FOG	Reference
	(g/L)	(g/L)	(g/L)	(mg/L)	
Slaughtering (poultry)	1.82	0.43	ND	0.17	Del Pozo & Diez
					(2005)
Slaughtering (hog)	7.08	1.59	1.42	0.17	Masse & Massé
					(2005)
Slaughtering/meat packing	1.4-3.6	ND	ND	0.45-	Miranda et al.
(cattle/hog) <sup>5)</sup>				0.96	(2005)
Slaughtering (cattle)	3.44	1.20	1.02	0.11	Mijaylova Nacheva
					et al. (2011)
Meat rendering	85.7	44.0	28.2	35.8	Jeganathan et al.
					(2006)
	54.9	39.2	20.2	22.5	
	59.3	38.2	21.1	15.1	
Meat rendering	55	39	20	23	Jeganathan et al.
					(2007)
Slaughtering (poultry)	4.12	0.79	ND	0.39	Del Nery et al.
					(2008)
Slaughtering	10.4	ND	4.2	1.3	Maya-Altamira et
					al. (2008)
Fish meal	4.2	ND	0.4	1.0	
Tuna processing <sup>6)</sup>	5.55	1.58	ND	1.45	Achour et al. (2000)

Red stream (bleeding) and green stream (pen cleaning & tripe processing), respectively. Calculated.
Before pre-treatment with screens & DAF.
Filtered slaughterhouse wastewater.
Composition after DAF pre-treatment
Effluent blend.

Table 1.5 COD, volatile suspended solids (VSS) and fat, oil & grease (FOG) concentrations of selected sources of industrial wastewater.

Type of wastewater	COD	TSS	VSS	FOG	Reference
	(g/L)	(g/L)	(g/L)	(mg/L)	
Olive oil	90.0	23.6	23	8.6	Beccari et al. (1996)
Palm oil	16.0	ND	ND	0.41	Faisal & Uno (2001)
Bakery products	8.7-20.7	1.4-8.4	1.3-7.1	0.85-3.5	Guven (2001)
Sunflower oil	5.6-15.3	4.3-12.8	3.7-9.68	0.54-	Saatci et al. (2003)
				7.64	
Palm oil	44.3	19.78	ND	4.85	Zinatizadeh et al.
					(2006)
Olive oil	130	36	24	ND	Fezzani & Cheikh
					(2007)
Palm oil	70.9	25.8	ND	ND	Wu et al. (2007)
Cutting oil	1.0-4.0	0.2-2.7	0.2-1.9	ND	Perez et al. (2007)

Type of wastewater	COD	TSS	VSS	FOG	Reference
	(g/L)	(g/L)	(g/L)	(mg/L)	
Olive oil	97.6	ND	ND	0.77	Azbar et al. (2008)
Olive oil <sup>1)</sup>	120-130	0.1	ND	0.03 -	Kapellakis et al.
				1.00	(2008)
	60-180	0.9	ND	0.5 – 2.3	
Vegetable fats & oils	11.3	ND	3.8	0.0	Maya-Altamira et
					al. (2008)
Palm oil	20.7-73.5	7.1-24.5	ND	2.5-16.1	Poh & Chong (2010)

1) For olive mill wastewater from pressure & centrifuge processing, respectively. TSS concentration in %, no basis indicated.

As can be seen, there is a strong variability in quality. When analysing the potential for anaerobic wastewater treatment within an industrial sector, it is convenient to define once more why anaerobic treatment might be interesting. It is not only the quantity of wastewater, but the combination of quantity and concentration that will determine economic viability. Tables 1.3 through 1.5 show that in spite of the strong variability in COD, TSS, VSS and FOG (fat, oil & grease) concentrations, COD concentrations are mostly not lower than 2 g/L, which means that most wastewater generated in dairy and meat processing industries would be suitable. Therefore, it is preferable to speak of the organic load that an industry produces. If this is known and high enough, one can be practically certain that anaerobic treatment technology is viable. Due to their size and due to the work already done on anaerobic LCFA removal from synthetic dairy wastewater within our lab (Pereira, 2003), a more in-depth analysis is given of the dairy and meat industry.

Things the dairy and meat processing industries have in common are that they are distributed all over the world and not too prone to seasonal influences as are *e.g.* vegetable or fruit processing industries. In spite of this, care must be taken. Danalewich et al. (1998) did a survey on wastewater production by the dairy industry in the US and stated that there are seasonal fluctuations; production being higher in summer time than in winter time. Furthermore, there are considerable fluctuations during daily operation. This, however, is easily resolved using proper equalisation.

Besides the obvious differences in raw materials & products, the first differences between the dairy and meat processing industry can already be noticed when looking at the size of dairy and meat operations. In recent years, due to economy of scale, there has been a tendency for the dairy industry to centralise their operations, specifically in the industrialised countries (Omil et al., 2003). In the European Union, for example, the number of dairy companies

gradually reduced from 13,155 to 12,945 in the period between 2004 and 2007 (Eurostat). Demirel et al. (2005) stated that the dairy industry is one of the biggest producers of wastewater and indicated for a typical European dairy operation a flow of 500 m³/day. The result is bigger, more capital intensive processing plants that normally operate 24/7. Consequently, wastewater normally is continuously produced and there is a continuous energy demand. Anaerobic treatment, which generally requires a bigger investment than *e.g.* aerobic treatment, should therefore be considered an interesting solution for dairy processing plants. Besides this, Saddoud et al. (2007) indicated that for the specific case of why wastewater aerobic processes are simply inappropriate due to the high strength of the wastewater. In general, wastewater generated in the dairy industry is strong and characterised by high BOD and COD concentrations Demirel et al. (2005). Furthermore, it normally is quite warm, thus making such wastewaters ideal for anaerobic treatment (Demirel et al., 2005). As potentially problematic for conventional anaerobic treatment are indicated the presence of lipids and high suspended solids concentrations (Demirel et al., 2005).

Many studies have been done on dairy effluent characterisation and treatment. These range from wastewater from normal milk industry to cheese & whey industry (Danalewich et al., 1998; Gannoun et al., 2008), all showing a strong variety in composition and COD concentration (Table 1.3). Janczukowicz et al. (2008) carried out an analysis of wastewater originating from various sections within the same dairy factory producing butter & cheese. COD ranged from 2,543 up to 73,445, depending on where the sample was collected. As sources of wastewater generation Rajeshwari et al. (2000) indicated spilled milk, spoiled milk, skimmed milk, whey and wash water. Although wastewater composition varies strongly, Omil et al. (2003) stated that the source of pollution is predominantly due to milk losses during processing and can reach up to 4 % of the incoming milk. The variability of wastewater composition is only logical, since wastewater is mainly generated from cleaning operations (Ramasamy et al., 2004; Demirel et al., 2005). Variability is further increased by the variety of products normally produced in dairy factories. Ramasamy et al (2004) estimated that wastewater produced is about 2.5 times the quantity of milk processed.

As Table 1.4 shows, the meat processing industry generates probably one the most polluting organic wastewaters, containing a variety of organic compounds originating, amongst other sources, from fat, meat, blood, gut content, excrements and skin (Rajeshwari et al., 2000), and with COD concentrations reaching as high as 96,660 mg/L for the specific case of pet food production (Nakhla et al., 2003; Jeganathan et al., 2006 & 2007). The kind of operation

determines the complexity and strength of the wastewater. Massé & Masse (2000a) defined 4 determining factors for wastewater quality:

- 1. Blood capture;
- 2. Water use. More efficient water use results in stronger wastewater, while total loads stay the same;
- 3. Type of animal slaughtered;
- 4. Type of industry.

Efficient blood retention is stated to be the main means to reduce the wastewater BOD load (Massé & Masse, 2000a). This is corroborated by Del Pozo et al. (2003). They stated that a very high COD concentration was found due to inefficient blood recovery. Ruiz et al. (1997) showed through analysis of different wastewater sources within a same slaughterhouse that blood was the major contributor in terms of organic load. Common ways of disposing of blood are by transfer to wastewater treatment plants, rendering, composting and, surprisingly, land application (Mittal, 2006). The same author lists the reasons why land application should not be an option: odour production, groundwater pollution, soil contamination, health hazard an d greenhouse gas emissions.

Nuñez & Martinez (1999) and Caixeta et al. (2002) stated that 40 to 60% of degradable matter present in meat processing wastewater is present in either colloidal or suspended form. Although solids considerably contribute to the wastewater strength (Table 1.4), in most cases it does not seem to reach this level. Although the type of animal slaughtered might have influence on overall wastewater composition, it is not apparent from a brief analysis of Table 1.4. According to Massé & Masse (2000a) slaughtering alone results in stronger wastewater than when other activities are included as well. Again, this is not confirmed by literature analysis; in fact it shows that meat rendering produces by far the strongest wastewater.

Specifically when looking at meat products, the meat industry is more diverse in size than the dairy industry. In the European Union alone there were 30,771 companies involved in this activity in 2006 (Eurostat), while there were only 13036 dairy companies. If slaughterhouses are taken into account as well, the number goes up to 44,000 enterprises. Thus it is probable that there is a big number of smaller sized meat processing companies that barely produces a high enough organic load to compensate for an investment in an anaerobic wastewater treatment plant. Nevertheless, there are enough big slaughterhouses in Europe

that have enough size for anaerobic treatment to be interesting. Furthermore, Del Pozo et al. (2003) indicated that anaerobic treatment should be considered as an efficient and cost-effective pre-treatment before aerobic treatment, since it considerably reduces the organic load. In fact, Torkian et al. (2003) considered aerobic treatment only not to be suitable for slaughterhouse wastewater treatment due to the high energy requirements for aeration, limitations in oxygen transfer and the large quantities of sludge produced.

South America appears to be an interesting market for anaerobic treatment of slaughterhouse wastewater and this is only enhanced by the possibility of applying Clean Development Mechanisms in which carbon credits can be earned. Specifically in Brazil and Argentina, slaughterhouses are big and for these operations investment in an anaerobic treatment system for energy recovery from wastewater might be an economically attractive option. Del Nery et al. (2008) indicated that the poultry industry in Brazil makes the most use of anaerobic technology for wastewater treatment. Brazil was the world's 3<sup>rd</sup> largest producer in 2005 with 9.4 million tons (Del Nery et al., 2008). This almost equalled the production of the EU in the same year (Eurostat) and already shows that there is strong potential for anaerobic treatment in Brazil for slaughterhouse wastewater. Martínez *et al.* (1995) reported that 1.4 million head of cattle were slaughtered annually in Uruguay alone.

## 1.3 Anaerobic reactor technology

When the right process conditions are applied, anaerobic treatment technology is ideal for treating wastewater or slurries with high concentrations of organic compounds. It combines the efficient conversion of organic compounds into biogas with low sludge production, since anaerobic micro-organisms are slow growers compared to aerobic ones. Anaerobic treatment technology can be divided into high-rate and low-rate treatment systems. With these systems, a feedstock range spanning from animal waste to municipal sludge, industrial wastewater and the organic fraction of municipal solid waste, becomes available for anaerobic treatment and biogas production.

The low-rate anaerobic treatment systems are normally applied for biogas production from organic slurries with high organic solids concentrations (manure, wasted activated sludge, food waste, etc.). They are either completely mixed or plug-flow systems without any solids retention: solids degradation is achieved by applying long hydraulic retention times (HRT) on average ranging from 12 (Gregersen, 1999) to as much as 50 days (Bieleveld Bioenergie,

personal communication), depending on substrate complexity and operation conditions. Furthermore, the application of long HRTs reduces the need for active process control and therefore investment costs. In practice it is quite normal for only the process temperature to be continuously monitored and controlled. The main goal of these low-rate systems is the production of biogas from organic solids present in the slurries. AD can only be considered a partial treatment, since it does not effectively remove compounds such as ammonium, phosphate or sulphide. The liquid effluent is a stabilised slurry called digestate and still contains most of the nutrients that entered the system. Due to the long hydraulic retention times applied and the stabilizing effect of anaerobic treatment on organic slurries, however, most biogas plants also have a function as agricultural nutrient redistribution system, since the digestate leaving the system can be directly used as fertilizer (Gregersen, 1999; Birkmose, 2007). This is done for example in Denmark, Germany and the Netherlands.

Because of environmental problems with land application of specifically pig slurry, there is a tendency to come to complete treatment systems including nitrogen and phosphorus removal (reference). They practically always include anaerobic digestion of the organic compounds. They differ in how the nutrients are removed. For nitrogen removal, for example, nitrification/denitrification as well as partial nitrification/Anammox are applied. Phosphate is commonly precipitated using ferric chloride, calcium hydroxide or magnesium hydroxide, which results in phosphate precipitation in the form of ferric phosphate (FePO<sub>4</sub>), calcium phosphate (Ca<sub>3</sub>(PO<sub>4</sub>)<sub>2</sub>) or magnesium ammonium phosphate (MgNH<sub>4</sub>PO<sub>4</sub>), respectively. Although these systems have already been shown to be technically feasible, they are not always economically viable, since they strongly depend on revenues from electricity generation using the biogas produced. There is a big difference in price paid per kWh of electricity produced in *e.g.* Germany or Portugal. The result is that Germany has experienced a booming market for AD of organic waste, while Portugal lags behind.

Lipid waste slurries are already fed to existing digesters to boost biogas production. However, it is known that care must be taken when doing so, since inhibition may occur and be persistent (Neves et al., 2009).

From a technological point of view, a more interesting case to look at is high-rate anaerobic wastewater treatment technology. From the beginning of the eighties, it has become a standard for a certain range of industrial wastewater treatment. More than two thousand full-scale installations were installed worldwide until 2007, treating mainly wastewater containing

readily degradable organic pollutants such as volatile fatty acids and carbohydrates. Table 1.6 reproduces results as reported by (Van Lier 2008) and clearly shows that anaerobic treatment has found its main application in the food processing & beverages industry. Therefore, it should not be surprising that the European Commission recognises anaerobic wastewater treatment as the best available technique for concentrated industrial wastewater in the most recently published BREF (reference documents on best available techniques) for food, drink & milk industries (BREF, 2006). This confirms its acceptance as a conventional treatment technology.

Table 1.6 Registered anaerobic reactors installed for industrial wastewater treatment (Van Lier 2008).

Industrial sector	Type of wastewater	Number of	%
		anaerobic reactors	
Agro-food industry	Sugar, potato, starch, yeast, pectin, citric acid, cannery,	816	36
	confectionery, fruit, vegetables, dairy, bakery		
Beverage	Beer, malting, soft drinks, wine, fruit juices, coffee	657	29
Alcohol distillery	Can juice, cane molasses, beet molasses, grape wine,	227	10
	grain, fruit		
Pulp & paper industry	Recycle paper, mechanical pulp, NSSC, sulphite pulp,	249	11
	straw, bagasse		
Miscellaneous	Chemical, pharmaceutical, sludge liquor, landfill	317	14
	leachate, acid mine water, municipal sewage		

The main difference between high-rate and low-rate treatment is that in the case of high-rate treatment the solids residence time (SRT) is separated from the HRT. As such, the anaerobic sludge, and therefore the biological activity, is retained inside the reactor. By separating the two, the HRT can be significantly reduced without consequences for the biological activity. Depending on the specific reactor design and separation technique applied, HRTs as low as 4 hours can be achieved (personal experience). Consequently, the reactor volume can be reduced: where for low-rate technologies reactor volumes of 3,000 m<sup>3</sup> are quite normal, for high-rate technologies reactor volumes do not normally surpass the 1,000 m<sup>3</sup> level.

To achieve the separation of SRT and HRT, different methods are available. It is on this principle that virtually all patented technologies concerning high-rate anaerobic treatment of wastewater are based. The difference between the technologies is the separation method applied to keep biomass inside the reactor. Table 1.7 lists a selection of the technologies

available to separate the SRT from the HRT. These have either been tested at lab scale or are already being applied at full-scale.

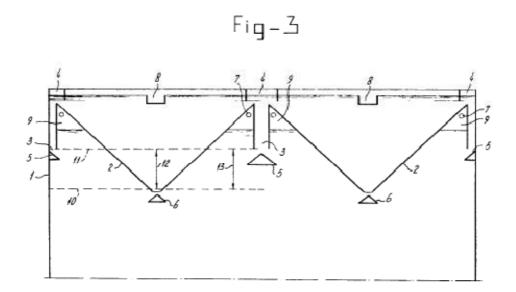
Table 1.7 Solids retention technologies applied in anaerobic wastewater treatment systems.

Technology	Separation principle	Industrial scale application
Three-phase hood	Sedimentation	UASB; IC® (Paques); Biobed® (Biothane);
separator		Biotim (Waterleau Group)
Fixed film	Fixation	Proveo (Naskeo)
Fluidised bed	Fixation/sedimentation	Anaflux (Ondeo IS)
	or floatation	
Anaerobic baffled reactor	Sedimentation	No applications found
Membrane separation	Size	ADI-AnMBR (ADI systems Inc.)
Dissolved biogas floatation	Floatation	Flotamet® (GWE); AFR (Paques)
Hybrid technology	Various	NewBio

### 1.3.1 Three-phase hood separator

The three-phase hood separator is the most common way to maintain high solids concentrations within continuous high-rate biogas anaerobic reactors. These are essentially inverted funnels in which biogas is accumulated. Wastewater continues upwards, while sludge settles back into a sludge blanket maintained in the bottom part of the reactor. This kind of separator has first been successfully applied in the UASB (Upflow Anaerobic Sludge Blanket) reactor, a technology initially developed by Gatze Lettinga, emeritus professor at Wageningen University, and co-workers. To the author's knowledge, the first patent protecting a three-phase hood separator stems from 1976 and was submitted by the Dutch company CSM (NL166000). Figure 1.2 shows a technical drawing taken from that patent. Interestingly enough CSM is a sugar producing company (Centrale Suiker Maatschappij, which stands for Central Sugar Corporation).

The efficiency of the three-phase separator strongly relies on the sedimentation properties of the anaerobic sludge grown inside the reactor. At the same time, the separator itself creates part of the conditions needed for growing sludge with good sedimentation properties. The sludge blankets inside such reactors usually consist of granular sludge, although flocculate biomass is also found, specifically in the treatment of low-strength wastewater.



**Figure 1.2 D**rawing of a three-phase hood separator taken from patent NL166000. 1. Three-phase separator, 2. Inclined wall of gas collection hood; 3. Water upflow channel, 4., 5. Biogas deflection baffle, 6. Biogas deflection baffle, 7. Biogas exit, 8. Water launder, 9. Biogas collection space.

It is common knowledge that any micro-organism will do all within its power to maintain itself inside a suitable environment for its propagation. Thus, a common strategy to agglomerate micro-organisms is the application of an HRT that is smaller than the doubling time of the slowest growing organism. If certain other process conditions are applied, one can stimulate micro-organisms to form aggregates with a high sedimentation velocities (as high as 60 m/h) (Hulshoff-Pol et al., 2004). First of all, inorganic, inert precipitates should be present to provide surface for bacteria to adhere to. Commonly, these precipitates are calcium salts and therefore calcium should be present in sufficiently high concentrations. Besides providing the surface, the inorganic precipitates also increase the aggregate's weight and it settlability. Secondly, shear forces applied should not be too strong, *i.e.* reactor mixing should be mild, yet sufficient to avoid mass transfer limitations. If these basic conditions are maintained, granules may be formed inside a reactor.

The UASB reactor was the first of its kind to provide the conditions that led to controlled anaerobic granule formation. By installing the three-phase hood separator in the top of the reactor and a water distribution system in the bottom section of the reactor, the mild mixing condition can be achieved. The wastewater to be treated is pumped into the reactor through the distribution system and provides a continuous upflow. To maintain a constant upflow,

treated wastewater may be recycled. Common upflow rates range from  $0.5~\text{m}^3/\text{m}^2$  reactor surface/h up to  $1.3~\text{m}^3/\text{m}^2/\text{h}$  (Driessen & Yspeert, 1999). Additional mixing is provided by biogas formation. Normal volumetric organic loading rates applied for industrial wastewater treatment go from 5 to 15 kg COD/m³/day (Driessen & Yspeert, 1999).

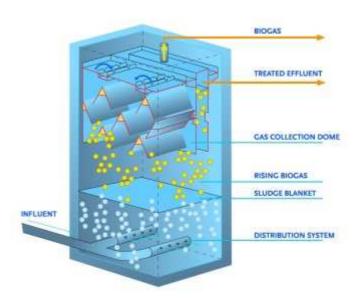


Figure 1.3 Schematical drawing of a UASB reactor (Paques Natural Solutions)

Due to widespread industrial application, the UASB concept evolved into more advanced anaerobic treatment technologies that all apply the three-phase separation technique. Three common ways were applied to improve treatment efficiency:

- Technological improvement of the three-phase hood separators;
- Increasing the height of the reactor;
- Putting two UASB reactors on top of each other.

All these developments allowed for better mixing conditions and less mass transfer limitations. Through some examples these three points will be further analysed.

Several patents exist that offer improvements of the three-phase separators. In patents registered by Paques and Passavant, the layer of hoods is increased from one to three as to increase biogas capturing efficiency (Vellinga, 1986 & 1987; Zumbragel et al., 1994). In the case of Paques, this concept evolved into the UASB reactor as it is currently put on the market.

Figure 1.3 shows a 3D representation of this reactor. Paques also developed a simpler threephase hood that makes it possible to put another treatment on top of a UASB reactor (Vellinga, 2003). This invention was specifically meant to be able to put compact complete treatment systems on the market.

To improve solids retention and get a more clarified effluent, some companies tried to incorporate another separation step right after the three-phase hoods. The Belgian company Biotim (currently part of the Waterleau Group) mounted a tilted plate separator (TPS) on top of the hoods (Pereira, 2000). The same kind of approach can be found in a patent filed by Paques (Vellinga, 1999).

A separate development was the use of the gas lift effect to swing solids back into the reactor and improve solids separation. Already in 1978 CSM filed a patent (NL7805584) in which a tilted baffle was put underneath the three-phase hood to make use of the biogas produced to swing solids back into the sludge blanket. This principle was also applied by Biothane (currently a Veolia company) for their UASB and they eventually took it to the next level of treatment by also increasing the height of the reactor. This resulted in the development of the Biobed® reactor, probable the most successful version of the EGSB or Expanded Granular Sludge Blanket.

By increasing the height of a UASB reactor, one increases the potential for blanket expansion. In other words, the upflow velocity can be increased and mixing can be improved. This way higher volumetric organic loading rates become possible. As indicated before, Biothane's Biobed® has become the most successful application of the EGSB. Figure 1.4 shows two 3D drawings of this kind of reactor (reproduced from the Biothane website).

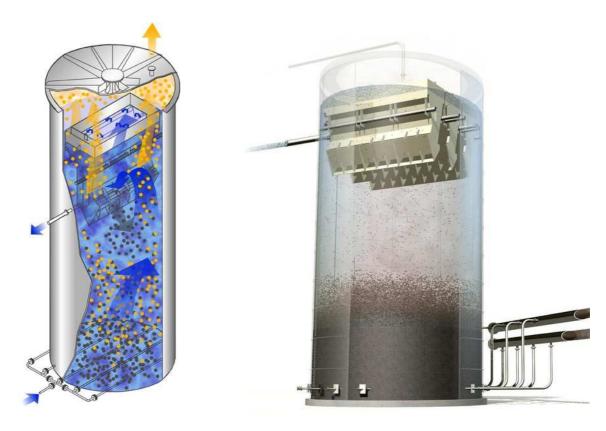


Figure 1.4 Schematical drawings of the Biobed® EGSB reactor (Biothane, Veolia Group).

The Biobed® three-phase separator (Heijnen et al., 1999; Versprille & La Vos, 2007) is a bit different from the UASB hoods in that it does not capture the biogas, but separates the liquid from the granular sludge and the biogas. Tilted baffles are installed underneath the separator and serve to separate the gas from the sludge/liquid mixture. At the same time, suspended solids are swung back into the reactor. Solids that are entrained by the liquid can still be separated in the section above the tilted baffles and it is specifically to this that patent EP1806324 (Versprille & La Vos, 2007) refers. In this section a TPS can be installed to improve solids separation. Whether this is as such applied is unknown to the author. In the Biobed® and EGSB reactors in general all reactor mixing is achieved through a liquid recycle and the biogas formation inside the reactor. Upflow velocities applied range up to 8 m/h. Volumetric loading rates can go up to 20 kg COD/m³/day.

At this moment, the reactor technology that is finding more application is the one in which two UASBs are put on top of each other and among these the Internal Circulation reactor (IC® reactor) developed by the Dutch company Paques Natural Solutions (Vellinga, 1986 & 1994; Vellinga et al., 2006) is by far the most sold. There are other companies that have patented

very similar technologies. These companies include Aquatyx (Knörle & Widak, 2007; Menke et al., 2010), US Filter (Rosmanith, 2003), Va Tech Wabag (Datschewski, 2004) and CT Umwelttechnik (Tippmann, 1997). Then there are private inventors as well that have registered such patents (Zao & Wang, 2008). Only the IC® reactor will be discussed.

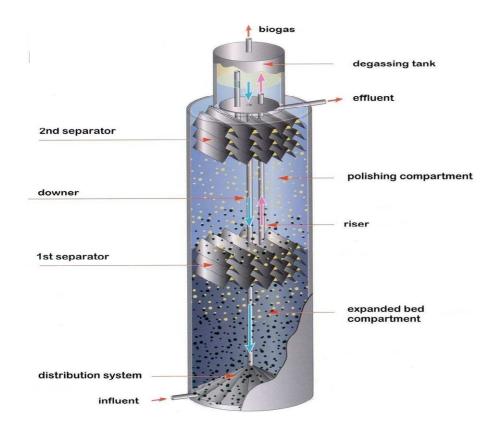
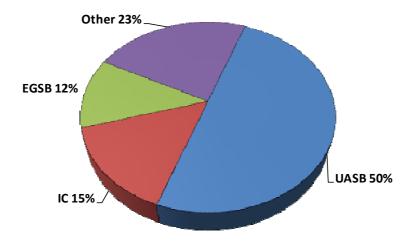


Figure 1.5 A schematical drawing of the IC® reactor as marketed by Paques Natural Solutions.

Figure 1.5 shows the internal design of the IC® reactor (Mulder, 2003). In the lower UASB section most of the biogas is produced, which is used to induce an internal gas lift loop providing an internal water circulation. The gas drags reactor contents up through riser tubes to a separation vessel mounted on top of the reactor. In this vessel, biogas is separated from the reactor fluid resulting in a density increase. As a result, the fluid flows through a downer tube to a distribution system in the bottom section of the reactor where it is joined with reactor feed wastewater. This circulation serves to increase reactor mixing and to neutralise and homogenise wastewater fed to the reactor. The top UASB serves as a polishing section in which lighter solids are allowed to settle and residual biogas is collected. Reactor mixing is obtained through the internal circulation provided by the gas lift loop, by recirculating reactor effluent with a pump and by the biogas produced. Upflow velocities applied in this kind of

reactor go up to 20 m³/m²/h in the lower UASB compartment and 8 m³/m²/h in the top compartment (Mulder, 2003). Volumetric loading rates applied at industrial scale go as high as 36 kg COD/m³/day (Driessen & Yspeert, 1999).

The UASB reactor and its direct descendents are the most applied high-rate anaerobic technology. Currently, about 75% of the global market is in hands of three companies that put this kind of technology on the market. These are Paques, Biothane and Biotim. The dominance of UASB type technologies is demonstrated by the pie-chart depicted in Figure 1.6 (Van Lier, 2008).



**Figure 1.6** Percentages of UASB type reactors of total registered anaerobic reactor installed worldwide between 1981 and 2007 (total of 2266) (adapted from Van Lier (2008)).

UASB type technologies have found strong application within the treatment of wastewater from industries where conditions for optimal operation can be easily assured. These are in general readily accessible COD (volatile fatty acids, carbohydrates, lower alcohols), a low suspended solids concentration (Massé et al., 2001) and the virtual absence of fat. The last mentioned is important due to the heavy reliance on sedimentation as primary means of solids retention. UASB technology can be used to treat wastewater laden with fat provided it is pretreated (Caixeta et al., 2002) with *e.g.* a floatation step (Del Nery et al., 2001). Industries served with the kind of technologies described above are primarily the brewing industry, pulp & paper industry and beverages industry. Besides these, less known applications of UASB

technology are found in the chemical industry, for the treatment of wastewater generated at facilities producing terephthalic acid (personal experience).

### 1.3.2 Fixed film reactors

In biofilm reactors a surface is provided to which micro-organisms can attach. The surface provided can either be fixed or moving; here the focus lies on the fixed kind and it is introduced into the reactor using media to which the bacteria can attach, such as activated carbon, PVC, rock or ceramic rings (Rajeshwari et al., 2000). Normally one tries to maximise the surface available for micro-organisms to grow on as to maximise the biomass concentration and minimise mass transfer limitations. There are several ways of doing this, including filling up a reactor with material with a high specific area (Särner, 1990; Odegaard, 1995), installing a large amount of small diameter tubes inside a reactor (Rey et al., 2006; Diez Blano & Del Pozo Fernandes, 2002) or installing parallel porous sheets inside a reactor (Herding et al., 1997 & 2005; Herding & Herding, 2005). Besides the kind of biofilm support or the way it is installed, a variety of fixed film reactors have been developed that differ from each other in terms wastewater distribution (Cummings, 2006) or the way the process is operated.

The main advantage of biofilm reactors is their simple design, potentially resulting in a reduction of investment costs, although Rajeshwari et al. (2000) actually stated that UASB investment costs are lower. Other advantages indicated are their resistance to shock loading and their stability at higher organic loading rates (Acharya et al., 2008). At the same time, as potential drawbacks the occurrence of mass transfer limitations and the likelihood of short circuiting due to excessive biomass growth can be indicated. One of the solutions introduced to counteract these problems is intermittent reactor backwashing (Torrijos et al., 2006) or reactor flushing (Thomas & Michotte, 2009). This way solids accumulation is prevented and biofilm thickness is controlled. The French company Naskeo recently successfully put such a technology (Torrijos et al., 2006) on the market under the name Proveo®. They claim that volumetric loading rates as high as 35 kg COD/m<sup>3</sup>/day can be achieved. At lab-scale, Iscen et al. (2007) applied loading rates up to 6.5 kg COD/m<sup>3</sup>/day for treatment of cake production wastewater with an upflow anaerobic packed bed reactor. They stated, however, that removal efficiency decreased with increasing loading rate. Acharya et al. (2008) reached loading rates of up to 31 kg COD/m<sup>3</sup>/day on distillery spent wash. At these high loading rates, however, removal efficiencies did not go beyond 50 %, thus putting question marks at the applicability of such loading rates. Rajeshwari et al. (2000) indicated loading rates applied between 1 and 50 kg COD/m³/day.

Besides the back washing or flushing, excessive wash-out of solids from fixed film reactors is prevented coupling the fixed film to some kind of separation technology, such as electrocoagulation (Blum, 2010) or membrane filtration (Bohm & Hennequin, 2010). This, however, is already a grey area in which not so much an apparatus is protected but a process consisting of two separate steps.

It does not require explanation that these kinds of reactor will normally have problems with wastewater with high solids concentrations and wastewater containing hydrophobic substances. In fact, Alves et al. (1997) reported that biomass adherence to support material diminished due to the presence of lipids. Although it is tried to increase surface area as much as possible as to grow sufficient biofilm, mass transfer limitation are bound to occur.

#### 1.3.3 Fluidised bed reactors

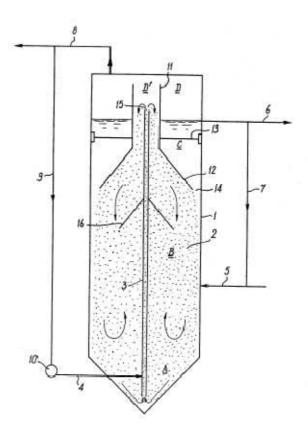
In a fluidised bed reactor biofilms are grown onto a bed of carrier material, which is subsequently expanded using a liquid recycle. Materials applied are the same kind as applied for fixed film reactors. Depending on the operating conditions, this carrier material either has a density higher or lower than water. Accordingly, two kinds of fluidised bed reactors may be found in literature:

- 1. The normal fluidised bed, in which a settling bed of carrier material is fluidised by applying a liquid circulation that is injected at the bottom of the bed, thus resulting in fluidisation. The carrier material essentially has a density higher than water;
- 2. The downflow fluidised bed, in which a floating bed of carrier material is fluidised by applying a liquid downflow. In this case, the carrier material has a density inferior to water.

The normal fluidised bed is a commonly applied technology and well described in be it scientific or patent literature. Specifically the patent literature seemed to have got stuck in the 80s of the past century. This is only natural, since the anaerobic fluidised beds recorded stay along the lines of those that already exist: aerobic fluidised beds (Heijnen et al., 1983) or chemical fluidised beds. Thus, most of the patents refer to add-ons, such as skimmers (Josse & Sutherlin, 2003) or pH control (Lias-Franken Leichtbaustoffe, 1989), several liquid recycles (De

Paoli et al., 1984) or a combination of anaerobic reactors (Barnes & Bliss, 1984). The basic design, however, practically stays the same. The downflow fluidised bed is less common and therefore shows a different picture.

As previously said, the conventional way of fluidising a biobed is by applying a liquid recycle. As is the case with the IC® reactor, one can enhance the fluidisation by applying a gas lift loop, a method patented by Degremont already in 1984 (Rovel et al., 1984). In this case compressed biogas is applied instead of creating a natural gas lift, as is shown in Figure 1.7. In contrast, however, Diez Blanco et al. (1996) reported that biogas production as such had little effect on the hydrodynamic regime within the fluidised bed reactor. Buffière et al. (1998) even stated that biogas production has an adverse effect on the contact between liquid and bioparticles, thus potentially reducing efficiency.



**Figure 1.7** Fluidised bed including an induced gas lift by recycling biogas (Rovel et al., 1984). 1. Reactor vessel, 2. Carrier material, 3. Riser tube, 4. Pressurised biogas, 5. Wastewater feed, 6. Water effluent, 7. recycle, 8. Biogas, 9. Biogas recycle suction side, 10. Pressure pump, 11. Separation funnel, 12. Flared wall, 13. Fastening arrangement, 14. Open space for carrier sedimentation and water upflow, 15. Riser outlet, 16. Turbulence deflectors.

Conventional fluidised beds are normally associated to higher volumetric loading rates than the reactor in which three-phase separation is applied. The higher applicable loading rates are due to their more turbulent hydrodynamic regime, eliminating mass transfer limitations (Buffière et al., 1998). Rajeshwari et al. (2000) indicated an operating range between 1 and 100 kg COD/m³/day. Organic loading rates of up to 40 kg COD/m³/day at industrial scale have been reported ((Buffière et al., 1998). However, to achieve this more turbulent regime a bigger energy input is required, making the fluidised bed less attractive than for example the UASB type reactors.

An interesting development taking place is the development of floating or down-flow fluidised bed reactor in which carrier material with a lower density than water is used. Carrier materials commonly used are plastics or foams. The patent literature mainly focuses on how to achieve bed expansion and apparently the best way of doing so is the application of a gas lift induced by recycling gas (Shimodaira et al., 1984; Antunes, 1998; Haridas & Majumdar, 2002). The inventions only differ in their potential applications and reactor compartmentalisation (Antunes, 1998). Applications in the absence of oxygen defined are denitrification (Shimodaira et al., 1984), sulphate reduction (Haridas & Majumdar, 2002) and actual biogas production (Antunes, 1998). Buffiére et al. (2000b) investigated a downflow fluidised bed with gas recycle and indicated several advantages such as easy bed height control, easy sludge recovery and less clogging problems due to application of a gas recycle.

Garcia-Calderon et al. (1998) reported on a down-flow fluidised bed reactor tested on wine distillery wastewater. They achieved an OLR of 11.3 kg COD/m³/day with an organic carbon removal efficiency of 85 %. On the same kind of wastewater Buffiére & Moletta (2000a) an OLR of 30 kg COD/m³/day at lab scale with 77% organic carbon removal efficiency, while Sowmeyan & Swaminathan (2008) reported achieving an OLR of 35 kg COD/m³/day with 84% COD removal efficiency.

## 1.3.4 Anaerobic baffled reactor

The anaerobic baffled reactor (ABR) has many faces. Basically baffles are introduced in a reactor vessel resulting in a compartmentalisation of the reactor. This way, the anaerobic treatment process can be divided into many compartments and as many treatment phases. The main goal is to improve solids retention by strategically putting baffles in place that direct

the water up and down. This would result in a more robust anaerobic process with promising commercial perspectives. Figure 1.8 shows a schematic representation of the ABR.

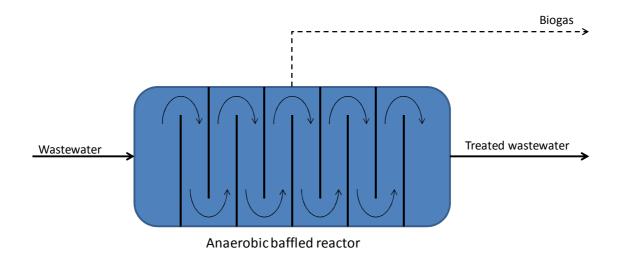


Figure 1.8 Schematic representation of the anaerobic baffled reactor (ABR).

Barber & Stuckey (1999) reviewed the use of the ABR for wastewater treatment in general and of its technological development. Another review was done by Liu (2010) and between the last and the previously mentioned review, practically no full-scale applications are reported despite of the long list of advantages given by Barber & Stuckey (1999). Removal efficiencies obtained and organic loading rates applied at lab scale for several kinds of wastewater are comparable to the ones achieved with conventional technologies. Akunna & Clark (2000) reported on a granular-bed anaerobic baffled reactor (GRABBR) for the treatment of whisky distillery wastewater. This reactor supposedly combines the advantages of the ABR and granular sludge. Thus, a reactor with efficient sludge retention and treatment phase separation was obtained. The organic loading rate applied, however, did not surpass 5 kg COD/m³/day at a minimum hydraulic retention time of 2 days. Faisal & Unno (2001) used a modified ABR (MABR) to treat palm oil mill effluent (POME) and applied loading rates up to 5.3 kg COD/m<sup>3</sup>/day with HRTs not going lower than 3 days. In their review, Barber & Stuckey (1999) reported OLR ranging from 0.9 to 28 kg COD/m<sup>3</sup>/day for several kinds of industrial wastewater, while Liu et al. (2010) reported a range from 1.5 to 13.4 kg COD/m³/day for high strength organic wastewater.

#### 1.3.5 Anaerobic membrane bioreactor

Between the indicated anaerobic technologies, the anaerobic membrane bioreactor (AnMBR) may be considered as different, since separation is essentially based on particle size and not on sedimentation. Configurations in which submerged membranes or external membranes are applied have both been investigated and tested. Figure 1.9 gives a schematic representation of both configurations. When looking at it, the question rises whether the external membrane configuration should actually be considered as an anaerobic membrane bioreactor as such, since basically two process units are connected in series: an anaerobic reactor of any kind and a membrane separation step.

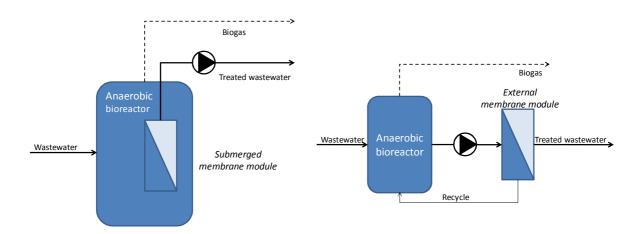


Figure 1.9 Anaerobic membrane bioreactors with submerged membranes (left) and external membranes (right).

The submerged membrane version of the AnMBR has found industrial scale application (ADI Systems Inc., Canada) for the treatment of industrial wastewater, although their application for municipal wastewater treatment is also being investigated (personal experience). The application of the AnMBR for anaerobic treatment was investigated for continuous anaerobic sulphate reduction using extremophiles (Valero, 2003). Fuchs et al. (2003) investigated the application of a stirred anaerobic reactor coupled with an external cross-flow membrane for the treatment of sauerkraut wastewater and slaughterhouse wastewater. Saddoud et al. (2007) successfully tested a two-phase anaerobic process coupled with external membrane for whey treatment.

One of the advantages indicated is the fact that neither granulation nor flocculation of biomass is required to retain biological activity inside the reactor. Other clear advantages are the

clarified effluent of the AnMBR and the possibility to operate outside the optimum temperature regime. As main disadvantage cake layer formation on the membrane has been indicated and not so much membrane fouling. This limits the applicable membrane fluxes and therefore the wastewater flow rates as well, thus making the AnMBR specifically interesting for more concentrated wastewaters (Jeison, 2007). It is not clear whether submerged or external modules are preferred. In terms of energy requirements, the submerged membranes seem to be superior. In terms of cake layer formation, the external modules offer better options, since the shear stress applied can be easily controlled by applying higher circulation flow rates. However, this might have a counteractive effect, since excessive shear rate might result in smaller particle formation due to aggregate breakup, which in its turn may lead to membrane pore fouling and reduced fluxes. Next to that, Padmasiri et al. (2007) stated that high shear stress has an adverse effect on bioactivity as well. In the case of applying submerged modules integrated in an anaerobic bioreactor, one of the preferred ways of counteracting membrane fouling is the application of gasification next to the membrane. For the AnMBR the obvious choice for this would be biogas, although nitrogen and helium gas have also been indicated as a possibility (Van Dijk & Ramaekers, 2005).

The applicability of the AnMBR for treatment of wastewater with a lot of particulate organic matter was investigated by Fuchs et al. (2003) and Jeison (2007 thesis) and it showed one of the dilemmas encountered when dealing with AnMBR. Inherent to the application of membranes is the almost complete retention of particulate matter, even when not biodegradable. This naturally results in accumulation of this material, which can only be discharged through the discharge of sludge. In this way the amount of active biomass retained inside the reactor is limited, thus limiting the volumetric loading rates that may be applied (Jeison, 2007). Fuchs et al. (2003), however, regards the complete retention as an advantage, specifically when combined with vigorous mixing, since it will make less accessible organic material more susceptible to anaerobic digestion. This is in contradiction with what Padmasiri et al. (2007) indicated concerning the negative effect of high shear on biomass. Typically, volumetric loading rates do not exceed 10 kg COD/m³/day. Fuchs et al. (2003), as well as (Jeison 2007), indicated that membrane fouling with organic material is probably the main challenge for AnMBR technology. One can expect that fouling could be even worse when lipids are present in the wastewater.

#### 1.3.6 Anaerobic sequencing batch reactor

As the name suggests, the anaerobic sequencing batch reactor (ASBR) is operated sequentially. Normally, its operation sequence consists of the following steps (Massé & Masse, 2000b):

- 1. Feed: during a defined period of time wastewater is fed to the reactor;
- 2. React: after feeding is stopped, the wastewater is contacted with anaerobic sludge by applying some kind of mixing. This might be by stirring, by applying a liquid circulation or a biogas circulation or through a combination of these three. During this phase most of the biogas is produced. It is common to link the duration of this period to the biogas production rate. Normally, one waits until the biogas production starts diminishing;
- 3. Settle: stirring is stopped and the anaerobic sludge is allowed to settle for a certain period of time until a clear supernatant is obtained. During this phase residual biogas leaves the liquid phase;
- 4. Draw: the cleared liquid is drawn off down to a predetermined level. This might be by gravity or by active pumping. In any case one needs to make sure not to draw off sludge with the liquid.
- 5. Idle: during this stage settled sludge may be discharged as required. After the fifth stage the sequence is started again.

Zaiat et al. (2001) indicated four main factors affecting ASBR treatment efficiency: agitation, initial food to micro-organism ratio, reactor configuration and feeding strategy. Specifically when looking at the third factor, one should contemplate whether it would not be better to speak about a process operation method, instead of an anaerobic reactor. In principle, any kind of reactor technology can be applied. For example, when the reactor is mixed using an upflow velocity one can always apply a UASB, a fluidised bed or even a fixed bed. When a gas recycle is applied, there is always the possibility to introduce a gas lift loop to stimulate mixing. Therefore, it would be wrong to consider the ASBR as another reactor technology. It is preferable to speak of way of operating a process and as such the sequencing batch operational mode is quite flexible and robust and should be considered for anaerobic treatment of LCFA containing wastewater.

The ASBR including gas or liquid recycles was filed for a patent in 1991 (Dague, 1993) and basically covers the whole ASBR sequence including using liquid or gas recycles for mixing. In

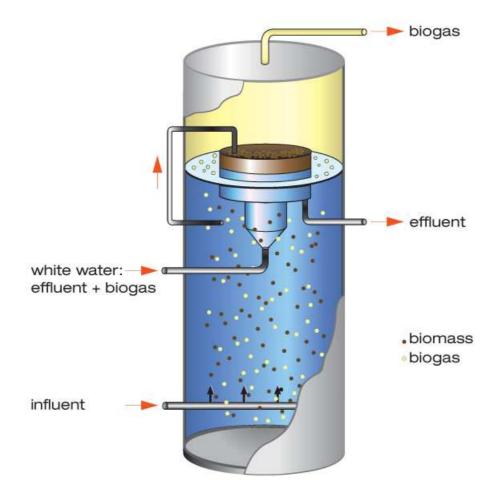
this patent the ASBR is not referred to as a reactor (apparatus), but as a process (method). This confirms what was previously indicated. Massé et al. (2001) indicated that the ASBR is highly suited for treatment of wastewater with high suspended solids concentrations, which would make it suitable for treatment of *e.g.* slaughterhouse wastewater. As advantages of ASBR technology were indicated its low investment and operating costs, and the minimum required daily maintenance (Massé & Masse, 2000b). Zaiat et al. (2001) listed several other advantages, such as the absence of primary and secondary settlers and no liquid or solid recycling. Possibly the biggest disadvantage of ASBR processing is the non-uniform biogas production (Massé & Masse, 2000b), which is an obvious result of the sequential operation.

The ASBR is another example of a promising technology (Zaiat et al., 2001) for which it is not clear whether it has actually found application at full-scale. Nevertheless, it is stressed again that it is a processing method and that in principle any kind of reactor can be operated like this, provided that enough equalizing flexibility is included in the treatment system.

#### 1.3.7 Selected hybrid anaerobic reactor technology

It is obvious that the world of anaerobic treatment is in continuous motion and inventive, novel reactor technology appears with regularity. A lot of the reactor technology that is introduced is in fact an optimisation or modification of already existing configurations. Rarely completely new concepts are encountered. Here a few concepts are dealt with that claim to be able to treat wastewater containing fat.

Recently the bio-nest reactor was filed (Dong & Yang, 2005). It is a reactor comprising at least two nests of fibrous material that retain biomass inside the reactor and to which biomass can attach. This technology was conceived to be able to directly treat lipids containing dairy wastewater, in which lipids form at least 25 % of the COD. COD concentrations, however, do not go beyond 4 g/L. Volumetric loading rates are indicated to lie between 10 and 20 kg COD/m³/day.



**Figure 1.10** The AFR® reactor as developed and commercialised by Paques Natural Solutions (Vellinga & Mulder, 2004).

The Dutch company Paques has recently developed a reactor that is capable of treating wastewater with high fat concentrations. It is called the Anaerobic Floatation Reactor (AFR®) and uses biogas to retain floating solids inside the reactor (Vellinga & Mulder, 2004). Figure 1.10 Shows a 3D representation of this reactor. Biogas is compressed together with wastewater and then expanded to create so called "white water" (water with very fine gas bubbles), a technology also used in dissolved air floatation (personal experience). With this white water solids with a tendency to float are separated from treated wastewater and retained, while the treated water is drawn off from underneath the floating layer. In the reactor an upflow distribution system is still applied.

NewBio registered a hybrid reactor that is capable of directly converting fat into biogas (Stormo, 1998 & 2002). It includes one or more submerged mechanical arms that distribute wastewater in a circular motion. Furthermore, sludge is fluidised from the bottom section. Finally, a filter bed is installed in the bottom of the reactor with which the treated wastewater

is filtered. The filter can be backwashed with another mechanical arm that moves through the filter. A big drawback of this technology is the inclusion of several mechanical arms that will be subject to considerable mechanical wear along time, thus resulting in increased maintenance cost.

Haridas et al. (2005) described the buoyant filter bioreactor (BFBR) as a reactor capable of directly treating wastewater containing fat and as such tested it on synthetic dairy wastewater. It is a partitioned reactor in which treatment takes place in a lower compartment that is connected to a second compartment by a buoyant filter containing polystyrene balls. The system includes a u-tube for automatic back flushing through gas action. Biogas is recycled using a compressor to induce the back flushing. Furthermore, accumulated scum on top of the water surface is collected in a scum collection chamber from which it is pumped and injected in the bottom of the reactor to provide additional mixing. They reached a loading rate of 10 kg m³/day and a COD removal efficiency of 90%. This reactor has been patented (Haridas, 2003).

## 1.4 Fat treatment problems identified

Reliable technologies, and specifically the UASB reactor and derived designs, promoted the confidence in AD technology. Although the anaerobic treatment of more complex industrial wastewater is being intensively investigated and even applied at industrial scale, high-rate anaerobic reactor technology is still essentially used for the treatment of effluents from distilleries, breweries, and the pulp & paper and beverages industries. Its position in the wastewater treatment and bioenergy market can be further increased, if the range of suitable substrates is expanded. Analysing the technologies, applications and problems, it is apparent that wastewaters with high lipid contents are not effectively treated with existing conventional high-rate anaerobic reactor technology. The identification of problems currently encountered may lead to a technologically viable solution for the anaerobic treatment of such wastewaters.

Fat and more specifically LCFA have several properties that make them relatively difficult to be removed under anaerobic conditions or even aerobic conditions. Pereira (2003) gave a nice overview of these specific problems and which will be briefly addressed in this section.

#### 1.4.1 LCFA density & surface activity

The presence of LCFA may lead to disintegration of aggregates. As a reason for disintegration the surface activity of LCFA is given (Pereira, 2003). LCFA have a hydrophobic tail and a hydrophilic carboxylic head and act as surfactants at neutral pH, lowering the surface tension and inhibiting aggregation of hydrophobic bacteria. Consequently, one of the main parameters for successful biomass retention in most of the currently applied anaerobic technologies is affected. Hwu et al. (1997a), for example, reported on wash-out of small particulate matter from thermophilic EGSB reactors fed with LCFA containing wastewater operated at different upflow velocities and HRTs, showing that more wash-out occurred from the EGSBs operated at higher upflow velocities and lower HRTs. Besides the hydrodynamics, they attributed this to the surface activity of LCFA (Hwu at al., 1997b).

A well-known characteristic of LCFA is their lower density than water, which gives them a tendency to float. As the first mechanism of LCFA removal, LCFA adsorption is indicated (Petruy & Lettinga, 1997; Hwu et al., 1998b; Nadais et al. 2003). Therefore, any reactor exposed to fat containing wastewater will contain some sludge with floating tendencies. One can only imagine what happens when relatively high fat concentrations are present. According to Pereira (2003) several authors reported on the failure of conventional reactors due to floating sludge. Nuñez & Martinez (1999) stated that the presence of fat leads to floatation, biomass wash-out and the production of poorly stabilised sludge. Hwu et al (1998a) observed three-phase hood clogging due to fat induced floating layers. They resorted to daily removal of the fat layer as to prevent hood clogging. Caixeta et al. (2002) stated that direct treatment of slaughterhouse wastewater with a conventional UASB would lead to solid accumulation in the three-phase separator, leading to biogas loss with the reactor effluent. Martínez et al (1995) concluded that high contents of solids & fats disturbed the proper operation of anaerobic treatment of slaughterhouse wastewater and recommended prior floatation for fat & solids removal to improve the anaerobic operation. In fact, floatation seems to be more critical for conventional reactor failure than potential LCFA inhibition or toxicity (Hwu et al., 1998b; Alves et al., 2001). Therefore, Mittal (2006) indicated that treatment efficiency increases if fats and suspended solids are removed prior to anaerobic treatment. This is specifically true if one wants to apply UASB technology (Rajeshwari et al., 2000).

A result of floatation is the formation of a layer on top of the water surface. As previously indicated, LCFA at neutral pH act as surfactants and as such also stimulate the formation of foam. It is not difficult to imagine what happens if a layer of surface active material is floating

on top of a liquid volume in which biogas is being produced: an ideal situation is created for excessive foam formation. This might result in problems in the biogas line and inefficient biogas recovery (Ganidi et al., 2009), if adequate action is not taken.

#### 1.4.2 LCFA toxicity& inhibition

Until recently LCFA were considered toxic to anaerobic biodegradation above certain concentrations (Hwu et al., 1996, Lalman & Bagley, 2000 & 2001). Specifically suspended & flocculent biomass showed to be more sensitive to inhibition than granular biomass, independent of the source of the biomass.

Pereira et al. (2002), however, showed that LCFA can be efficiently mineralised in batch vials under anaerobic conditions after being encapsulated by LCFA. Pereira (2003) confirmed that LCFA can be degraded under anaerobic conditions and that there was no permanent toxic effect towards the anaerobic consortia. Furthermore, Hwu et al. (1997b) reported that recycling washed out particulate matter from an EGSB treating oleate containing synthetic wastewater improved its removal considerably, thus showing that the success of an LCFA treating reactor relies on appropriate design. Therefore, taking into account that floatation and particulate biomass wash-out in conventional reactors occur before inhibition, the potential toxicity of LCFA is not considered a problem to be resolved.

## 1.4.3 Mass transfer limitations

Another important problem occurring when dealing with LCFA is the occurrence of mass transfer limitation. Rajeshwari et al. (2000) listed three important factors for reactor choice: amount of biomass to be retained, contact opportunity between wastewater and biomass and diffusion of substrate within the biomass. Basically all of these factors have to do with mass transfer and these should be taken into account as to minimise limitations.

Contact area between substrate and biomass is important to look at. Pereira (2003) reported that flocculent anaerobic sludge was more efficient in degrading LCFA than granular sludge, suggesting that the specific contact area between LCFA and sludge is of importance. Hwu et al. (1997b) carried out batch tests with crushed and normal granules from an EGSB treating wastewater with oleate and showed that the crushed granules had a higher specific methane

activity, confirming that mass transfer should be taken into account. Massé & Masse (2000b) operated ASBRs with slaughterhouse wastewater and inoculated with sludge from two different sources: granular sludge from a milk processing plant and anaerobic flocculent sludge from a municipal wastewater treatment plant. They stated that the flocculent sludge initially performed better, but eventually granular sludge caught up with flocculent sludge possibly because it disintegrated over time. Thus the increasingly better performance of the granular sludge might have been due to an increasing contact area between wastewater and sludge.

Encapsulation with whitish biomass is reported several times (Hwu et al., 1998b; Alves et al., 1997 & 2001) and this appears to be the results LCFA adsorption onto the sludge. LCFA adsorption was also reported on by Nadais et al. (2003). Thus, a hydrophobic layer may be created around the sludge aggregate making mass transport from and to the aggregate more difficult (Figure 1.?). Consequently, products may accumulate and these include methane, carbon dioxide, but potentially volatile fatty acids as well. Possibly, the encountered irreversible toxicity had to do with VFA toxicity instead of LCFA toxicity. In any case, mass transfer limitation should be avoided a much as possible. Hwu et al. (1998a) showed, however, that one cannot simply apply more turbulent conditions as to avoid mass transfer limitations. They stated that normal EGSB conditions, *i.e.* "higher-rate" conditions, should not be applied and preference should be given to milder mixing conditions. Although Hwu et al. (1998a) used EGSB technology and granular sludge, for a novel technology it may be beneficial to come to a compromise and apply relatively mild mixing conditions.



Figure 1.11 Biomass encapsulated by LCFA (Pereira et al., 2005)

This brief analysis of problems encountered with LCFA forms the basis of the next step to be taken: the conception of a novel high-rate anaerobic reactor for the treatment of industrial wastewater containing fat. The objective of the research reported in this thesis was to conceive, develop & test, demonstrate at pilot scale and scale up a technology capable of doing so. To all of these stages a chapter is dedicated. Finally, some future perspectives are given.

### 1.5 References

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# 2 Reactor conception: The Inverted

# **Anaerobic Sludge Blanket**

## 2.1 Introduction

With the main processing problems of lipids identified, it is now possible to define a technological solution that makes it possible to directly treat industrial wastewater in a high rate reactor. One has all the freedom in the world to define the solution. However, in this case, a few restrictions were defined as to limit the number of possible technological solutions. The restrictions are the following:

- Avoid the inclusion of mechanically moving parts inside the reactor. When including
  moving parts, e.g. a mechanical stirrer or a moving arm, a mechanical seal needs to
  be put in place as well. Furthermore, mechanically moving parts are more susceptible
  to ware and therefore require more intensive maintenance. Therefore, mixing should
  be done by applying liquid recycles and using the biogas produced;
- Aim for a concept that allows for continuous wastewater treatment. Although several authors (Pereira, 2003; Cavaleiro et al., 2009; Alves et al., 2009) indicate that a sequencing batch approach would be the best way to stably remove lipids, in the author's view it should be possible to come to a continuous operation. This would result in the need for less buffering capacity at industrial scale.

Taking into account these two restrictions and the earlier identified existing process problems, a novel reactor concept could be drawn up: the Inverted Anaerobic Sludge Blanket (IASB) reactor.

### 2.2 IASB technology

The basis of IASB technology makes use of two properties of LCFA: its lower density than water and its amphothericity. Furthermore, optimisation of biodegradation should be promoted by preventing as much as possible any mass transport limitations. Finally, it should be taken into account that LCFA are supposed to be degraded. In other words, LCFA induced floatation might disappear along time, resulting in biosolids that settle.

In an anaerobic reactor capable of degrading fat one may expect all the following to happen:

- Presence of floating anaerobic sludge due to LCFA adsorption;
- Presence of settling sludge, since LCFA is being degraded;
- Foam formation in the head space of the reactor, which is result of biogas formation and the presence of surface reducing compounds in the reactor top;
- Potential encapsulation of biogas due to the presence of LCFA;
- Adverse conditions for the formation of biomass aggregates with high settling velocities.

Therefore, a high-rate anaerobic reactor should at least include the following:

- Floating sludge retention;
- Settling sludge retention;
- Foam counteraction;
- Biogas liberation;
- Contact surface maximisation.

Floating sludge is easily retained if instead of upflow liquid injection, such as applied in practically all conventional high-rate technology, downflow injection is applied. This way, a separation between floating and settling sludge is right away obtained. To retain settling sludge inside the reactor, a compact separator offering enough settling area may be applied. A commonly used technology is the tilted plate separator (TPS), as briefly discussed in the previous section. However, in this case it should not be top-mounted above a three-phase separation device, but bottom mounted.

Foam formation may be counteracted by spraying the floating layer with reactor effluent. This will also result in some biogas liberation. However, mixing near the floating layer should be

induced to maximise biogas liberation. This may be achieved by injecting liquid into a downer as to stimulate sludge entrainment and apply mild shear stress to open up encapsulated flocks and liberate the biogas. This will also result in the demotion of strong aggregate formation, thus maximising the contact surface between wastewater and the anaerobic population. Besides this, the settled sludge should be exposed again to incoming wastewater so that biodegradation is stimulated. Therefore, a sludge recycle should be applied and joined with incoming water. These two together can then be injected into the downer.

Figure 2.1 shows the first conceptual design of the Inverted Anaerobic Sludge Blanket (1). Wastewater containing fat or LCFA (2) is mixed in-line with a settled sludge recycle (5) and injected in a downward motion in the top section of the reactor. LCFA present in the wastewater adsorbs to at least part of the anaerobic sludge and induces floatation of this part. The injection of the sludge/wastewater influent mixture results in a downward turbulent flow inside at least one reactor downer (6). The downward injection should be big enough to stimulate sludge entrainment from the top, so that the floating layer created by LCFA adsorption becomes a dynamic floating layer where initial LCFA degradation takes place and biogas is liberated. Furthermore, the turbulence inside the downer section (6) should result in additional liberation of biogas produced. The final idea is to create circular mixing between the downer (6) and the riser sections (8) present in the anaerobic reactor. Since LCFA is being converted, at least part of the anaerobic sludge will show a tendency to settle. To retain this settling sludge, a tilted plate separator (4) is included. The treated wastewater leaves the reactor through this TPS, while the settled sludge is recycled using an adequate pump (7) and contacted again with wastewater entering the reactor. The mixing regime inside the reactor can be controlled with the sludge recycle and the wastewater flow rate. Not included in the first reactor conceptual design was the spraying of treated wastewater in the head space of the reactor to control foam formation.

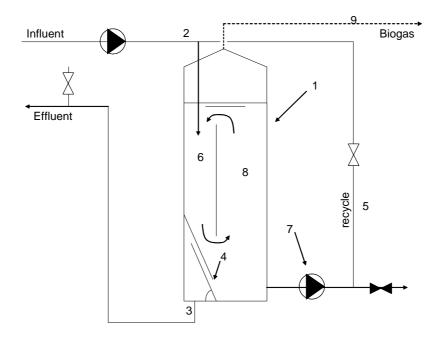


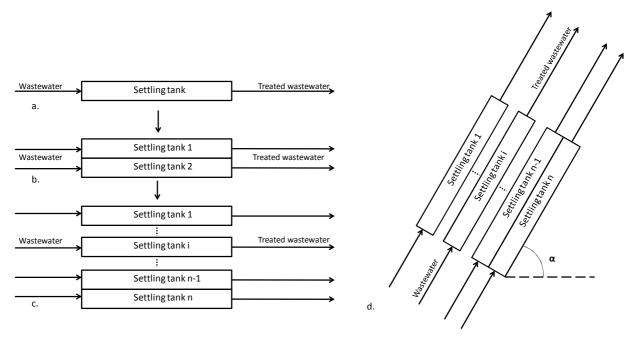
Figure 2.1 The Inverted Anaerobic Sludge Blanket concept.

The concept described was submitted for a patent application at the end of 2005 and published in 2007 (Alves et al., 2007).

# 2.3 The Tilted Plate Separator

The factor that will be determining in the design of the reactor is TPS. It needs to incorporate sufficient settling area for solids to settle and ensure biomass retention. Therefore, an analysis of TPS design criteria was necessary.

The TPS is an ingenious way of maximizing available separation area within a compact volume. It is well known that the efficiency of a rectangular settling tank is not determined by its depth, but by the settling area (WEF, 2005). This being the case, one could imagine increasing the settling area available by installing n settling tanks or plates on top on top of each other (Figure 2.2c). This, however, would be a unpractical, since it would be difficult to efficiently collect the solids that settle at the bottom of each plate. If inclined, however, at an angle  $\alpha$  with the horizontal (Figure 2.2d), the solids may slide off the bottom of each plate towards the lowest point. By inclining the plates, however, the effective area available for solids settling is the projected area of the n tanks or plates. In spite of this, one can considerably reduce the separator's footprint.



**Figure 2.2** From simple sedimentation tank to tilted plate settler. a. One rectangular settler; b. Two rectangular settlers; c. n rectangular settlers; d. n rectangular settlers inclined at an angle  $\alpha$ .

With reference to Figure 2.3, the following equations apply for the design of a TPS unit (WEF, 2005).

$$u_{sx} = u - u_s \sin \alpha \tag{2.1}$$

$$u_{sy} = -u_s \cos \alpha \tag{2.2}$$

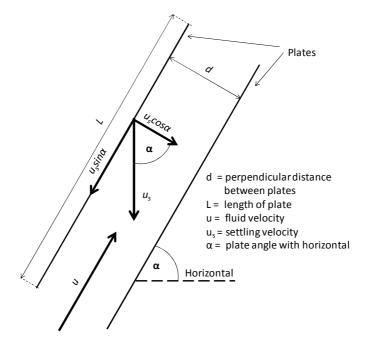
In which:

 $u_{sx} = particle settling velocity parallel to the plate (m/h)$ 

 $u_{sv} = particle \ settling \ velocity \ perpendicular \ to \ the \ plate \ (m/h)$ 

 $u = fluid\ velocity\ (m/h)$ 

Particles that need to settle in the TPS have to travel a maximum distance d (m), which is the perpendicular distance between the plates. Thus, one needs to provide a settling time  $t_s$  (h) that is less than the residence time  $t_{TPS}$  of the fluid between in the TPS. This results in the following design definition.



**Figure 2.3** Parameter definition for TPS design. Units of parameters used: L (m), d (m), u (m/h),  $u_s$  (m/h),  $\alpha$  ( $^{o}$ ) (adapted from WEF (2005)).

$$t_{S} \le t_{TPS} \tag{2.3}$$

$$\frac{d}{u_{sy}} \le \frac{L}{u} = \frac{nLWd}{Q_f} = \frac{A_{TPS}d}{Q_f} \tag{2.4}$$

$$u_{sy} \ge \frac{Q_f}{A_{TPS}} \tag{2.5}$$

In which:

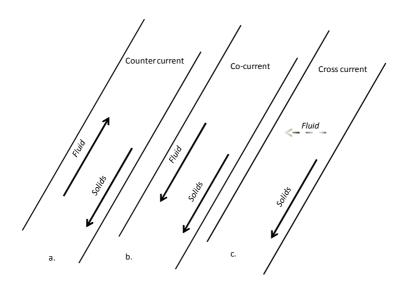
 $W = plate \ width \ (m)$ 

 $A_{TPS} = total TPS plate surface area (m^2)$ 

 $Q_f = fluid \ flow \ rate \ through \ TPS \ (m^3/h)$ 

Typical values for the inclination angle  $\alpha$  lie between 50 and 70°; the perpendicular plate distance between 0.05 and 0.10 m. A further defining factor is the hydrodynamic conditions. For undisturbed settling laminar flow conditions need to be maintained, which can be characterised by the Reynolds and Froude Numbers, Re and Fr, respectively. Literature indicates that Re should be less than 2000 and Fr should be at least bigger than  $10^{-5}$  (WEF, 2005).

Variations on the TPS have been patented many times since it, since it can be operated in several ways. The most applied for solids separation is counter current separation (Figure 2.4a), but co-current (Figure 2.4b) and cross-current (Figure 2.4c) also exist (Metcalf & Eddy, 2003).



**Figure 2.4** TPS with a. counter current, b. Co-current and c. cross current flow.

The first TPSs found in the patent literature mostly refer to oil/water separation using cocurrent upflow separators (Jakubek, 1974, Harris, 1973). Naturally, this can be combined with gas separation as well (Stevenson & Knott, 1981), since it only helps efficiency. Efficiency is further improved by introducing corrugated (Toft, 1978) or V-shaped (De Jong, 1979) plates to coalesce oil droplets. Co-current separators are less common in the patent literature for liquid/solids separation (Khudenko, 1999). Normally counter current separators are found and patent mostly differ from each other in the way they distribute the solids laden liquid underneath the inclined plates (Soriente & Gurnari, 1989; Kosonen, 1975; Hellman & Garrison, 1986) or half way using baffles to initially direct the liquid in co-current with the settling solids (Probstein & Yung, 1979). In another patent, a plate package is put inside a sedimentation basin to improve solids removal efficiency (Lindstol, 1987). Corrugated plates are applied as well (Pos, 1993) and one patent reports the inclusion of a co-current and a counter settler in series to remove gas and solids, respectively (Vellinga & Jorna, 2009). It is also attempted to include some kind of tertiary treatment after a TPS by including a bed of adsorption material

such as activated coal (St. Germain & Zuback, 2005). Another interesting improvement is the inclusion of a cleaning mechanism by temporarily putting the inclined plates at an angle of 90° (Morin, 2006). Less common in the patent literature are the cross-flow TPSs. These can be used for the removal of lighter substances such as oil (Davies & Hardwich, 1983) or for heavier solid material (Dorgeloh, 1998). Finally, some patents have been filed in which the main purpose of the TPS is liquid/gas separation. One is specifically meant for biogas collection from digesters and includes V-shaped plates with a biogas hood incorporated (Gilles, 1985). Another patent describes a vertically stacked TPS (practically all TPSs are horizontally stacked) for gas separation from liquid (Gagliardi, 1971).

This brief review of the patent literature illustrates the variety of applications of tilted plate separators. It must be stressed, though, that the basic principles are not tampered with: exceptions left aside, inclination angles practically always remain between 50° and 60° and perpendicular plate spacing lies between 0.05 m and 0.10m. Materials applied for the plates are steel or plastic, such as PVC or PP.

In the patents, it is the distribution of wastewater over the plates that received the most attention, since uneven distribution may lead to short circuiting of at least part of the settling are available. To the knowledge of the author, up to now a TPS has not been used in the way it is envisioned in the IASB reactor. What comes closest is probably the tilted conical separator (TCS) as described by Barthelme (1994). This TCS resides in the bottom section of a 3-in-1 apparatus for sludge coagulation, flocculation and sedimentation.

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# 3 Performance testing at lab scale

## 3.1 Introduction

One thing is to come up with a reactor concept; another thing is to come to a working reactor at lab-scale to demonstrate that the actual concept works. Initially a very practical and pragmatic approach was chosen, *i.e.* it was decided to leave theory aside, simply build a reactor, operate it and observe what would happen. Thus, three different lab-scale configurations were tested that corresponded to the description in the patent application (Alves et al., 2007). From a constructive point of view, a cylindrical reactor is preferred at industrial scale, because it is easier to build. However, the common way of applying tilted plate separators is, as the name already indicates, by installing parallel rectangular plates. Therefore, a rectangular shaped reactor was also considered. Both approaches are discussed in this section.

# 3.2 Experimental part

All three reactors were operated with synthetic dairy wastewater as described by Pereira (2003). Peristaltic pumps (Watson Marlow) were used for reactor feeding and sludge recycling. A biogas counter was used to register biogas production (Sinagawa Corporation, Tokyo, Japan). Reactors were inoculated with anaerobic granular sludge from a UASB reactor treating brewery wastewater.

Analyses were done according to need, since the main objective was to get as fast as possible to a working reactor concept. These included COD, total suspended solids (TSS) and volatile suspended solids (VSS) analyses of reactor contents. All were determined according to Standard Methods (APHA et al., 1998). Sporadically the biogas was analysed for methane and carbon dioxide using by gas chromatography using a Pye Unicam GCD gas chromatograph (Cambridge, England) using a Porapack Q (100–180 mesh) column. Helium was used as carrier gas (30 mL/min) and the temperatures of injection port, column and flame ionization detector were 110, 35 and 110 °C, respectively.

## 3.3 Cylindrical reactors

The first lab-scale reactor was a reproduction of the earlier presented reactor concept. Figure 3.1 shows the technical drawing forming the base of this reactor (total volume of 5 L). Main design criterion taken into account was the division of the reactor in a downer and a riser section. No special attention was given to the TPS design. The riser occupied 2/3 of the cross-sectional area, leaving 1/3 for the downer. A wall was placed to separate the downer from the riser. This wall covered 2/3 of the reactor height and was positioned 50 mm below the water surface.

This reactor configuration resulted in inadequate mixing, since the downer cross-sectional area was too big. The envisioned entrainment of sludge did not occur. Moreover, the dividing wall was too long resulting in biogas separation already in the downer. The prospected circular flow over downer and riser did not occur. It was obvious that two things needed to be adapted:

- 1. The downer cross-sectional area needed to be reduced as to increase the local downward velocity. This way entrainment would be stimulated;
- 2. The height of the wall needed to be reduced, so that biogas liberation would occur in the riser section and not so much in the downer section.

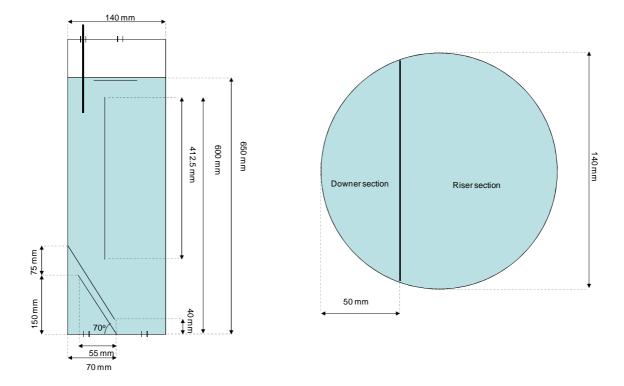
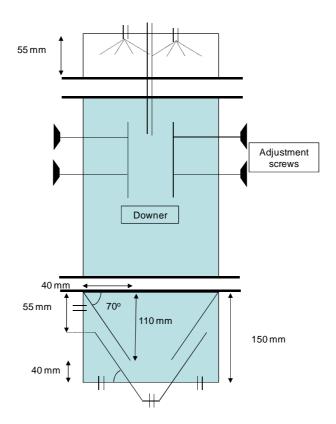


Figure 3.1 Side and top view of the first cylindrical lab-scale IASB reactor

An additional problem that was observed was the excessive loss of sludge through the separator section. This was due to not enough projected settling area. Thus, the area offered should be increased. If this were done using flat inclined plates, this would result in a bigger diameter reactor. Therefore, it was decided to try and incorporate conical settling plates concentrically installed with the cylindrical reactor. This resulted in the second lab-scale reactor concept. The technical measures and schematical representation can be seen in Figure 3.2.



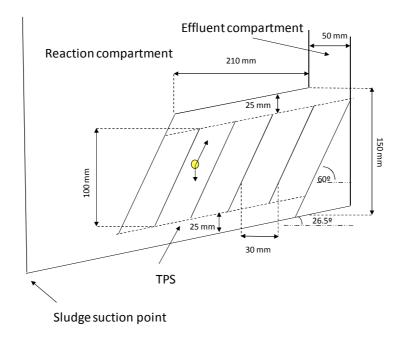
**Figure 3.2** Schematical drawings of the second cylindrical labscale IASB reactor with a conical tilted plate separator.

In the second reactor (total volume of 13 L; liquid volume of 11 L) a cylindrical downer was applied with a smaller relative cross-sectional area than the previous one only covering one ninth of the reactor cross section. The diameter was 71 mm, while the height was 200 mm. The downer was centred using adjustable screws. The TPS consisted of two concentric cones with the same inclination as in the previous reactor, but twice the settling area. This time two spray heads were included to break foam in the reactor head space

In spite of the improvements applied, the new reactor was not successful in providing appropriate mixing, retaining solids or foam breaking. Due to its design, the reactor was completely short-circuited from feed injection down to the tilted plate separator. In other words, it was a reactor only with downflow directed towards the conical separator. This suggested at least one more improvement and that was the inclusion of at least one baffle below the downer to prevent liquid from directly flowing towards the separator. Besides this, it was decided that if the concept would need to be proven, the reactor would probably be best adapted to the separator shape instead of the other way around. Therefore, a more rectangular reactor was designed with a TPS in the bottom section. Consequently, the reactor bottom was inclined as well.

## 3.4 Rectangular reactor

A rectangular reactor was designed and built with a total volume of 56 L and useful volume of 45 L. About 15 L of the useful volume was occupied by the separator. It was clearly divided into a reaction section, a separator section and an effluent section where treated wastewater was drawn off. A free settling velocity of 1.5 m/h was taken as a base, since this is a settling velocity normally encountered for activated sludge systems (Metcalf & Eddy, 2003). This resulted in a TPS with 8 inclined plates at an angle of 60° with the horizontal and each with an area of 0.029 m². The total area was 0.23 m² and the projected area was 0.12 m². The bottom below the separator was inclined at an angle of 26.5° so that sludge would slide off and could be collected at the sludge recycle suction point. Figure 3.3 shows a schematic drawing of the separator.



**Figure 3.3** TPS design of the 56 L lab reactor. Indicated are the reaction compartment above the TPS, the sludge collection & suction point and the effluent compartment.

In the reaction section two rectangular downers (Figure 3.4) were included, each with a cross sectional area of  $0.0014~\text{m}^2$ . Right under these downers a triangular baffle was put to prevent reactor short circuiting. The water level was just above the downer top and downer downflow velocities were between 4 and 12 m/h. The reactor was equipped with a spray to prevent excessive foam formation.

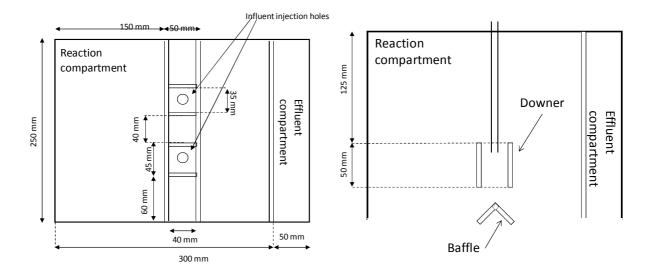


Figure 3.4 Top view (left) and side view of the downer section of the reactor.

The reactor was operated for about 4 months and was adapted according to need. Of all three reactors, this one showed best mixing conditions. No obvious short circuiting took place and sludge was kept in suspension throughout the reactor. Furthermore, sludge entrainment was noticed near the downers. It must be stated, though, that it showed to be difficult to equally divide the injection flow rate over the two downers. It was noticed that solids in the reactor compartment had a tendency to rise to the top due to biogas and/or LCFA adsorption. The importance of the spray was also obvious in controlling the foam formation.

The separator functioned reasonably well. One was able to observe sludge settling on the plates and sliding off to the bottom section. The bottom inclination, however, did not seem to be adequate, since sludge accumulated and had difficulties sliding to the recycle suction point. Additionally, the spacing between tilted plates and the bottom plate did not seem to be enough hindering the sludge from sliding off to the sludge suction point. As a consequence, the TPS started to get clogged and only a part of the plate area was used for sludge settling. This was not enough to retain sludge inside the reactor: sludge accumulation was noticed inside the effluent section. Besides the short circuiting itself, it was obvious that some of the sludge still had a tendency to float either due to LCFA adsorption, to biogas encapsulation or both. Therefore, it was decided to replace the bottom plate by a more inclined one of 45°. This partially resolved the sludge accumulation problem.

However, some sludge still floated into the effluent section and accumulated there. This resulted in the sludge lift idea. A sludge collection hood was mounted on effluent section side on the wall separating the effluent section from the reaction section. The hood was equipped with a riser tube with a diameter of 10 mm as to transport sludge and biogas to the reaction section again. The sludge was discharged above the water level. In this way, a gas lift recycle loop is prevented, since this would result in an extra hydraulic load of the TPS in the bottom section of the reactor. The sludge hood had a triangular form and side wall inclination of 15º as to direct sludge to the riser tube.

Although problems were encountered with directing the sludge towards the riser tube and although the riser tube got frequently clogged, we were able to demonstrate the principle of the sludge lift system. Sludge was effectively transported from the effluent section to the reaction section through the riser tube. In future applications, however, a riser tube with a bigger diameter and sludge collection hoods with side walls with a bigger inclination will be

required. The observational results, however, were considered good enough to continue developing this technology.

The third lab reactor showed that the IASB concept worked. To really demonstrate the technology, however, a new reactor would have to be built incorporating the knowledge gathered from the operation of the third reactor. Furthermore, to demonstrate robustness, the technology should be treated on real industrial dairy wastewater and not synthetic dairy wastewater. Therefore, instead of building another lab reactor, a proposal was submitted to obtain funds to test the reactor at pilot scale.

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### 4 Pilot test

### 4.1 Introduction

Any wastewater treatment technology that needs to be taken to industrial scale benefits from a pilot test. It gives the opportunity to simulate real industrial conditions and demonstrate the adequateness of a novel technology for its eventual purpose at a reduced cost. Furthermore, in the case adequateness is shown, data extracted from the pilot operation can be used to further optimize the technology before full-scale application. Last but not least, investment in a pilot scale test can prevent excessive investment in a first industrial scale plant, since potential process problems may be identified and prevented on industrial scale.

The IASB reactor is no exception to the rule and therefore a pilot scale test was projected. It was decided to construct a mobile pilot plant, so that the technology could be tested at several industrial locations as to show its potential for the anaerobic treatment of and biogas production from different kinds of wastewater containing fat. Therefore, this mobile plant was to incorporate only the equipment required to test the IASB reactor. It was not conceived to demonstrate a complete wastewater treatment process.

# 4.2 <u>Design</u>

Lab scale installations treating synthetic dairy wastewater (previous chapter) served as design basis for the mobile pilot plant. Furthermore, additional equipment was included so that the pilot plant could operate independently from the source of industrial wastewater. This resulted in the following basic equipment list:

- 1. A wastewater influent tank from which the reactor is fed;
- A heating circuit to heat up the wastewater and maintain the reactor at the desired operation temperature;
- 3. The IASB pilot reactor including a sludge recycle loop and a sampling loop;
- 4. A treated water tank to make reactor spraying possible using a submerged pump;
- 5. Nutrient dosing;
- 6. Caustic dosing for pH control;
- 7. Programmable logic controller (PLC) for process control.

Figure 4.1 shows the process flow diagram (PFD) of the pilot plant as it was initially proposed. For more detailed piping and instrumentation diagrams (P&IDs) please refer to Appendix?.

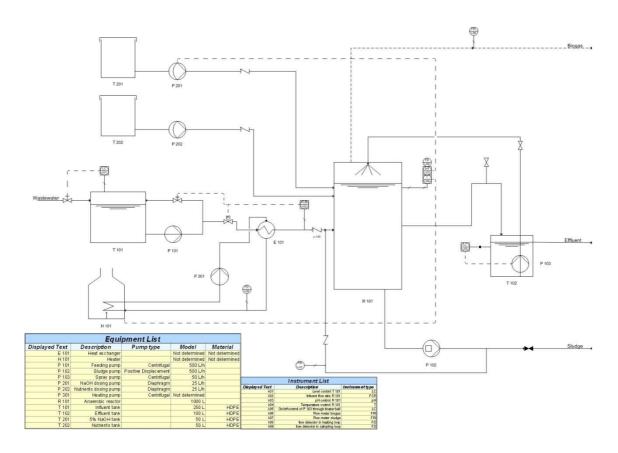


Figure 4.1 Pilot plant process flow diagram (PFD).

### 4.2.1 General process description

Industrial wastewater is collected in influent tank T101 by level control. T101 has a volume of 500 L giving the opportunity for proper reactor feed control. Feed pump P101, with a capacity of 500 L/h, is used to mix tank T101 and feed the IASB reactor R101 at the same time. The actual feed flow rate is controlled by adjusting a valve in the recycle line to the feed tank. Reactor feed is mixed with a sludge recycle taken from the bottom of reactor R101 using pump P102, thus providing intimate contact between fat laden feed and settled anaerobic sludge. Subsequently, the mixture is heated up and injected into the reactor at the top. Reactor effluent flows by gravity to effluent tank T102 in which submersible pump P103 is installed, which provides the spray as to prevent excessive foaming inside the reactor R101. Process effluent is discharged through an overflow in effluent tank T102, which has a volume of 80 L. Biogas is collected in the top of the reactor, and its flow rate and composition are measured

on-line. Reactor R101 is further equipped with a temperature measurement for temperature control, and a pH measurement for pH control. The pH can be adjusted by dosing a 4 % wt. caustic soda solution from storage tank T201 (volume of 50 L) using dosing pump P201. Finally, if required nutrients can be dosed from storage tank T202 (volume of 50 L) using dosing pump P202.

In the following sections a more detailed description of the main process unit making up the water treatment line is given.

### 4.2.1.1 Influent tank

The influent tank T101 was meant to provide the pilot plant with feed buffer capacity and the possibility to homogenize the influent and prevent peek loads. It was equipped with a pressure sensor at the bottom for level control. At a low level, the level control actuates a pneumatic valve to let fresh wastewater into the influent tank up to the wanted high level. In this way, continuous operation of the pilot plant is made possible, provided that wastewater is continuously produced as well.

The wastewater was either recycled or fed to the reactor & recycled using a peristaltic pump P101 (Ismatec, Glattbrugg, Switzerland) with a capacity of up to 500 L/h. Although the flow rate was adjustable, the maximum capacity of the pump was much more than the indicated maximum feed flow rate of 175 L/h. The reason was that the pump was also meant to provide mixing of the influent tank. Under no feeding conditions, all the water pumped by P101 is recycled over the influent tank. When reactor feeding is required, a manual needle valve in the recycle line is adjusted to get to the wanted flow rate towards the reactor. The actual feed flow rate was measured on-line.

#### 4.2.1.2 IASB Pilot reactor

The IASB pilot reactor R101 was a direct replica of the third lab reactor tested. All lessons learned from the operation of this third reactor were taken into account for the design of the pilot reactor. Thus, the reactor included:

- 1. A tilted plate separator with a bottom and top with adequate inclination;
- 2. A deflection baffle to prevent reactor short circuiting;

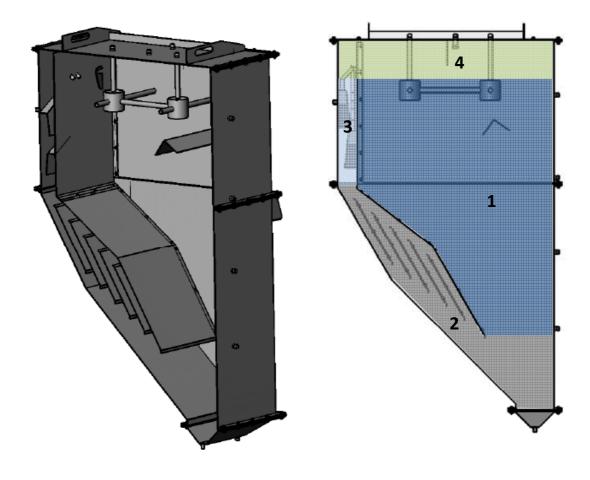
3. Two spray heads inside the reactor each capable of creating a full conical spray with an angle of 120°.

Due to the promising results of the sludge lift hood installed in the last IASB lab-scale reactor, it was decided to include two sludge collection hoods on opposite sides in the effluent compartment of the pilot IASB reactor.

Figure 4.2 shows a three-dimensional representation of the IASB pilot reactor R101. Figure 4.3 shows a photograph of the actual pilot reactor. Technical drawings of the reactor are included in Appendix ?.

The reactor was completely made out of stainless steel 316 (Metalogonde, Oliveira de Azemeis, Portugal) and thermally insulated with UV protected PE foam (Thermaflex, Oisterwijk, Netherlands). It had a total volume of 1.18 m<sup>3</sup> and was subdivided into four main compartments:

- 1. A reaction compartment of 700 L, in which polluting compounds present in the wastewater are biologically degraded;
- 2. A separation compartment of 280 L, in which settling sludge is separated from the treated wastewater;
- 3. An effluent compartment of 35 L, in which the treated water leaves the reactor an two sludge collection hoods are installed;
- 4. The head space of 165 L, where the biogas is collected and the anti-foam sprays are installed.



**Figure 4.2** Three dimensional drawing and compartmentalization of the IASB pilot reactor: 1 - reaction compartment; 2 - separation compartment; 3 - effluent compartment; 4 - head space.

#### 4.2.1.2.1 The reaction compartment & head space

This is the reactor's main compartment and most of the anaerobic sludge is present here either as suspended sludge or as part of a floating fatty scum layer at the water surface. Two cylindrical downers, each with a diameter and a length of 100 mm, were installed in the top section of the reactor compartment into which raw wastewater mixed with sludge recycled from the bottom of the reactor was injected. The average downer downflow rate was controlled between 4 and 8 m/h to create a mildly mixed environment under the floating sludge layer. A deflection baffle was installed beneath the downer above the sludge settling zone to prevent short circuiting of the reactor and direct the flow towards the sides. Due to the presence of fat in the wastewater a scum layer will be formed on the water surface. Excessive scum formation was counteracted by spraying treated wastewater through two maximum free passage full cone sprays (Bete Fog Nozzle Inc., Greenfield, MA, USA) installed in

the reactor headspace. They sprayed the water at an angle of 120° so that a maximum scum layer surface is covered.



Figure 4.3 Mobile pilot plant including reactor in the forefront.

The minimum envisioned hydraulic retention time in this compartment was of 4 hours. Therefore, the maximum allowable net flow rate through this compartment was of 175 L/h.

For proper reactor pH control, a sample loop was installed through which water from the reaction compartment was continuously recycled using a membrane pump. In this sampling loop a pH probe was installed, by which the caustic dosing using diaphragm pump P201 was actuated. Furthermore, the sampling loop was equipped with a flow detection device. The reactor temperature was directly measured in the reactor compartment with a Pt temperature sensor for adequate temperature control. Heating was achieved with four direct heating devices (Paralab, Gondomar, Portugal) with internal heating coils. To be able to maintain the required reactor temperature even when the reactor was not fed, the mixture of sludge and raw wastewater was heated. To prevent excessive heating, *i.e.* temperatures above 40°C, one of the heating devices was equipped with a temperature sensor.

Besides caustic dosing, nutrients may be dosed. The composition of the nutrient solutions depends on the wastewater composition. As to create optimum conditions, at least a micronutrient solution should be dosed.

The biogas produced in the reaction compartment was collected in the headspace and leaves the reactor through the biogas exit point. The biogas flow rate was measured on-line, as well as the percentages of methane and carbon dioxide. After that, the biogas was discharged to the atmosphere.

#### 4.2.1.2.2 The separation compartment

In the separation compartment suspended anaerobic sludge is separated from treated wastewater and collected in the bottom of the reactor. For the design, considerations for TPS design as described in Clarifier design,  $2^{nd}$  Edition (WEF, 2005) were taken into account. The total maximum flow rate  $Q_f$  passing the TPS is the sum of the spray flow rate (500 L/h; see paragraph on the effluent tank in this section) and the feed flow rate (175 L/h at 4 h HRT in the reaction section). 0.8 m/h was taken as the design free settling velocity  $u_s$ . The TPS plates were set at an angle  $\alpha$  of 60° with the horizontal and the perpendicular distance d between the plates was 45 mm. Using equations (2.2) and (2.5) the total required plate area was calculated at 1.68 m². This was divided over a total of six inclined plates, each with a length L of 0.40 m. The TPS bottom was set at an angle of 45° so that sludge separated could further settle to the bottom of the reactor, from which it could either be recirculated and mixed up with raw wastewater, or be discharged. A peristaltic pump P102 (Ismatec, Glattbrugg, Switzerland) was used for sludge recycling. Sludge recycle flow rates applied varied from 50 to 150 L/h. The flow rate was measured using an on-line flow measurement device.

#### 4.2.1.2.3 The effluent compartment

After sludge separation, the water enters the effluent compartment from which the water is discharged through two exit points below the water surface. The actual water level in the reactor was set by applying siphon breakers right after these water exit points. The effluent compartment was further equipped with two sludge collection hoods on opposite sides as to collect sludge that managed to pass through the TPS. Each collection hood has a riser tube through which collected sludge was transported back to the reaction compartment by gas lift action induced by biogas. The sides of the collection hoods had an angle of 30° to facilitate the transport of sludge to the riser tubes.

### 4.2.1.3 Effluent tank

The treated wastewater flowed by gravity from the reactor to effluent tank T102 in which a submersible pump P103 with a float actuator was installed. The pump had a capacity of 500 L/h and recirculated treated water to the two spray nozzles. The effluent tank was equipped with an overflow through which the treated wastewater was discharged.

# 4.3 Pilot operation

#### 4.3.1 Introduction

Since most of the research on the anaerobic treatment of fat containing wastewater within the LEB had been done with synthetic dairy wastewater (Pereira, 2003; Cavaleiro et al., 2009), it was only logical to want to test the IASB technology on actual dairy wastewater at a dairy factory. Although the pilot plant has a basic layout that makes it possible for it to be operated at any industrial location, the pilot plant was designed and constructed to be first operated at a dairy factory. After this first test it could be adapted and tested with a different kind of wastewater. Due to unforeseen circumstances, however, the pilot operation at the envisioned industrial site was not possible and it needed to be relocated and operated at a slaughterhouse: the Regional Slaughterhouse of Barroso and Alto Tâmega (BAT), Montalegre, Portugal (Matadouro Regional de Barroso e Alto Tâmega). This required last minute changes to the pilot plant layout as to make it apt for slaughterhouse wastewater treatment.

Nevertheless, this change of plans gave the opportunity to test the robustness of the IASB concept on arguably the most polluting kind of wastewater produced in the food industry. Slaughterhouse wastewater is known to be highly concentrated and, if discharged as such into the environment, to potentially cause irreversible damage to natural habitats (see Introduction). Depending on the production process and the animals slaughtered, it may contain fat, protein, gut content and blood, resulting in a very potent cocktail of organic pollutants. Therefore, if it were demonstrated that the IASB reactor can directly treat this kind of wastewater and even convert additional fat into biogas, the road to treating wastewater from other food industries such as dairy should be cleared.

At the BAT slaughterhouse mainly cattle are slaughtered. The production process is energy intensive because of the high consumption of hot water used for hygienisation. Besides this, the wastewater generated, which contains fat, blood and gut contents, is currently treated in an aerobic treatment plant. Due to the high organic loading rate associated to the wastewater, this aerobic treatment also requires a high energy input in the form of aeration. Additionally, one of their by-products is animal fat, which is currently transported off-site without any benefits associated to it for the slaughterhouse. Taking these three points into account, an eventual anaerobic treatment process capable of directly treating the BAT slaughterhouse wastewater generated at this facility could result in three advantages:

- 1. Anaerobic treatment of the wastewater could potentially result in a reduction in organic loading of 80 to 90 %, thus relieving the aerobic treatment plant and considerably reducing energy consumption associated to aeration;
- Since the IASB reactor is a reactor capable of directly treating fat containing wastewater, the opportunity arises to even add more fat so as to maximize the biogas production. This way beneficial use can be made of the waste fat generated at the slaughterhouse;
- 3. The biogas produced in the IASB reactor can be used for hot water production, thus reducing the energy bill associated to it.

Besides showing the robustness of the IASB technology for the anaerobic treatment of fat containing wastewater, the optimization of the technology and the identification of potential process problems a fourth objective can be defined for this pilot operation: biogas production maximization by adding waste fat generated in the slaughterhouse production process.

### 4.3.2 Theoretical part: Assumptions & calculations

To facilitate the daily pilot plant operation a calculation sheet was set up to which all analytical data served as input, so that theoretical assumptions could be compared with practical experience. As such, a mean biomass composition (CH<sub>1.8</sub>O<sub>0.8</sub>N<sub>0.2</sub>P<sub>0.05</sub> (reference)) was assumed. In anaerobic treatment practice, it is normally assumed that about 10 % of COD converted goes to biomass (Mulder, 2003). In this case a higher percentage of COD converted was allocated for biomass growth (20 % of converted COD goes to biomass). Although Pereira (2003) indicated that COD requirements for growth in anaerobic systems treating LCFA are less than in conventional systems, this high COD percentage was assumed for biomass growth, since adaptation would be one of the crucial points in pilot success. For this a complete "make-

over" of the microbial population was possibly necessary. The assumed biomass composition and COD needed for growth right away determine the macro-nutrient requirements during pilot operation.

To try and characterise the IASB reactor in hydrodynamic terms, some characteristic times were defined for subsequent correlation with process parameters measured along the pilot trial. Besides the hydraulic retention time (HRT), the following characteristic times were defined:

The mixing time  $t_m$  (h) inside the reaction compartment, which is the average time needed to transport the reaction compartment liquid volume from the top to the bottom of the RC. It is equal to the Reaction Compartment volume divided by all the main liquid flows entering and leaving this compartment.

$$t_m = \frac{V_{RC}}{(\phi_{feed} + \phi_{underflow} + \phi_{spray})} \tag{4.1}$$

In which:

 $V_{RC}$  = volume of the reaction compartment (m<sup>3</sup>);

 $\Phi_{\text{feed}}$  = feed flow rate (m<sup>3</sup>/h);

 $\Phi_{\text{underflow}}$  = reactor underflow flow rate (m<sup>3</sup>/h);

 $\Phi_{\text{spray}} = \text{spray flow rate (m}^3/\text{h)};$ 

Since the nutrient and caustic dosing flow rates were very low compared to the other three, they were neglected in these calculations.

The time to effluent tank  $t_{et}$  (h), which is the average time needed to transport the total liquid reactor volume from the top of the RC, over the separator to the effluent tank. It comes in handy to evaluate the separator performance. It is equal to the total liquid reactor volume divided by the flow rates that enter the RC from the top and leave the reactor through the effluent compartment.

$$t_{et} = \frac{V_R}{(\phi_{feed} + \phi_{spray})} \tag{4.2}$$

In which  $V_{\mbox{\scriptsize R}}$  is the total liquid reactor volume.

### 4.3.3 Experimental part: Daily operation and analysis

The pilot plant was equipped with a control system programmed in Labview (National Instruments, Austin, TX, USA). Although it is tempting to automate all of the process, a pilot test surely benefits from minimizing automated process control, since it assures the proper attention from the operator. Therefore, it was decided to keep automated process control only to the essential parameters, *i.e.* pH and temperature. Besides these, only the influent tank level was linked to the opening or closing of a pneumatic valve. Flow rates were not automatically adjusted, but needed to be set manually. Other pneumatic valves could only be opened or closed manually. For the rest, the control system mainly served for logging of process parameters, such as reactor pH & temperature, influent & sludge recycle flow rate, biogas flow rate and biogas composition. An internet connection was available for remote access.

The IASB reactor was operated at a temperature between 30 and 35 °C. The pH was kept above 7 to prevent any possible inhibition from undissociated VFA. To create the best growing conditions, micro-nutrient solution was dosed at a rate of 1 mL/L of feed. This solution had the composition as reported by Pereira (2003). Due to the complexity of the wastewater, in general an increase in organic loading rate was only considered if the soluble COD in the reactor effluent was below 1,000 mg COD/L. However, more than one increase a week was never allowed.

Besides actual pilot operation, daily activities could be divided into two main sections: problem solving and data collection. Although data collection is highly important, previous personal experience has shown that you cannot assure it without a properly running process. Even the most careful planning and design will not make it possible to avoid putting perseverance and creativity at work as to assure continuity. Therefore, problem solving probably is one of the most crucial activities to the success of a pilot operation to demonstrate robustness; specifically when operating with a complex wastewater such as slaughterhouse wastewater.

To further assure proper pilot operation and optimize problem solving, checklists were set up for the operator for daily process parameter verification and visual checks of all process equipment and instruments. The checklists used are compiled in Appendix ?.

Table 4.1 shows the pilot plant sampling scheme. All samples taken were grab samples. The sampling frequency was adjusted as required for proper process control and characterisation. Analyses were divided into those done at the pilot plant and those done at the Laboratory for Environmental Biotechnology (LEB), University of Minho, Braga. At the pilot plant a mini-lab was set-up including an Eppendorf centrifuge (Braun Biotech International, Melsungen, Germany), a spectrophotometer (Hach-Lange, Düsseldorf, Germany) and thermal digestion block (Hach-Lange, Düsseldorf, Germany). Analyses done at the pilot were total & colloidal/soluble COD, total N, sulphate and phosphate using Hach-Lange Cuvette tests. Furthermore, Imhoff settling tests were done and the pH was measured with a portable pH measurement device (WTW, Weilheim, Germany). At the University ammonia, volatile fatty acids (VFA), long chain fatty acids (LCFA), total suspended solids and volatile suspended solids were analysed for. Ammonia was determined using the Hach-Lange system. The VFA determined were formic, acetic, propionic, n-butyric, iso-butyric and valeric acid. All were analysed by GC as described by Cavaleiro et al. (2008). The LCFA determined were stearic, linoleic, oleic, palmitoleic, palmitic, myristic and lauric acid and were analysed as previously described (Neves et al., 2009).

Table 4.1 Sampling points and analyses.

	рН	C	OD	VFA	Sol	ids	Imhoff	LCFA	N		Р	SO <sub>4</sub> <sup>2-</sup>	CH₄	CO <sub>2</sub>
		Total	Dissolved		TSS	VSS			Total	$NH_4^{\dagger}$				
Influent	х	х	х	х		х	х	х	х	х	х	х		
Reactor	Х	х	х		Х	х	х	Х						
Underflow					х	х	х	Х						
Effluent	Х	х	х	х	Х	х	х	X	x	X	Х	Х		
Gas													Х	Х

### 4.3.4 Results: General operation

The pilot plant was operated from April 2009 until May 2010 for a period of 375 days. The operation period can be divided into four main phases:

- Phase 1. Start-up (190 days), during which the pilot reactor was inoculated several times with anaerobic sludge from several sources and adapted to the slaughterhouse wastewater;
- Phase 2. Activity build-up (92 days) by feeding only wastewater;

- Phase 3. Organic load increase by adding raw waste fat (63 days);
- Phase 4. Organic shock loading with fat (34 days).

These separate phases will be dealt with further on.

During the operation the pilot plant had to be adapted several times (mostly during Phase 1) to be able to deal with the slaughterhouse wastewater. Specifically tube clogging was problematic due to the presence of coarse solids, *e.g.* lumped fat or pieces of tripe dragged along with the wastewater. The main adaptation was the different set-up of the reactor feeding system at the beginning of the Phase 1. Instead of using peristaltic pump P101 for reactor feeding, it had to be used for pumping wastewater into influent tank T101 feeding. Consequently, an extra pump needed to be included for reactor feeding. For this purpose peristaltic pump P104 (Watson-Marlow, Cornwall, GB) was installed with a capacity of 80 L/h. A consequence was that the mixing of T101 had to be sacrificed. Figure 4.4 shows the adapted PFD for the treatment of slaughterhouse wastewater and fat.

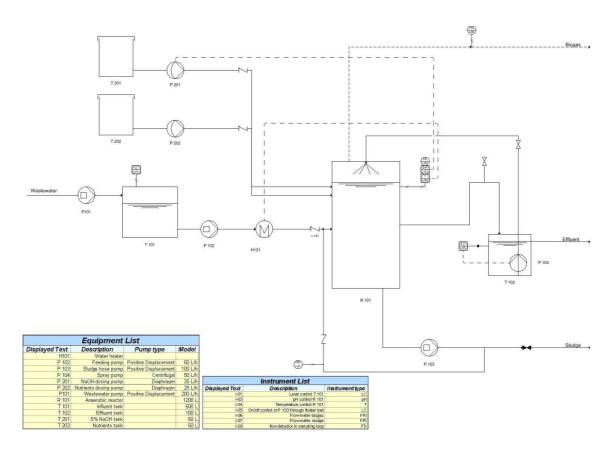


Figure 4.4 Adapted PFD for slaughterhouse wastewater treatment (drawing needs to be set up).

#### 4.3.4.1 Phase 1: Start-up

The start-up phase lasted for 190 days. During this phase the reactor was inoculated 4 times, of which the first one was done on day 0 with 75 L of anaerobic sludge from an activated sludge digester located at Freixo municipal wastewater treatment plant (Porto, Portugal). On day 44 the reactor was inoculated with 15 L of lab sludge adapted to synthetic dairy wastewater (Cavaleiro et al., 2009). On day 92, 20 L of Freixo sludge were added. On days 105 and 162, 20 L and 5 L of granular sludge from an anaerobic reactor treating brewery wastewater (WWTP at Sociedade Central de Cervejas e Bebidas, Vialonga, Portugal) were added, respectively. This sludge showed a high methanogenic activity on brewery wastewater and was specifically added to increase the population of methanogenic micro-organisms.

Phase 1 was further characterized by 3 periods of batch operation to give the sludge the chance to adapt to the wastewater. These periods lasted for 20, 50 and 20 days, respectively. These periods were interceded by two periods of 50 days in which continuous operation was applied to see where activity build-up was already possible. Both periods of continuous feeding were terminated (days 70 and 170, respectively) because the dissolved effluent COD was above 1,000 mg/L and showed a tendency to increase.

On day 147 a mechanical biogas counter was installed (Sinagawa, Tokyo, Japan) due to the fact that the on-line digital measurement was not properly functioning. From day 182 on additional phosphate was provided so that growth limitation due to lack of phosphate could be ruled out.

#### 4.3.4.2 Phase 2: Activity build-up

During this phase, which lasted 92 days, the organic loading rate was increased by increasing the wastewater feeding flow rate until 4 kg COD/m³/day were reached. Due to the high variability in wastewater composition and influent line clogging problems, it showed to be a tough task to maintain constant loading rates. When the soluble COD concentration in the reactor effluent stayed below 1,000 mg/L for at least a week, the loading rate was increased with 0.5 kg/m³/day.

From day 203 on (13 days into Phase 2), intermittent spraying was initiated. It was thought to have a beneficial effect on the activity build-up.

#### 4.3.4.3 Phase 3: Start of raw fat addition

Big chunks of raw fat were directly collected at the slaughterhouse and grated as to increase the specific surface area. The fat loading rate was increased once a week until reaching the desired total loading rate of 8 kg COD/m³/day.

#### 4.3.4.4 Phase 4: Shock loading with fat

The IASB reactor was shock loaded with fat for a period of 12 days with loading rates going up to 16 kg COD/m3/day. Subsequently, the recovery of the reactor was monitored for another 22 days after which the pilot test was ended.

### 4.3.5 Results: Wastewater & fat characteristics

The pilot plant feed wastewater was collected on the days that slaughtering took place (normally 3 days/week). The wastewater quality and quantity varied considerably along the day. At certain times it would contain more coarse solids, other times it would be mostly diluted blood or contain more fat. It was always attempted to pump wastewater into the feeding tank during the whole production day as to get the most representative wastewater composition. However, due to clogging problems this was not always possible. Consequently a highly variable wastewater quality was obtained with the consequence associated that the wastewater feed flow rate needed to be adjusted accordingly to try and keep the organics loading rate as constant as possible.

Table 4.2 shows mean concentrations of the raw wastewater along the pilot trial of the main analytical parameters. It showed some similarity with the slaughterhouse wastewater composition, although pre-treated, reported by Mijaylova Nacheva et al. (20?), specifically concerning the concentration of inorganic species. The table further shows that especially the solids concentration was tough to control. Although TSS concentrations during this pilot test were considerably higher, the VSS/TSS ratio was comparable to the one reported by Ruiz et al. (1997) at a value of about 90%. The pH was relatively constant and slightly acidic. Dissolved COD appeared to be one of the more stable parameters and formed about 30 % of the total COD. Of the macro-nutrients phosphorus was considered to be possibly limiting. Therefore, phosphate dosing was started at the end of Phase 1 to keep the reactor effluent P concentration always above 30 mg/L. About one third of the nitrogen was present as ammonium. Due to the origin of the wastewater, it is thought unlikely that nitrates were

present in the wastewater. It is more plausible that the rest of the nitrogen was present as protein. An indication of this might be obtained by observing an ammonium concentration increase over the treatment system, since ammonium is one of the products of protein hydrolysis.

The wastewater contained a considerable concentration of sulphate, which will result in some loss of biogas production potential due to the biological production of hydrogen sulphide ( $H_2S$ ). As a rule of thumb (Mulder, 2003), the undissociated hydrogen sulphide concentration should never exceed a concentration of 100 mg/L, so that inhibition of methanogenesis is prevented. Around the pH at which the reactor was operated (between 7.0 and 7.5), undissociated  $H_2S$  can form between 25 and 50 % of the total sulphide concentration. In the worst case (pH = 7.0) total sulphide concentrations of up to 200 mg/L were allowed. Therefore, potential inhibition of methanogenic organisms was considered under control.

**Table 4.2** Mean concentrations (± standard deviation) of the raw wastewater.

Parameter	Unit	Mean value (±SD)
рН	-	6.6 (±0.4)
COD	mg COD/L	13426 (±7754)
Dissolved COD	mg COD/L	4148 (±2077)
Total N	mg N/L	565 (±354)
NH <sub>4</sub> <sup>+</sup>	mg N/L	176 (±121)
TSS	mg/L	2917 (±2617)
VSS	mg/L	2601 (±2476)
Total P	mg P/L	35 (±18)
SO <sub>4</sub> <sup>2-</sup>	mg S/L	111 (±88)

To give an impression of the variability of the wastewater quality, Figure 4.5 shows the total, dissolved and LCFA-COD of the raw wastewater as collected in the influent tank. The total COD concentration fluctuated between 1 and 44 g/L. The dissolved COD was more constant and mostly remained below 5 g/L, indicating that there is a firm base of more easily accessible organic COD. As can be seen in Figure 4.5 fat was present in the raw wastewater. The LCFA-COD reached highest values during Phase 1 (up to concentrations as high as 13.3 g COD/L), but remained below 2 g COD/L during subsequent phases.

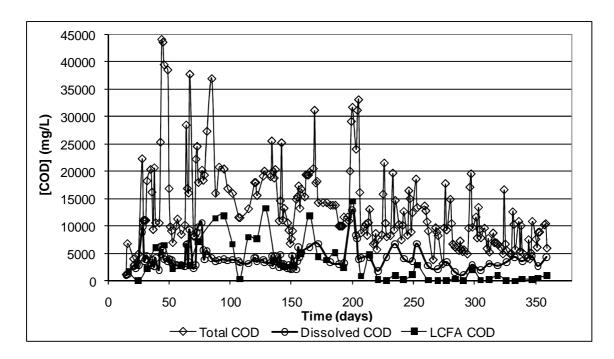


Figure 4.5 Total, dissolved and LCFA COD concentrations of the raw wastewater present in the influent tank.

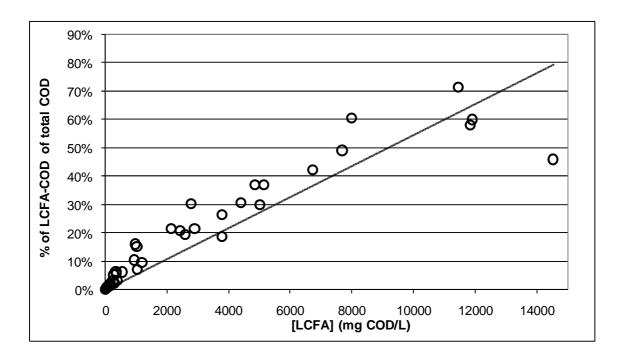
Table 4.3 lists the mean composition of the LCFAs present in the wastewater in weight terms and COD terms. Clearly, more than 90 % of the LCFAs were present as palmitic, stearic and oleic acid. Therefore, it may be concluded that most of the LCFAs were saturated. This is in contrast with the synthetic dairy wastewater used in lab scale experiments, in which mostly oleic acid was present (Pereira, 2003). Hwu et al. (1998b) used a mixture of oleic (50%), stearic (15%) and palmitic (35%) acid to simulate the genuine wastewater composition of a local slaughterhouse. Jeganathan et al. (2006) gave a qualitative analysis of FOG present in pet food wastewater comparable to the composition reported by Hwu et al (1998b). It is obvious that both strongly contrast with the composition reported in this thesis. Practically all solid state COD present in the wastewater was in the form of LCFA-COD (close to 85 %). The rest of the solid state COD was presumably present as protein.

**Table 4.3** Mean LCFA composition in terms of weight and COD % of total LCFA concentrations in the wastewater.

Long Chain Fatty Acid	Wt. %	COD %
Linoleic acid	1.5	1.4
Oleic acid	7.6	7.6
Stearic acid	34.9	35.4
Palmitoleic acid	0.5	0.5

Palmitic acid	49.9	49.7
Myristic acid	4.0	3.9
Lauric acid	1.6	1.6

The LCFA concentration showed to have a strong influence on the total COD concentration; this is shown in Figure 4.6. As can be seen, the contribution of LCFAs to the total COD linearly increased with increasing LCFA concentration. A baseline COD concentration could be calculated at which the LCFAs did not contribute to the COD concentration. This is the interception of a linear fit of the total COD concentration versus the LCFA COD concentration and has a value of 8200 mg COD/L. This base line COD concentration can be further subdivided into soluble and insoluble COD, which were calculated at 5000 mg/L and 3200 mg/L, respectively. Above this baseline concentration, the total COD practically only increases with an increase in solids concentration and therefore LCFAs. Although these concentrations should only be considered as an indication, the measured COD concentrations showed to fluctuate around these values during Phase 2 through 4.

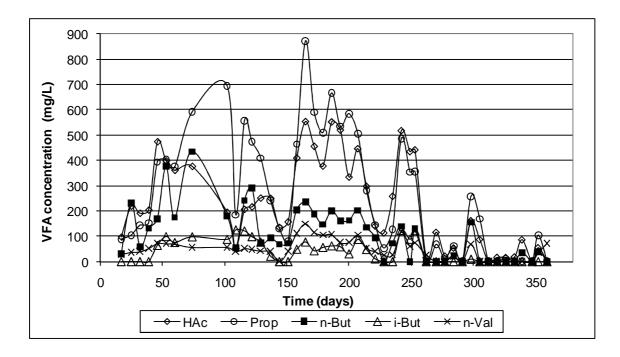


**Figure 4.6** Percentage of LCFA-COD of total COD as a function of the total LCFA concentration present in the wastewater.

The suspended solids present in the wastewater were mainly organic and therefore, in principle, susceptible to biological degradation. The calculated VSS-COD from Table 4.3 was

3.18 g COD/g VSS, which is very high. The average VSS content of TSS, however, was "only" 85 %, thus suggesting that possibly very long chain fatty acids (VLCFA) were present.

As previously indicated, the raw wastewater dissolved COD concentration was relatively constant; its composition, however, appeared to be dependent on the hydraulic retention time of raw wastewater in the influent tank. Figure 4.7 shows the influent VFA concentrations. Acetic, propionic and butyric acid were most prominent, and specifically when influent HRT was bigger. Thus, continuous operation at low reactor loading rates and batch operation apparently resulted in a more acidified wastewater (VFA-COD/total COD x 100%) than at higher loading rates and higher degrees of acidification during Phase 1. Lumped VFA concentrations of up to 25 meq/L were attained. During subsequent phases acidification gradually decreased with increasing loading rate until stabilizing below 5 meq/L. VFA only formed a minor fraction of dissolved COD, thus leaving a gap to be filled in by other types of organic compounds, such as proteins.



**Figure 4.7** VFA concentrations in raw wastewater. HAc = acetic acid; Prop = propionic acid; n-But = n-butyric acid; i-But = *iso*-butyric acid; n-Val = valeric acid.

The raw fat added to the reactor in Phase 4 came directly from the slaughtering facility and was white with a pink shine to it, most probably due to blood inclusion and muscular tissue still attached to the fat. Furthermore, a kind of organic membrane could be noticed wrapped

around smaller chunks of fat. The fat was grated before adding it to the reactor. Figure 4.8 shows a photograph of the grated fat. It showed to be difficult to measure the COD content of this fat. The classic COD titration method (APHA et al. 1998) gave the most satisfying results and this was used for further calculation of organic loading rates. The measured value was 1.9 g COD/g raw fat. This value is considerably lower than the COD associated to pure LCFAs, such as oleic and palmitic acid. This was most probably due to presence of other organic compounds and water. Thus more oxygen is incorporated in raw fat. Further on, however, for ease of calculation it was assumed that all raw fat COD was present as LCFA.



 $\textbf{Figure 4.8} \ \textbf{Photograph of the grated fat before addition to the reactor}.$ 

## 4.3.6 Results: Process Performance

Overall process performance was satisfactory. The most difficult part of the process operation showed to be providing enough wastewater to the system. Many times tubing needed to be unclogged and many times reactor feeding stopped overnight, due to chunks of fat or other organic material getting stuck. Thus, besides the strongly fluctuating concentrations in the raw wastewater, organic loading rates were also influenced by clogging of the influent system. These problems mainly occurred during start-up and further details will be spared. For actual process performance evaluation only Phases 2 to 4 were taken into account.

### 4.3.6.1 *COD* loading

During Phases 2 through 4 the organic loading rate was gradually increased. Most of the organic load was provided by solid state COD. Soluble COD only had a minor contribution; specifically during Phase 3 and 4. At first it was attempted to apply a continuous load, *i.e.* 7 days a week. However, eventually it was decided not to continue like this because of practical reasons and feeding was stopped during weekends during Phases 3 and 4. Loads during weekend and holidays could not be guaranteed because of the likeliness of clogging while no one being around. Additionally, from Phase 3 on, raw fat was added and the weekends were ideal for the anaerobic sludge to adapt to the increasing loading rates.

Figure 4.9 shows the organic loading rates (OLR) applied and effluent COD concentrations during Phase 2; Figure 4.10 shows these data for Phases 3 and 4. Loading rates went from 1 to 4 kg COD/m³/day during Phase 2. Total effluent COD concentrations fluctuated between 1500 and 3000 mg/L, while soluble and colloidal COD stayed mostly below 1000 mg/L. During Phases 3 and 4 the VLR was increased up to 16 kg COD/m³/day by adding fat. Total effluent COD and soluble & colloidal COD concentrations basically showed the same tendency as during Phase 2. As can be seen in both Figures 4.9 and 4.10, the OLR showed strong variations due to reasons previously indicated. In spite of this, the COD removal efficiency mostly remained between 70 and 90 %, showing a performance comparable with conventional anaerobic technology (Mulder, 2003).

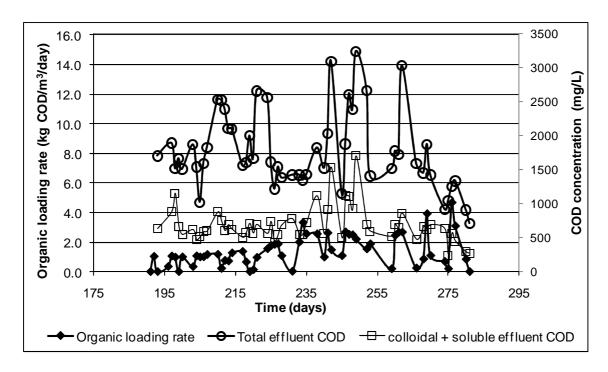


Figure 4.9 Organic loading rate (kg COD/m³/day) and effluent COD concentrations (mg/L) during Phase 2.

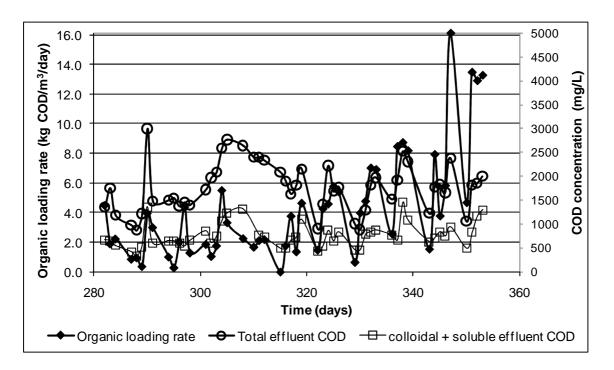


Figure 4.10 Organic loading rate (kg COD/m³/day) and effluent COD concentrations (mg/L) during Phases 3 & 4.

Fat formed a big part of the COD loading rate, specifically at the end of the pilot trial. Although the fat loading rate was continuously increased during Phase 3 and 4 by adding raw fat, the effluent LCFA load stayed remarkably stable below 500 g COD/day. Even at a loading rate of 6

kg of LCFA COD/day (overload situation), the effluent load stayed below 500 g COD/day (Figure 4.11). This removal efficiency was more due to retention of fat inside the reactor because of its lower density than water. It did not result in a direct increase in biogas production due to biological degradation.

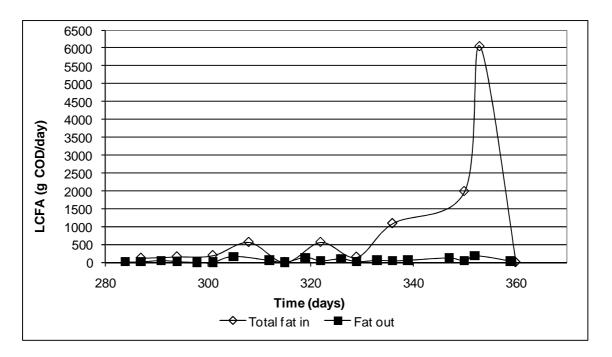
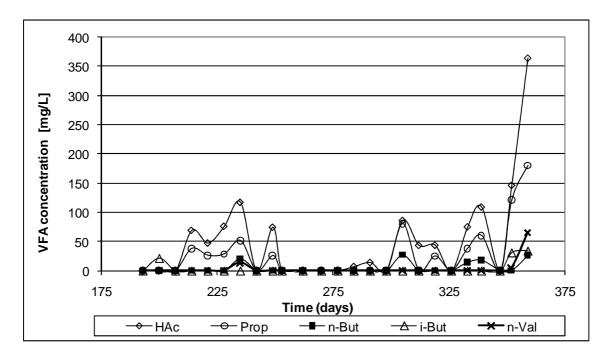


Figure 4.11 Total fat load going into the IASB reactor and total fat load leaving the reactor during Phases 3 and 4.

Although not specifically analysed for, it is highly likely that a part of the COD was present in the form of protein. This was partially confirmed by the fact that there was an increase in ammonium concentration over the treatment system. Overall, the mean ammonium concentration increased from 130 mg N-NH<sub>4</sub><sup>+</sup>/L to 200 mg N-NH<sub>4</sub><sup>+</sup>/L. This was accompanied by a decrease in total N concentration from 680 mg N/L to 640 mg N/L. Thus, at least partial protein degradation seemed to take place in the reactor. Besides that, the total N decrease over the reactor indicated that N was apparently incorporated in biomass and therefore growth took place. It must be stated, though, that some care must be taken, since feed and effluent samples on same days were compared. There was a time lag between these concentrations because of the reactor HRT. Nevertheless, the N concentration trend indicated that protein was being degraded and nitrogen was incorporated in the biomass.

VFA never reached high values until Phase 4 when an overload of fat was applied (Figure 4.12). Acetate occasionally would reach values over 100 mg/L. Propionate and n-butyrate were the

only other VFA to reach values close to 80 mg/L. According to Massé et al. (2001) this may be considered an indication of reactor overloading and this would have occurred in the beginning of Phase 2 and about 10 days after raw fat addition was started (day 292). The reactor, however, always showed a tendency to recover. Furthermore, the lumped VFA concentration (expressed in meq of HAc/L) always stayed below 5 meq/L. In industrial wastewater treatment practice this is considered a very good performance (personal experience). Only after applying an overload of fat in Phase 4, acetate reached a value 364 mg/L and propionate reached 180 mg/L. At that time, the lumped VFA concentration reached 10 meg/L.



**Figure 4.12** Effluent VFA concentration during Phases 2 through 4; HAc = acetic acid; Prop = propionic acid; n-But = n-butyric acid; i-But = *iso*-butyric acid; n-Val = valeric acid.

### 4.3.6.2 Biogas production

Biogas quality was occasionally verified by GC and showed to be stable around 70 %  $CH_4$  and 30 %  $CO_2$ . The on-line  $CH_4$  and  $CO_2$  measurements could only be considered as trend indications, since they normally showed readings of which the sum resulted in percentages above 100 %.

The measured biogas production did not reflect the theoretical one. Although a gradual increase was noticed in the biogas production measured, it never attained the theoretically attainable production. Figure 4.13 shows the measured biogas production through Phases 2, 3 and 4. Maximum biogas rate measured was 25 nL/h during Phase 3. During Phase 4 a fat

overload was applied on day 347. At first, this did not seem to affect biogas production until day 352. From that day on, the biogas production rapidly decreased.

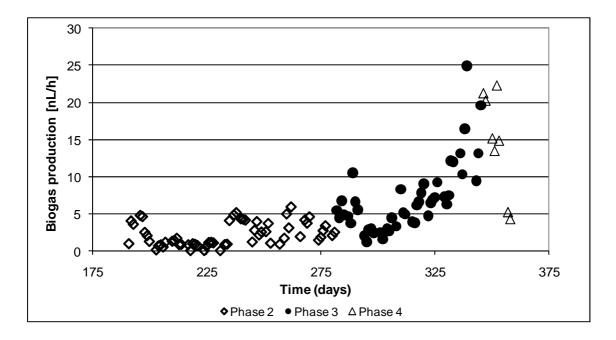


Figure 4.13 Measured biogas production during Phases 2, 3 and 4.

Overall, the measured biogas production was about 20 % of the theoretical biogas production. This is comparable to the low methane conversion efficiencies reported by Hwu et al. (1998a). In spite of reasonable COD removal efficiencies of up to 70%, they only achieved 7-15% methane conversion efficiencies. They attributed the low methane production rates to LCFA adsorption and inhibition. Hwu et al. (1998b) proposed a model for LCFA adsorption and degradation which included 5 phases. In phase 4 of this model about 60% of and LCFA mixture is adsorbed onto biomass, while only about 25 % is converted to methane. Possibly during the pilot test, the process never got passed phase 4 of the proposed model. This might have been due to the strong loading rates applied during operational Phases 3 and 4, when raw fat was added. It must be stated, though, that there were no indications of inhibition of the process. Dissolved COD and VFA concentrations remained low until the reactor was overloaded with raw fat. Only then a sharp increase in VFA concentration was noticed.

### 4.3.6.3 Sludge growth and nutrients

During operation it was made sure that nutrients were never lacking, thus making it possible for bacteria to grow under optimised conditions. Nitrogen was always present in sufficient amounts in the wastewater, while additional P and micro-nutrients were dosed. Taking assumptions earlier indicated into account, the calculated sludge growth was always higher than volatile suspended solids wash-out. Over the reactor system the sludge ash content increased about 7 % overall from 10 % to 83 %. It appeared that inorganic solids were successfully incorporated in the sludge increasing its sedimentation capacity.

Figure 4.15 shows the TSS and VSS concentrations in the effluent. As can be seen, they mostly stayed below 1,000 mg/L and mostly close to 500 mg/L. Effluent solids concentrations measured were comparable with a recently commissioned full-scale conventional anaerobic reactor treating brewery wastewater (Central de Cervejas, Vialonga, Portugal). There the TSS concentrations fluctuated between 200 & 900 mg/L. At the pilot plant, effluent concentrations only increased at the end of Phase 4 due to spray operation in continuous mode: this resulted in a hydraulic overload of the TPS leading to solids washout. Further details on TSS and VSS retention efficiencies will be dealt with in the section on reactor performance.

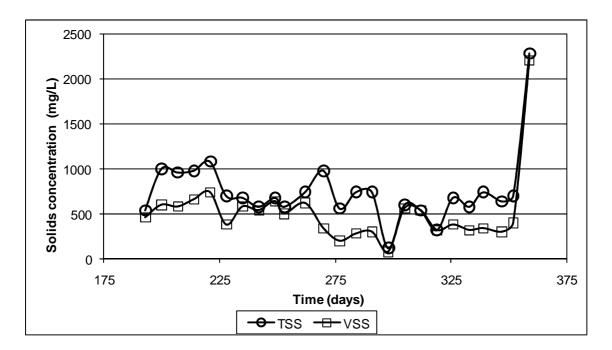


Figure 4.14 Treatment TSS and VSS effluent concentrations.

### 4.3.7 Results: Reactor Performance

Overall the reactor performed well. The reactor was very capable of retaining biomass at high loading rates and specifically high fat loading rates. The biggest problem seemed to be biogas liberation inside the reactor. The hydraulic regimes that could be applied apparently were not sufficiently turbulent enough to assure biogas liberation inside the reactor. Below follows a more in-depth analysis of reactor performance by looking at all parameters measured and/or defined.

#### 4.3.7.1 COD mass balance

Specifically due to the difference between theoretical and measured biogas production, there was a big gap in the COD balance. Figure 4.16 shows a cumulative COD graph including all COD going into and leaving the reactor during Phases 2 through 4. At the end of the test the gap between ingoing and outgoing COD had increased up to 150,000 g. This would mean that the mean COD concentration inside the reactor compartment would have gone up to close 220 g/L. Through visual inspection of the reactor contents this could be readily excluded. It seems therefore obvious that the gap was due to biogas unaccounted for.

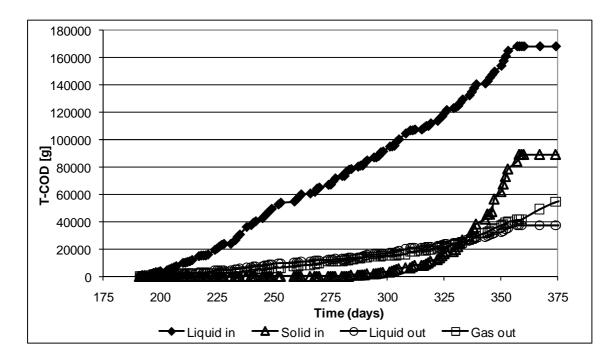


Figure 4.15 Cumulative COD going into and leaving the reactor.

The COD removal efficiency was calculated based on daily measurements and based on the cumulative COD balance over the reactor. Based on the first, one obtains a graph with efficiencies roughly fluctuating between 70 and 90 %. This is in good agreement with efficiencies obtained at industrial scale. When looking at the cumulative efficiency, fluctuations level out and the efficiency shows to be quite stable between 80 and 85 %. This is shown in the subsequent graphs (Figure 4.17). Throughout the operational Phases both calculated efficiencies show a tendency to increase.

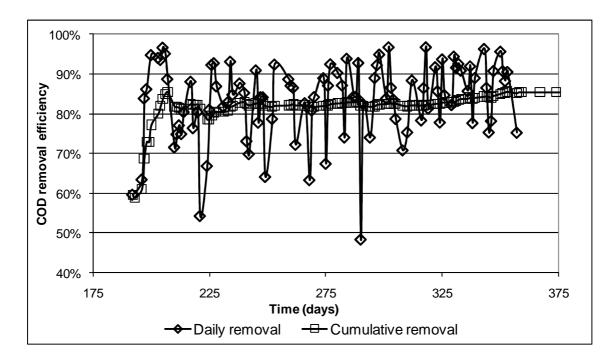


Figure 4.16 COD removal efficiencies calculated based on daily samples and cumulative COD removal.

### 4.3.7.2 LCFA degradation

As previously indicated fat was removed from the wastewater and retained inside the reactor. The LCFA concentration inside the reactor was followed throughout all the Phases. Figure 4.18 shows the LCFA concentration during Phases 2, 3 and 4. Until the addition of a fat overload in Phase 4, the LCFA concentration never exceeded 3000 mg LCFA-COD/L and was well under control. This might be considered as an indication that LCFA were being degraded. The fat overload applied during Phase 4 resulted in a severe increase of the reactor LCFA concentration.

Table 4.4 shows the LCFA composition during Phases 1 & 2 only, so that a fair comparison could be made with the reactor influent and the influence of raw fat addition could be eliminated. Stearic and palmitic acid formed more than 94% of all LCFA present in the reactor, while forming 98.2% of LCFA present in the effluent. Unsaturated LCFA were practically not present, forming only 2.8% of the LCFA present in the reactor and only 0.7% in the reactor effluent. It seems fair to state that the anaerobic population had more difficulties degrading saturated LCFA than unsaturated LCFA, which makes sense in chemical terms since the double bond is considered more reactive than a single bond.

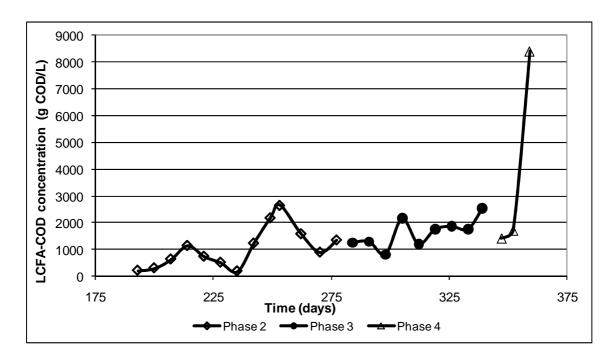


Figure 4.17 Total Reactor LCFA-COD concentration during Phases 2, 3 and 4.

**Table 4.4** LCFA composition of reactor influent, reactor and reactor effluent.

Long Chain Fatty Acid	Influent		Reac	tor	Effluent	
	Wt.%	COD.%	Wt.%	COD.%	Wt.%	COD.%
Linoleic acid	0.7	0.6	0.2	0.2	0.0	0.0
Oleic acid	6.1	6.1	2.4	2.4	0.3	0.3
Stearic acid	36.6	37.1	44.6	45.1	47.2	47.6
Palmitoleic acid	0.2	0.2	0.2	0.1	0.4	0.4
Palmitic acid	51.6	51.3	49.4	49.4	51.0	50.6
Myristic acid	4.8	4.6	3.1	3.0	1.5	1.5
Lauric acid	0.0	0.0	0.0	0.0	0.0	0.0

The addition of raw fat slightly changes the picture and there is a bigger contribution of unsaturated LCFA (Table 4.5). The weight percentage of stearic acid is considerably lower, while the contribution of linoleic acid in the reactor effluent has gone up from 0.0% to 3.9%.

Table 4.5 LCFA composition of reactor and reactor effluent

Long Chain Fatty Acid	Reac	tor	Effluent			
	Wt.%	COD.%	Wt.%	COD.%		
Linoleic acid	0.9	0.9	3.9	3.9		
Oleic acid	3.4	3.4	3.0	3.0		
Stearic acid	38.9	39.3	39.5	39.9		
Palmitoleic acid	0.0	0.0	1.4	1.3		
Palmitic acid	53.6	53.2	50.9	50.6		
Myristic acid	3.2	3.1	1.3	1.3		
Lauric acid	0.0	0.0	0.0	0.0		

Figure 4.19 shows the LCFA accumulation onto reactor sludge. Until the application of a fat overload at the end of Phase 4, LCFA adsorbed onto the reactor sludge did not exceed 1.2 g LCFA-COD/g TSS. Pereira et al. (2004) previously determined that an accumulation of 1 g LCFA-COD/g TSS is the optimum amount for methane production maximisation. Jeganathan et al. (2006) indicated an even higher allowable LCFA accumulation onto the sludge of 1.04 g FOG/g VSS, which would traduce to around 2.5 g LCFA-COD/g TSS. Thus, it may be concluded that inhibition by LCFA accumulation did not occur and LCFA was presumably converted into biogas.

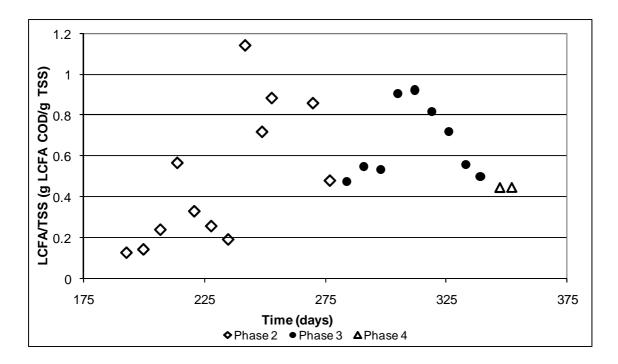
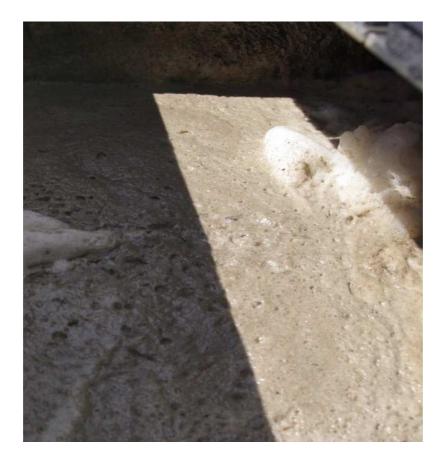


Figure 4.18 LCFA accumulation onto the reactor sludge throughout Phases 2 to 4.

#### 4.3.7.3 Solids

In the reactor three zones could be defined for suspended solids: The floating layer, the actual suspended solids zone and the sedimentation zone. It was difficult to characterise the floating layer. Regular visual inspection showed that the layer formed was fluffy and not to thick until addition of fat was started. This eventually resulted in a relatively thick layer of fat with foamy biomass on top (Figure 4.20). No solids determinations were done of the floating layer.



**Figure 4.19** Floating layer in the top section of the reactor after addition of raw fat.

The TSS concentration of the suspended solids zone and the sedimentation zone (reactor underflow) are shown in Figure 4.21 together with the reactor effluent concentration. The graph clearly shows that solids retention was efficient. The reactor TSS concentration was quite stable around 2 g/L and showed a tendency to increase after raw fat addition was started. The underflow TSS stayed between 10 and 12 g/L throughout Phase 2 and showed a drastic decrease some time after fat addition was started. This may have been due to fat adsorption and subsequent floatation.

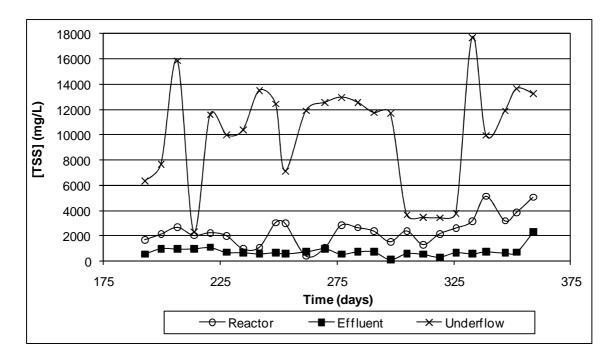


Figure 4.20 TSS concentration of the suspended solids zone (reactor), effluent and sedimentation zone (underflow).

The fraction of volatile solids showed an interesting picture. This is shown in Figure 4.22. The VSS fraction of the underflow was very stable between 89 and 90% wt.. The reactor and effluent VSS fraction fluctuated more and was generally lower than the underflow fraction. One would expect a more similar behaviour. This might be an indication of the presence of refractory inorganic solids that are hard to settle. Another indication for this was found in the sludge volume index (SVI): The reactor solids SVI was considerably lower than the underflow SVI. Reactor SVI was normally 10 ml/g, while underflow SVI stayed above 40 ml/g. Thus, it appeared that the underflow TSS were more fluffy, normally an indication of more organic material. It must be stated, though, that the underflow SVI was still a lot lower than SVIs normally encountered in activated sludge systems: here SVIs between 80 and 120 ml/g may be considered normal (personal experience). Therefore, the settling solids in the IASB reactor seem to be quite compact indicating adequate settlability.

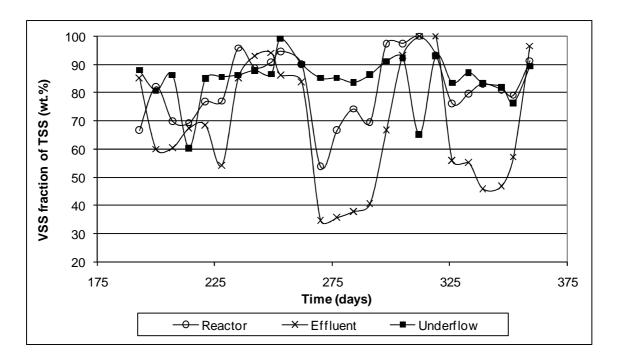


Figure 4.21 VSS fraction of the TSS (wt.%) for the reactor, effluent and underflow TSS.

The importance of retention of sufficient sludge in the reaction compartment is shown in Figure 4.23, which shows the relation between biogas production and reactor VSS concentration. As can be seen, a significant biogas production increase was noticed when the VSS concentration increased from 2000 mg/L to 3000 mg/L.

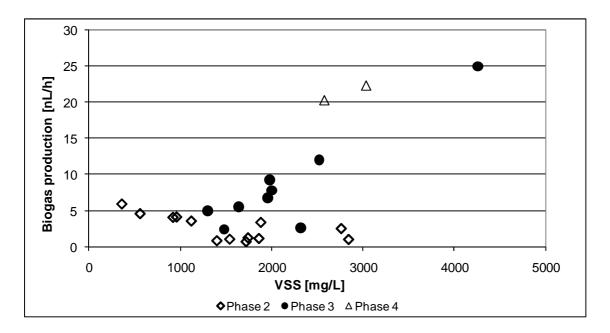


Figure 4.22 Biogas production vs. reactor VSS concentration during Phases 2, 3 and 4.

Taking into account the measured solids concentrations present in the reactor and underflow, the specific organic loading rate (kg COD/kg VSS/day) can be estimated. Assuming an even distribution of the VSS concentration over the reactor and separator section of the reactor VSS concentration and underflow concentration, respectively, it is possible to calculate the total amount of solids present in these two sections and come to an estimated overall VSS concentration. This exercise resulted in the following graph.

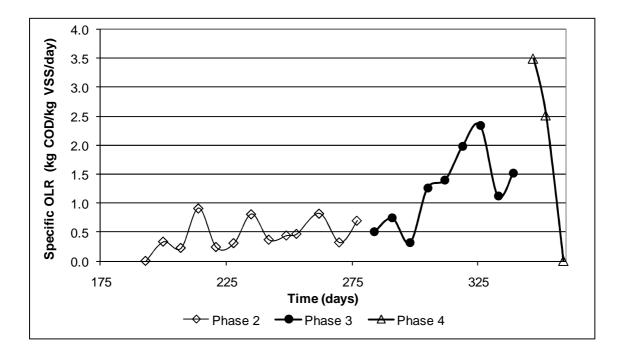


Figure 4.23 Specific OLR during Phases 2 through 4.

The specific OLR stayed reasonably constant during Phase 2 and showed a tendency to increase during Phase 3. The highest specific loading rate was achieved during Phase 4 on application of a fat overload.

### 4.3.7.4 Hydrodynamic characterisation

Due to the strongly varying flow conditions it was difficult to come to a straight forward hydrodynamic characterisation. Influent COD concentration dictated the hydraulic retention time, resulting in strong variations. Figure 4.24 shows the net HRT in the Reaction Compartment during the various Phases. During continuous operation it fluctuated between 1.3 and 14 days and showed a tendency to decrease with increasing loading rate. However,

due to the high wastewater COD concentrations, it was not possible to reduce the HRT to less than one day. Highest net flow rates applied were between 20 and 23 L/h.

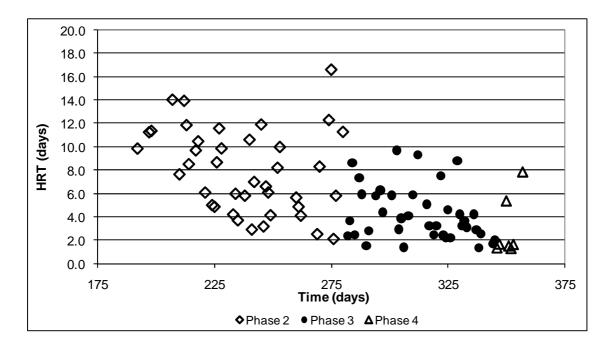


Figure 4.24 Net HRT in the reactor compartment during Phases 2, 3 & 4.

Most of the mixing energy was introduced into the reaction compartment by the spray flow rate and the reactor sludge recycle flow rate. Both determine the mixing time  $t_m$  and this would therefore be the more indicated parameter to analyse the hydrodynamic conditions inside the reaction compartment.  $t_m$  fluctuated between 4 and 10 h. Unfortunately no clear correlations could be found between reactor performance and the mixing time  $t_m$ . The same could be said for the time to effluent tank  $t_{\rm et}$ .

In terms of flow rates applied, the spray flow rate had a determining effect on separator performance. As indicated above, the net feed flow rate did not exceed 23 L/h, while the highest mean spray flow rate that could be applied was 500 L/h. Over the pilot trial, the spray was operated intermittently and the on/off frequency and therefore spray flow rate were determined in accordance with hands-on experience during the pilot, *i.e.* if there were obvious indications that increasing the mean spray flow rate or the on/off frequency resulted in better operation, measures were taken to achieve that potentially better operation. This resulted in an increase in spray flow rate over time from mean hourly flow of 1 to 125 L/h at the end of the pilot trial. These flow rates did not influence the effluent, reactor or underflow TSS/VSS

concentrations. Thus, it seems that settlable solids were retained and mostly colloidal solids left the reactor.

The spray itself had a limited effect on biogas liberation and foam breaking. This was mostly due to the fact that the spray heads were too close to the liquid surface and therefore they covered only a small part of the liquid surface. In the presence of a lot of animal fat in the floating layer, the layer breaking capacity of the spray seemed to be limited. Additionally, even flow rate distribution over the spray heads was difficult. Taking this into account, sequential injection should be considered an option to increase foam breaking capacity and be in complete control of even spraying.

The downflow velocity through the downers was controlled between 5.5 and 10 m/h. Although entrainment could be noticed at these downflow velocities, it did not seem to have a strong effect on biogas liberation and top layer mixing. Therefore, it is only logical to apply higher downflow velocities at full-scale. Also with the downflow injection, it showed to be difficult to evenly distribute the flow over the two downers. Again, sequential injection seems to be the way to eliminate uneven distribution over the downers and to increase the downer flow rates without having to resort to very big pumping capacities.

# 4.4 Performance comparison with other reactors

The treatment of fat containing wastewater has had quite some attention in recent years, thus creating the possibility to compare the IASB performance with other studies. Table 4.6 shows results from a literature review. The table has been adapted from Alves et al. (2009)

**Table 4.6** Treatment of wastewater containing lipids and LCFA in different anaerobic reactors.

Type of wastewater	Type of Reactor	T (ºC)	HRT (d)	OLR (g COD L <sup>-1</sup> d <sup>-1</sup> )	Specific OLR (g COD gVSS <sup>-1</sup> d <sup>-1</sup> )	COD removal (%)	CH <sub>4</sub> yield (LCH <sub>4</sub> /g COD)	Ref.
Slaughterhouse wastewater	IASB	30- 35	1.5-14	0.5-16	0.6-3.5	80-85	ND	This study
LCFA mixture	EGSB	30	0.25- 0.13	4-8	0.20-0.41	44-69	ND	Hwu et al. (1998a)
LCFA mixture	EGSB	55	0.25- 0.13	4-8	0.23-0.47	66-73	ND	
LCFA mixture	UASB	35	0.7- 1.2	3.2-9.4	0.09-0.25	82-93	ND	Hwu et al., (1998b)
LCFA mixture <sup>a</sup>	CSTR + UASB	35	2.9	0.2-2.7	ND	60-95	ND	Kim et al. (2004)
Oleate (+milk) <sup>b</sup>	EGSB	37	1	4-8	1.39-2.78	69-97	0.03-0.28	Pereira et al. (2002)
Oleate (+milk) <sup>b</sup>	AF	37	3.3- 0.64	0.7-12.5	0.09-1.67	80-95	0.09-0.36	
Oleate	EGSB	37	1.18	3.3	0.85	75-86	0.03	Pereira et al. (2005)
Palmitate	EGSB	37	1.14	3.2	0.83	90-95	0.03	
Saccharose+oleate	UASB	35	1	4.2-6.3	0.25-0.38	76-90	ND	Miranda et al. (2006)
Saccharose+oleate	DAEB	35	1	4.2-6.3	NA	77-93	ND	
Saccharose+stearate	UASB	35	1	4.2-6.4	0.25-0.38	77-90	ND	
Saccharose+stearate	DAEB	35	1	4.2-6.4	NA	76-90	ND	
Dairy wastewater	UASB		6-40	2.0-4.5	ND	79-99	ND	Gavala et al. (1999)
Dairy wastewater	IFB	35	63.6-3	0.5-10	ND	75-98	ND	Arnaiz et al. (2003)
	ITB	35	66.6-3	0.5-12	ND	75-98	ND	
Dairy lab wastewater	AF	35	1.85-7	2-5.9	ND	66.8-93.0	ND	Omil et al. (2003)
Dairy wastewater	BFBR	-	0.3- 0.5	10 (up to)	ND	85-90	approx. 0.37	Haridas et al. (2005)
Ice-cream factory wastewater	AF	35	0.9	6.4	ND	67	0.36	Hawkes et al. (1995)
	contact process	35	5.5	1.1	ND	82	0.39	
	FBR	35	1.5	4.2	ND	56	0.37	
Whey wastewater	Two-stage AnMBR	37	4	3-19.8	ND	98.5	0.3	Saddoud et al. (2007)
Whey wastewater	PT + UAF	35	2-5	4	ND	98	0.09-0.28	Gannoun et al. (2008)
	UASB	35	1.6	2.2	ND	49	0.19	
Food processing wastewater	UASB or AF	15- 30	0.33-1	0.2-4.2	ND	80-90		Oliva et al. (1995)

Type of wastewater	Type of	Т	HRT	OLR	Specific	COD	CH <sub>4</sub> yield	Ref.
	Reactor	(ºC)	(d)	(g COD L <sup>-1</sup> d <sup>-1</sup> )	OLR (g COD gVSS <sup>-1</sup> d <sup>-1</sup> )	removal (%)	(LCH <sub>4</sub> /g COD)	
Food processing wastewater	multi-stage UASB	55	0.14	50	2.29	60-70	ND	Tagawa et al. (2002)
Food processing wastewater	UASB	35	5	2.7-5.2	0.19-0.37	94-98	0.24-0.32	Jeganathan et al. (2006)
	UASB	35	2.5- 1.25	1.3-8.0	0.07-0.42	84-89	0.24-0.48	
	(PBR+) UASB	35	2.5- 1.25	1.3-4.2	0.06-0.18	86-90	0.18-0.42	
Food processing wastewater	(PBR+) UASB	35	1.25- 2.5	1.3-4.2	-	90	-	Jeganathan et al. (2007)
Slaughterhouse wastewater	UASB	37	1.2- 6.5	1.0-6.6	ND	59.0-93.0	-	Ruiz et al. (1997)
	UAF	37	0.5- 7.1	0.9-11.2	ND	28.4-83.6	-	
Slaughterhouse wastewater	UASB + UAF	35	1.5	2.5-6.9	-	90-96	0.29-0.35	Borja et al. (1998)
	UASB + UAF	35	0.5- 1.35	7.7-20.8	-	93.4-95.9	0.29-0.35	
Slaughterhouse wastewater	EGSB	35	0.2	15 (up to)	ND	67	ND	Núnez & Martínez (1999)
Slaughterhouse wastewater	DAF+UASB	35	0.4	4	0.43	90	0.34	Manjunath et al. (2000)
	UASB	35	0.4	3.5	0.16	70	0.28	
Slaughterhouse wastewater	ASBR	35	0.63- 4.5	0.6-6.1	-	86	-	
Slaughterhouse wastewater	mUASB	35	0.58- 0.92	2.7-10.8	ND	76.6-90.6	ND	Caixeta et al. (2002)
Slaughterhouse wastewater	AnMBR	30	1.2	6-8	-	97	0.12-0.32	Fuchs et al. (2003)
Slaughterhouse wastewater	AFFR	20	0.2- 0.6	3.6-9.2	-	54-70	<0.375	Del Pozo et al. (2005)
Slaughterhouse wastewater	ASBR	30	2	0.7-4.9	-	95	-	Massé & Masse (2000b)
Slaughterhouse wastewater	ASBR	20	2-3	2.25	0.14	-	0.31	Massé et al. (2001)
Claushtadaaaaaaaa	ACDD	25	2.0	2.85	0.12		0.26	Marana et al. (2002)
Slaughterhouse wastewater	ASBR	25	2.9	31.3 <sup>c</sup>	ND	94	ND	Masse et al. (2002)
Slaughterhouse wastewater	UASB	33	0.3-	13-30	1.0-2.5	60-93	0.20-0.28	Torkian et al. (2003)
Slaughterhouse wastewater	UASB	20- 27	0.3- 0.88	4-15	0.31-1.2	76-90	0.24-0.27	Mijaylova Nacheva et al. (2011)
Slaughterhouse wastewater	UASB	30	0.7- 1.0	1.5-2.5	0.09-0.15	65-85		Del Nery et al. (2008)
Slaughterhouse wastewater Slaughterhouse +olive mill	Draw-and-fill reactor	35	20	0.20	-	48	0.30 <sup>d</sup>	Fountoulakis et al. (2008)
wastewater (1:1) Olive mill	. 20001	35	20	2.1	-	85	0.17 <sup>d</sup>	,,
Wastewater		35	20	4		75	0.11 <sup>d</sup>	
Palm oil mill wastewater	MABR	-	3-10	1.6-5.3	ND	87-95	0.32-0.42	Faisal & Unno (2001)
Palm oil mill wastewater	UASFF	38	3-1.5	1.8-23.2	0.18-1.23	89-97	0.31-0.35	Najafpour et al. (2006)
Sunflower oil factory wastewater	UASB	37	2-2.8	1.6-7.8	ND	87	0.16-0.35	Saatci et al. (2003)

HRT – hydraulic retention time; OLR – organic loading rate; COD – chemical oxygen demand

UASB – up-flow anaerobic sludge bed reactor; CSTR - continuously stirred tank reactor; EGSB – expanded granular sludge bed reactor; DAEB – downflow anaerobic expanded bed; IFB – inverse fluidized bed reactor; ITB – inverse

turbulent bed reactor; BFBR – buoyant filter bioreactor; AF – anaerobic filter; PBR– packed bed reactor (containing immobilized lipase beads); ASBR – Anaerobic sequencing batch reactor; UASFF – up-flow anaerobic sludge-fixed film reactor; MABR – modified anaerobic baffled bioreactor; PT – pre-treatment

ND-not determined; NA-not applicable; (-) – information not available

Compared with other reactors it can be stated that the IASB reactor showed a good performance in terms of OLR and COD removal. The specific OLR range was comparable to ranges reported by Pereira et al. (2002) and Tagawa et al. (2002). The biogas yield stayed behind. The probable reasons for this have been previously explained. Specifically concerning LCFA removal the IASB reactor showed excellent results. Manjunath et al. (2000), for example, tested the combination of DAF and UASB for slaughterhouse wastewater treatment and recommended the inclusion of DAF to increase treatment efficiency of the UASB system. Obviously the increase is due to the prior removal of complex organic compounds such as fats. They managed to remove up to 90% of fats using the DAF. Faisal & Unno (2001) reached a FOG removal efficiency of up to 90% from POME wastewater with an initial FOG concentration of 400 mg/L using a modified anaerobic baffled reactor (MABR). Caixeta et al. (2002) tested a modified three-phase separator for slaughterhouse wastewater treatment and obtained results comparable to those with the pilot plant with OLRs going up to 10.8 kg COD/m<sup>3</sup>/day with influent fat concentrations reaching up to 607 mg/L. The three above mentioned studies showed results comparable to fat removal efficiencies reached with the IASB reactor. Fat influent concentrations applied in the IASB reactor were higher, specifically when raw fat was added, thus indicating that fat removal and retention inside the IASB reactor was efficient. Only Jeganathan et al. (2007) reported comparable FOG loading rates in a two reactor system in which they combined a packed bed reactor (PBR) with immobilised lipase enzyme beads and a UASB reactor. They applied FOG loading rates of up to 4.9 kg FOG/m<sup>3</sup>/day (Jeganathan et al., 2007) and a total OLR of 12 kg COD/m<sup>3</sup>/day to the PBR, without destabilisation of the process. The UASB reactor, however, received a much lower FOG OLR of 0.6 kg/m<sup>3</sup>/day, since its volume was 5 times bigger than the PBR volume. Thus, it seems a bit artificial to say that they actually applied a FOG OLR of 4.9 kg FOG/m<sup>3</sup>/day.

Fuchs et al. (2003) tested an anaerobic membrane bioreactor configuration on three types of wastewater (artificial, sauerkraut brine and slaughterhouse wastewater). The lowest OLRs were obtained on slaughterhouse wastewater, showing the difficulties in treating this kind of wastewater, even with complete retention of organic matter. The highest reported OLR was 8

<sup>&</sup>lt;sup>a</sup> glucose was used as a co-substrate during reactor operation; <sup>b</sup> skim-milk was used as co-substrate during the start-up period; <sup>c</sup> calculated with basis on the 1 h time feeding of the reactor (subsequent reaction and settling phases lasted for 69 h); <sup>d</sup> LCH<sub>4</sub>/gCOD added.

kg COD/m³/day and resulted in an overload. Comparably, the IASB reactor showed a better performance, since it only showed signs of overloading at OLRs above 12 kg/m³/day. Furthermore, in the case of the IASB reactor this overloading was mainly due to the very high fat loading rate applied.

Miranda et al. (2006) operated 3 UASB reactors and 2 downflow anaerobic expanded bed (DAEB) reactors at lab scale with synthetic wastewater either containing oleate, stearate or a mixture of both. They found that specifically stearate was difficult to degrade and that the DAEB reactors showed better performance. In all reactors problems were already encountered at OLRs of 5 to 6 kg COD/m³/day with decreasing COD removal efficiencies. Comparably, the IASB reactor performance was much better up to the application of COD overloads at the end of the pilot test.

Nuñez & Martinez (1999) reached OLR of up to 15 kg COD/m³/day with reasonable COD removal efficiencies. The fat concentrations in their slaughterhouse wastewater, however, were relatively low compared to ones applied in this study.

Ruiz et al. (1997) compared the performance of a lab-scale UASB and a lab-scale UAF reactor for slaughterhouse wastewater treatment with mean fat concentrations of up to 415 mg/L. Above an OLR of 5 kg COD/m³/day both reactors destabilised with the UAF showing the worst performance. Sludge floatation was induced above this OLR in both reactors, indicating that sludge retention through floatation is crucial for a reactor to efficiently treat wastewater containing fat. Ruiz et al. (1997) also applied shock loads which resulted in acidification. This was comparable with what happened in Phase 4 of this pilot trial, during which a fat shock load was applied resulting in total VFA concentrations rising up to 10 meg/L.

Miranda et al. (2005) followed the operation of an industrial scale UASB system for slaughterhouse wastewater treatment for a period of three years. They stated that the ratio between FOG and total influent COD should stay below 0.2 to assure proper reactor operation. This is not in agreement with findings with this pilot trial.

Other studies were less specific about the presence or absence of FOG. Del Pozo et al. (2005), for example, operated an anaerobic fixed film reactor (AFFR) for slaughterhouse wastewater treatment and achieved volumetric loading rates up to 9.2 kg/m³/day at 20°C. This was accompanied by a low removal efficiency of 54%. No fat seemed to have been present in their wastewater and they mainly attributed degradation difficulties to the presence of blood and reactor short-circuiting. Besides this, the influent COD concentrations applied were quite low

(up to 2 g/L). Thus, comparably, the IASB reactor performed exceptionally well, since the wastewater fed contained blood and FOG. Borja et al. (1998) used a hybrid lab-scale reactor combining a UASB with an anaerobic upflow filter consisting of polyurethane foam. They reached organic loading rates of up to 20 kg/m³/day on actual slaughterhouse wastewater, which should be considered as a very good performance. Unfortunately, it is again not clear, however, whether the wastewater contained fat as well, since it was not characterised in terms of suspended solids or fat. Torkian et al. (2003) operated a 1 m³ UASB reactor fed with slaughterhouse wastewater. They achieved very high loading rates (30 kg COD/m³/day) and managed to maintain good removal efficiencies. It is surprising, however, that the influence of LCFA on reactor performance is not mentioned in spite of the fact that there is a notable difference of 2.2 g/L between reported soluble and total COD concentrations. The most reasonable explanation is that fat was removed prior to treatment and that the particulate and colloidal COD was probably the contribution of protein COD.

A comparison with anaerobic reactor treating pre-treated wastewater also shows the good performance of the IASB. Oliva et al. (1995) reported OLRs between 0.21 and 4.2 kg COD/m³ day for full-scale UASB reactors and anaerobic filters treating wastewater from the food industry. Influent concentrations applied, however, were quite low with the maximum COD concentration being 4.7 g/L. They further indicated that the wastewater was pre-treated before biological treatment. Therefore, it is likely that the reactors were not exposed to wastewater containing fat. Thus, the IASB reactor performance may be considered as exemplary. Mijaylova Nacheva et al. (2011) operated a lab scale UASB reactor at ambient temperature on pre-treated slaughterhouse wastewater, *i.e.* fat and solids were removed prior to processing. They obtained stable operation and good efficiencies up to 15 kg COD/m³/day.

Massé et. al (2001) did not mention encountering any problems with the treatment of slaughterhouse wastewater containing fat in ASBRs operated at 20 and 25°C. They did mention, however, that particulate hydrolysis seemed to be the rate limiting step at 25°C. In their case, methanogenesis was not found to be rate limiting at all. This indicates the importance of retaining the right micro-organisms inside the reactor by applying appropriate technology. LCFA concentrations during Phases 2 and 3 did not rapidly increase. In fact, they were quite stable, thus suggesting that the right micro-organisms were being retained inside the reactor.

## 4.5 Conclusions

Overall it may be concluded that the IASB showed a good performance with OLR reaching up to 16 kg of COD/m³/day. At this OLR the IASB reactor was overloaded due to excessive fat addition. Nevertheless, the IASB reactor showed to be robust and capable of dealing with a strongly varying wastewater composition. No LCFA accumulation was noticed, until excessive quantities of raw fat were added during Phase 4. This resulted in a reactor overload.

Up to the raw fat overload, no LCFA inhibition was noticed. LCFA accumulation reached values of up to 1.2 g LCFA-COD/g VSS, which is comparable to optimised LCFA loading indicated in the literature. Specific OLR were estimated between to fluctuate between 0.3 and 3.5 kg COD/kg VSS/day.

The TPS was capable of efficiently retaining biomass inside the reactor. The hydraulic rate over the TPS was determined by the spray flow rate. Therefore, during scale-up it should be attempted for the spray flow not to go through the TPS as to reduce its size as much as possible.

When the LCFA load was increased, LCFA emulsification seemed to take place, resulting in a silvery shine. This may have conducted to biogas encapsulation which was then mainly released in the effluent tank. This was thought the reason for the impossibility to account for all biogas produced.

Intermittent spraying seemed to be best for the process, instead of continuous spraying. Reactor mixing needs to be optimised so that biogas encapsulation is counteracted. This should be done by increasing the downflow velocity through the downers and the application of sequential injection.

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# 5 IASB scale-up

## 5.1 Introduction

Final goal of the IASB technology development is its application at full-scale. For this the IASB technology needs to be scaled up. This is a phase of compromise, since the technology needs to work, be affordable and practical at the same time. The starting point of scaling up is the pilot scale design. It does not necessarily need to be the end point, however.

# 5.2 Some scale-up considerations

The biggest scaling-up challenges are associated with the TPS. Of all technologies incorporated in the IASB, the separator occupies the most space and will definitely influence the price of a full-scale IASB reactor. Therefore, one of the main goals should be to try and reduce its size and its influence on final reactor design as much as possible without reducing separation efficiency.

Besides its size, the TPS will be exposed to thickened sludge and also offers the biggest surface area for bacteria to potentially adhere to. Biofilm formation should be counteracted in all ways possible, so that the TPS separation efficiency does not deteriorate over time.

Secondly, there is the mixing challenge. At pilot scale problems were encountered with the proper injection of the wastewater/sludge recycle mixture. It showed to be tough to distribute it evenly over two injection points. Furthermore, the downflow velocity did not seem to be high enough to ensure the envisioned entrainment into the two downers installed.

Thirdly, foam breaking efficiency should be ensured without influencing too much the TPS sizing. At pilot scale, the spray flow rate largely determined the hydraulic loading rate over the TPS. One of the considerations to be taken into account is to use other water for spraying than reactor effluent, thus uncoupling the spray flow rate from TPS design.

Finally, one needs to take into consideration the fact that different reactor sizes of the IASB technology should become available. The Dutch company Paques for example offers its anaerobic IC®-reactor at standardised sizes of 100 m³, 250 m³, 500 m³ and 1000 m³. They are

able to do so due to the modularity of the technology. Therefore, the IASB technology should preferentially be modular as well.

## 5.3 <u>Design basis</u>

For scaling-up purposes, initially the concrete case of Barroso & Alto Tâmega Slaughterhouse was taken into account. Although higher loading rates were achieved at pilot scale, to ensure reactor stability and removal efficiency the mean volumetric loading rate was set at 10 kg COD/m³/day. On a production day, the organic load at the slaughterhouse fluctuated between 750 and 1200 kg of COD/day. Therefore, a mean loading rate of 1000 kg COD/day seems reasonable, resulting in a useful reactor volume of 100 m³. This should be considered as the smallest reactor size in the future IASB range of reactor sizes.

Since no industrial wastewater was created equal, the volumetric loading rate should be applicable for a range of concentrations and flow rates. It is convenient to define a minimum hydraulic retention time at which stable reactor operation is still guaranteed. For the IC® reactor the minimum HRT limit is set at 4 hours (personal experience) and is applicable for effluents with easily degradable COD, such as generated at breweries. Since the IASB reactor was initially designed for the treatment of water containing fat, an HRT of 4 hours might be considered ambitious. Nevertheless, if the goal is a modular design that can be applied in reactors of several sizes, an ambitious goal should be set for the base case of a 100 m³ reactor. Thus, a maximum flow rate for the separator of 20 m³/h was set, resulting in a minimum HRT of 5 hours.

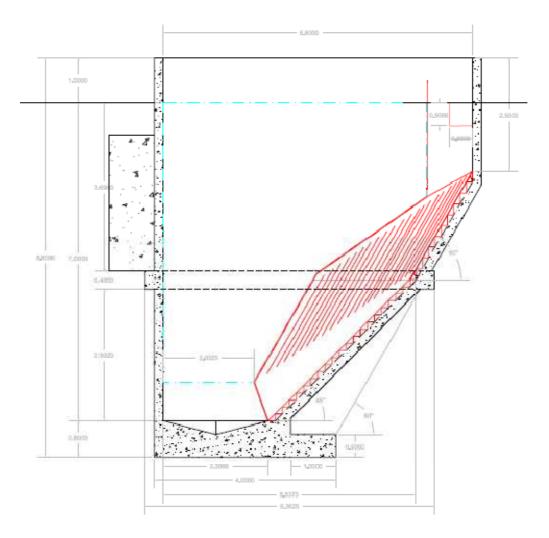
The pilot-scale reactor showed that mixing could be improved. The applied downflow velocities through the two downers were not sufficient to obtain adequate entrainment for biogas liberation and proper reactor mixing. Furthermore, the synchronous even distribution through the downers was practically impossible to maintain. Therefore, another approach was chosen for the full-scale design. To avoid the installation of excessive sludge recycle pumping capacity, sequential injection through the downers was chosen. The sludge recycle should be able to maintain the desired mixing regime when no wastewater is fed to the reactor. Therefore, the maximum sludge recycle rate was set at 20 m³/h, *i.e.* the same rate as the maximum feed flow rate.

This same approach was chosen for spraying. This way a more powerful spray can be applied without having to install a big pump. The chosen maximum spray flow rate was 15 m<sup>3</sup>/h. It is important to define this flow rate, since it may have a big influence on the TPS design, if one chooses to let the spray flow rate pass through the TPS.

The defined design basis was applied for the definition of two different scenarios. In one scenario, the pilot reactor design as such was used as basis for full-scale design. In the second scenario, a more modular approach was taken into account. For both cases it must be stressed that this is the first design basis and that it is in no way a fixed basis. Only actual full-scale application will show whether the choice was right or whether it should be adapted.

# 5.4 Scale-up of pilot geometry

Since a defined geometry had already been tested at pilot scale, it was only logical to try and come up with an industrial scale reactor with the same kind of geometry. Thus, the first scale-up attempt was the reproduction of the pilot scale reactor at full-scale. Figure 5.1 shows the result of this exercise. For this design several kinds of materials were considered. For the reactor walls concrete was considered. For the separator carbon steel and glass fibre reinforced polyester were considered. Finally, for the reactor cover, only glass fibre reinforced polyester was considered.



**Figure 5.1** First full-scale design of the IASB reactor (courtesy of MonteAdriano Engenharia & Construção, Póvoa de Varzim, Portugal).

For the calculation of the required settling area, both the spray flow and the wastewater flow were considered, since this was the case for the pilot plant. Therefore, on top of the 20 m<sup>3</sup>/h of feed flow rate, the earlier indicated spray flow rate of 15 m<sup>3</sup>/h was taken into account as well.

The scaling up of the pilot geometry has one big drawback: the reactor is not cylindrical. Although in practice also more rectangular geometries are applied, most high-rate anaerobic reactors are cylindrical. A few reasons can be indicated for this: the cylindrical structure is known as being a strong structure. Consequently, one can save on material (thinner walls) and in some cases even use material with a lower mechanical strength (specifically when the reactor is not too high). Cylindrical reactors are also easier to build, thus reducing installation

costs. Finally, cylindrical reactors are less prone to the creation of dead spaces. To be able to come to a cylindrical reactor, a modular approach was necessary.

# 5.5 Scale up: Modular approach

For the first scale-up attempt, what was already known was taken as basis, resulting in a very expensive design. If one wants to reduce reactor production costs, it is best to try to come up with a module that can be produced in series and incorporates at least part of the essence of IASB technology. The best candidate for module design would be the TPS, since it showed to have the biggest influence on reactor construction cost when scaling up pilot reactor geometry. Therefore, this is the one piece that needs to be moulded into a module that can in principle be applied in any kind of cylindrical reactor.

This also creates more freedom in the application of different kinds of material for the module, since the modular approach increases the potential of serial production. Three materials were evaluated: carbon steel, polypropylene (PP) and dicyclopentadiene polymer (DCPD). Both carbon steel and PP are commonly used in wastewater treatment. DCPD is less known, but is already applied for the production of compact wastewater treatment system (personal communication Rimsys). Specifically the polymers have smooth surfaces and are therefore less susceptible to biofouling. PP, however, does not have the mechanical strength of steel and DCPD requires a mould, which results in a higher initial investment cost. In conclusion one could say that every material has it pros and cons.

The most critical design criterion in the design is the plate area to be incorporated in the module, since it will largely determine its size. It is essential to reduce this area as much as possible without compromising separation efficiency too much. The most logical choice is not to let the spray flow go through the TPS and simply use reactor contents for spraying. This way the maximum design flow rate over the TPS will be 20 m³/h, almost cutting the required area in half. Naturally care should be taken in choosing the spray nozzles, since more solids will flow through them.

The design settling velocity was set at 0.8 m/h, which may be considered as quite conservative (WEF, 2005). The perpendicular plate distance was chosen to be 100 mm, which coincides with plate distances commonly used for TPS design (WEF, 2005) and ensures that the

sedimentation process between plates is not disturbed. Plate inclination was chosen to be 60°, so that appropriate self-cleaning ability is incorporated.

The first concept design is shown in Figure 5.2. It simply is a TPS module with an extended settling zone to prevent biogas formation in the reactor from influencing the settling process. The module incorporates the TPS and the effluent compartment and gives the opportunity for placement either completely or partially inside the reactor. In the last case, the effluent compartment is placed outside the reactor and should be closed at the top and connected to the biogas network. The last mentioned option was developed further, since in this way more space becomes available for actual anaerobic wastewater treatment.

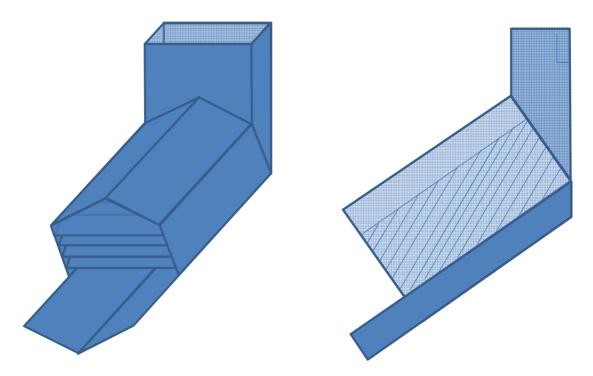


Figure 5.2 Conceptual design of the TPS module.

If the module is mounted partially on the inside, part of the reactor cylindrical wall can be used as the dividing wall between the reaction section and the effluent section, provided that the materials of the reactor and the module are compatible and a gas tight connection can be guaranteed.

The incorporation of the module results in the creation of potential dead space underneath the TPS bottom. However, this might also be considered the ideal spot for pumping reactor liquor for spraying in the head space, since the suspended solids concentration may be lower than elsewhere in the reactor.

The TPS efficiency might further be influenced by its position. If positioned too high, *i.e.* too far off the bottom, the same problems with floating sludge will be encountered as with top-mounted separators. If positioned too low, an anaerobic sludge buffer cannot be built up. Some sludge accumulation at the bottom is desirable due to the changing regimes of sludge recirculation as to provide sufficient reactor mixing. If during a time of lower sludge recirculation too much sludge accumulates at the bottom, it might eventually interfere with the TPS functioning. Figure 5.3 shows the technical side view of a cylindrical 100 m<sup>3</sup> reactor with the TPS module incorporated at a satisfactory height. As can be seen, the lowest tip of the TPS module bottom is positioned 850 mm from the reactor bottom. The reactor bottom is conically shaped so that sludge can be collected at the centre and recycled. About 15 m<sup>3</sup> of sludge can be stored in the bottom section without disturbing the TPS settling process.

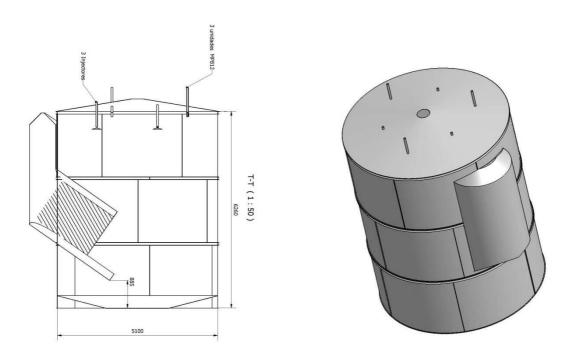


Figure 5.3 Side view of an IASB reactor with a TPS module incorporated and a 3D representation of the same reactor (courtesy of AGOVI, Braga Portugal).

For the 100 m<sup>3</sup> reactor 3 feed/sludge recycle mixture injection nozzles are projected to provide the required mixing. These nozzles can be subdivided into 3 separate sections: the actual point

of injection, the small downer cylinder to stimulate floating sludge entrainment and a conical baffle to prevent reactor short-cutting. Figure 5.4 shows a drawing of these nozzles. If it comes to serial TPS module production, one should consider serial production of these nozzles as well, since they are easily injection moulded. In this preliminary design stage all nozzles point downwards and thus provide a downward flow. It might be interesting, however, to investigate whether a combination of downward and tangential injection results in better reactor mixing and more biogas production.

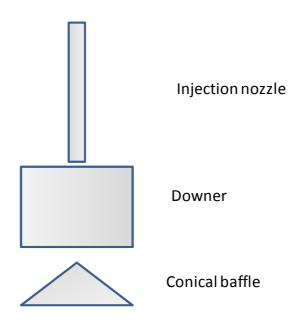


Figure 5.4 Drawing of the IASB injection nozzles.

At pilot scale spray 2 nozzles providing a 120º full cone were used. It is hard to say whether these nozzles were efficient or not, since biogas production lagged behind. At full scale better control of the biogas production is expected and more biogas production as well, resulting in a bigger production per m² of reactor area. In fact, a simple calculation shows that the area specific biogas production at full-scale might be about ten times bigger than the one measured at pilot scale. Therefore, stronger foam formation should be expected and stronger foam breaking power should be installed. This will be partially achieved through the envisioned spray flow rate. Nevertheless, three 90º full conical spray nozzles were chosen for foam breaking. A 90º full conical spray distributes the water over smaller area when sequential spraying is applied, thus resulting in a bigger impact and better foam breaking.

Besides the patented technology, proper sampling should be considered. Therefore, at least sampling points at three different reactor heights will be installed for monitoring purposes.

### 5.6 Further development

The IASB technology has come to the stage of industrial application. This does not mean that its development and optimisation have come to an end. The first few applications at full scale should be intensively monitored to identify potential technology bottlenecks that need to be resolved and opportunities for optimisation.

Further development also refers to the technology that will not be incorporated in the IASB application, but has strong potential to find full scale application in wastewater and organic waste treatment. This technology will be dealt with in the next chapter: the Sludge Lift System.

#### 5.7 References

WEF (Water Environment Federation) (2005); Clarifier design – WEF Manual of Practice No. FD-8; 2<sup>nd</sup> Edition; McGraw-Hill

## **6 The Future: The Sludge Lift System**

#### 6.1 Introduction

The optimisation of the IASB design at lab-scale resulted in the development of an innovative reactor technology that already has been submitted for a patent application (PT105128). It was named the sludge lift system (SLS) and uses both the gas lift effect as well as floatation to stimulate reactor mixing and sludge retention. The technological basis of the invention will be addressed in this chapter.

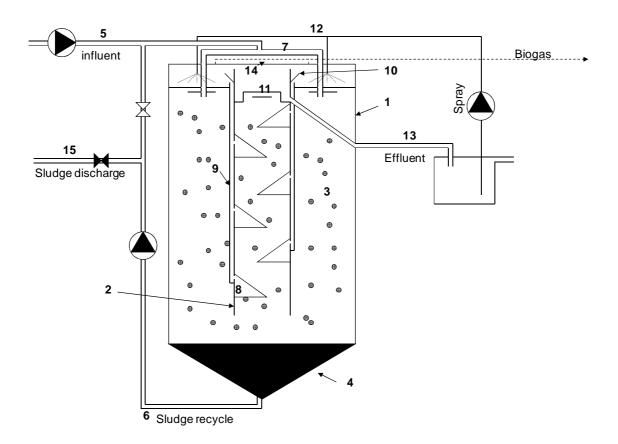
## 6.2 SLS technology explained

In brief, the SLS relates to an apparatus for the retention of (bio)solids inside reaction vessels comprising a vertically elongated effluent compartment, equipped with two or more solids/gas collection hoods. These two or more hoods are then connected to one or more riser tubes that transport sludge to the reaction compartment using the gas lift effect. The effluent compartment is equipped with a treated wastewater outlet system in its top section. This outlet system may for example be a circular overflow weir.

Since the SLS originated as a complementary technology in the IASB reactor to prevent sludge accumulation inside the IASB effluent compartment, it was first envisioned for high-rate anaerobic treatment of wastewater. However, the fact that bio-slurries are transported through riser tubes and that the gas lift effect is used to achieve that, opens up more possible applications of this technology. Bio-slurries may be anaerobic sludge, but also organic waste, thus making it possible to use the SLS for co-digestion reactors for biogas production from organic waste material, such as manure or wasted activated sludge. And since wasted activated sludge might be transported, one could also substitute biogas for air and use the SLS for sludge retention in an aerobic treatment reactor. The most ambitious application envisioned is combined biological treatment: join for example anaerobic and aerobic treatment in one reaction vessel.

Figure 6.1 shows a conceptual drawing of the SLS (2) installed in a cylindrical anaerobic reactor (1) with a conical bottom (4) for settling sludge collection and recycling. In this specific case the same kind of configuration as with the IASB reactor is applied, *i.e.* a sludge recycle (6) is mixed

in-line with wastewater (5) to be treated and then injected in the top of the reactor into the reaction compartment (3) outside the SLS using a circular injection header (7). Biogas is produced inside the reaction compartment and also underneath the SLS. In the SLS sludge and biogas collection hoods (8) are installed, which are connected to riser tubes (9). Due to the collection of both biogas and sludge, eventually the sludge is transported to the reaction compartment by biogas induced gas lift action. As seen at lab-scale and pilot-scale, sludge will be sluggishly ejected from the riser tubes essentially above the water level. Therefore, to prevent sludge from hitting the reactor cover, sludge deflection plates (10) are installed above each riser tube. Enough sludge/biogas collection hoods should be installed to clarify the water flowing upwards through the SLS. This clarified water is then collected in an overflow weir (11) and flows into an effluent collection tank. In this case the possibility is incorporated to recirculate treated wastewater (13) for spraying (12) as to counteract foam formation inside the anaerobic reactor. Biogas is collected in the top of the head space (14).



**Figure 6.1** Schematical drawing of an anaerobic reactor with the SLS installed.

Although it might seem trivial, one of the essential differences between the SLS and existing anaerobic technology is the ejection of sludge and biogas above the water line. In this way only the gas lift effect is utilised and biogas may be produced anywhere inside the reactor. If the sludge discharge point was positioned under the water surface, the system would not work as effectively due to more flow resistance. This kind of gas lifting is applied in the oil industry to enhance oil production from almost exhausted oil wells (Guet & Ooms, 2006). Here, optimisation is mainly focussed on the prevention of slug formation. They try to get a fine bubble formation to improve transport. In the case of the SLS this might be possible if biogas is compressed and sparged inside the riser tubes. Whether this should be considered as a viable option is doubtful. Optimised transport from oil wells is important since transport may take place over hundreds of meters (Guet & Ooms, 2006). This is definitely not the case for the SLS.

Nevertheless, slurry transport through the riser tubes can be optimised in other ways and specifically through the choice of riser tube material. Materials with a smooth surface should be utilised so that biofilm formation is prevented. Furthermore, the riser tubes should have an appropriate diameter to ensure that the slurry is transported at a big enough velocity. Finally, any transport resistance should be eliminated and that is exactly why the sludge should be discharged above the water level.

An existing technology that is comparable to the SLS system is the IC® reactor (Mulder, 2003). There are several differences that can be pointed out, however. First of all, the IC® reactor includes a completely closed gas lift loop. Secondly, although sludge recycling might occur in an IC® reactor, it is definitely not a wanted thing. Gas lift loop itself results in a highly turbulent regime inside the IC downer and may tear granules apart. The IC® reactor is normally operated in such a way that the sludge blanket is not entrained into the first three-phase separator.

As indicated before, it is also considered a possibility to apply the SLS for biomass retention inside an aerobic reactor. Figure 6.2 shows a schematical drawing. In this case, aeration using a compressor would be required. In a normal air lift reactor, aeration should be only located underneath the riser tube. The SLS, however, makes it possible to operate the reactor as a bubble column and aerate over the whole reactor surface. For the rest, the basic functioning is the same as for an anaerobic reactor system.

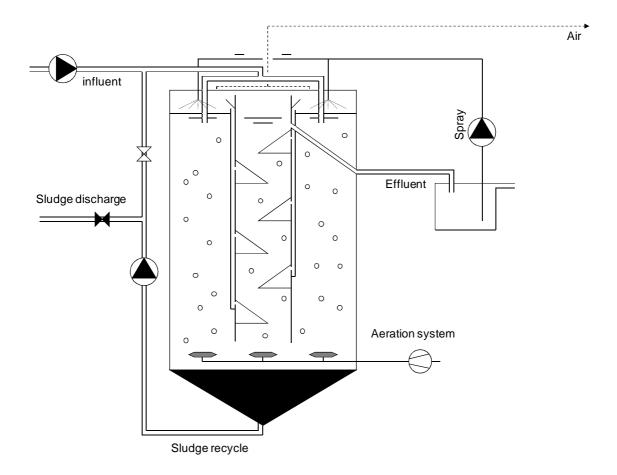


Figure 6.2 Schematical drawing of an aerobic reactor with the SLS installed.

Yet another possibility would be to incorporate for example anoxic and aerobic treatment in the same reactor system. One could think of incorporating the CANON process (Completely Autotrophic Nitrogen removal Over Nitrite) inside such a reactor (Ten Have & Van Kempen, 2004) by incorporating and oxygenated and an oxygen free zone. The same could be possible for a completely biological removal of sulphate by first reducing to sulphide and then oxidising to solid elemental sulphur. These kinds of applications require continued research, however.

## 6.3 SLS development stage

Due to being a very promising technology, big steps are being taken to develop it and take it to market as fast as possible. Recently, hydrodynamic simulations have been carried out using the CFD (Computational Fluid Dynamics) software package Fluent as to obtain an initial optimisation of the SLS and the sludge transport through the riser tubes. These optimisations have been included in the design of a 15 m³ pilot reactor which will be used for SLS

demonstration for the co-digestion of organic waste material. Figure 6.3 shows a technical drawing of the actual pilot reactor and a 3D representation of the SLS. The actual pilot has recently been installed at a waste treatment site near Porto run by Lipor.

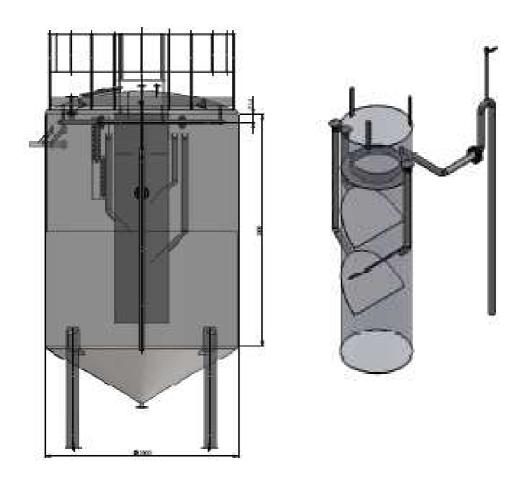


Figure 6.3 Technical drawings of the pilot plant installation. On the right is a 3D representation of the SLS internal.

## 6.4 References

Guet, S. & Ooms, G. (2006); Fluid mechanical aspects of the gas-lift technique; *Annu. Rev. Fluid. Mech.* **38**; 225-249

Mulder, R. (2003); Biological Wastewater Treatment for Industrial Effluents: Technology & operation; Paques BV; ISBN 90-807754-1-X

Ten Have, R. & Van Kempen, C. (2004); Rejectiewaterbehandeling geëvalueerd – SHARON, effluentkwaliteit, alternatieven en markpotentie; STOWA report 2004-20; ISBN 90.5773.254.8

#### 7 General discussion and conclusions

In overview this thesis gives a recipe for full technological development of novel wastewater treatment technology until market introduction. This excludes the actual fundamental research associated to the anaerobic mineralisation of LCFA. Therefore, a very practical discussion focusing more on the process rather than the result seems to be more in place. Looking at the recipe, it can be broken down into the following phases:

- Market niche identification (direct fat removal from industrial wastewater with associated biogas production);
- Current technical problem identification (fat leads to floatation and disruption of conventional treatment processes and therefore needs to be removed prior to treatment);
- 3. Technological solution to the problems identified (use the problems identified to your advantage);
- 4. Concept development (combine the solutions into one apparatus);
- 5. Protection of intellectual property (patent submission);
- 6. Further concept development and demonstration at lab-scale;
- 7. Further concept development and pilot demonstration;
- 8. Apparatus scale-up to first industrial application.

The identification of the market opportunity is of considerable importance to be able to make the rest of the steps more feasible. From a scientific point of view, it is probably one of the most difficult phases in the whole process. In this step the opportunity is identified, which may result in project funding.

In steps 6 and 7 with further concept development the use of computational fluid dynamics (CFD) for reactor optimisation is meant. In the case of IASB development, CFD simulations were not carried out. Basic optimisation was done by building several lab-scale reactors. Although licences for commercial CFD programs (Comsol, Fluent) are expensive, one should verify whether a more prominent role should be given to CFD to prevent the construction of

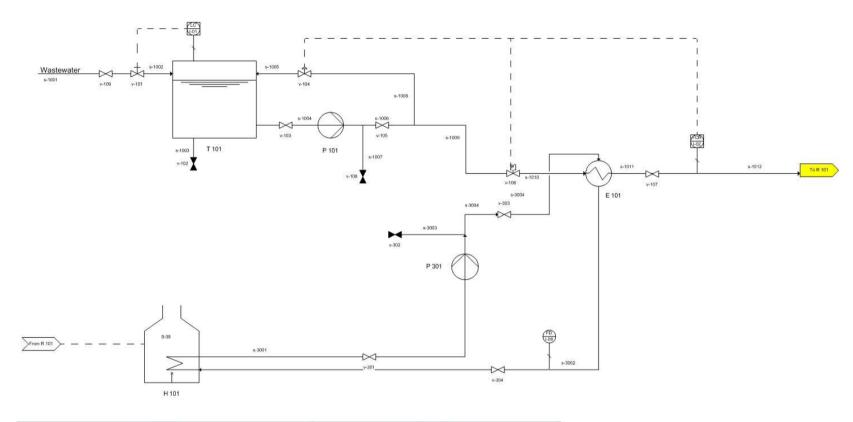
too many lab reactors. Specifically the time that might be saved can show to be a money saver in the future, since the time to market may be shortened. Besides this, free CFD software is available (OPENFoam) and apparently comparable results can be obtained in the simulation of existing industrial processes (Dr. Ir. Eelco van Vliet, personal communication). One should keep in mind, though, that CFD will not be able to substitute lab and pilot scale testing. CFD has already been applied for SLS pilot design optimisation.

One of the key factors in new reactor development is proper funding. Reactor development simply costs a considerable amount of money and insufficient funding may delay the process of development and increase the time to market. Besides this, it may result in less adequate testing. Finally, there is the question of protecting the technology. Submitting a technology for a patent costs considerable amounts of money, specifically if protection is sought on an international scale. One needs to be aware of the market to be addressed, so that actual costs can be limited. Furthermore, although it is tempting to write an elaborate patent text with a lot of claim, this should be avoided. Extensive texts and a lot of claims contribute to patenting costs. This is due to costs associated to translations and, for European patents, a price that needs to be paid per claim when more than 15 claims are submitted.

For pilot demonstration a 1.2 m<sup>3</sup> reactor was constructed together with a container with all the ancillary equipment required to operate the reactor. The container was envisioned to make the pilot a mobile unit. However, it is the 1.2 m<sup>3</sup> reactor that determined the mobility, since it was simply too big and too heavy to be transported inside the container itself. Therefore, in the opinion of the author, the reactor size should be adapted to improve mobility.

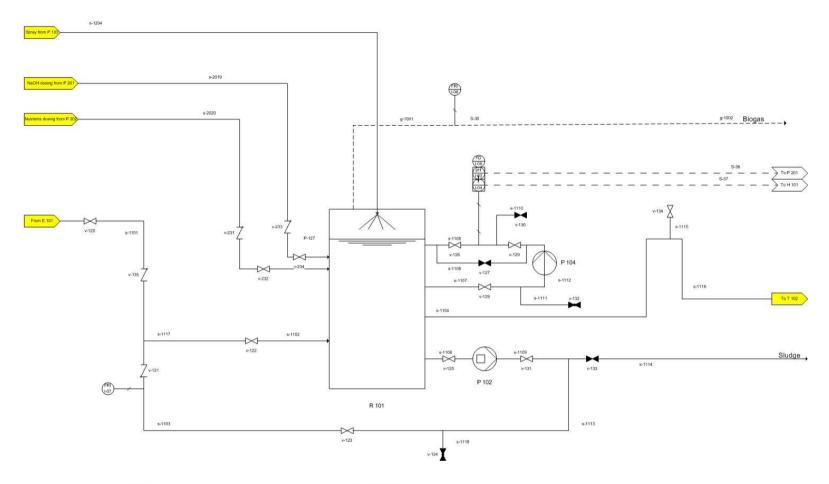
# 8 Appendices

# **Appendix 1: Process & Instrumentation Diagrams of the pilot plant**



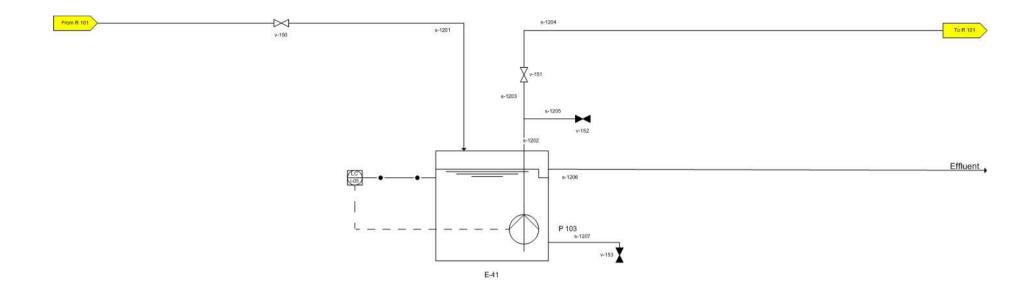
	Equi	oment Lis	t		Instrument List							
Displayed Text	Description	Pump type	Model	Material	Displayed Text	Description	Instrument type					
E 101	Heat exchanger		Not determined	Not determined	I-01	Level control T 101	LC					
H 101	Heater		Not determined	Not determined	I-02	Influent flow rate R 101	FCR					
P 101	Feeding pump	Centrifugal	500 L/h		1-08	flow detector in heating loop	FD					
P 301	Heating pump	Centrifugal	Not determined									
T 101	Influent tank		250 L	HDPE								

Figure 8.1 P&ID of the influent system



	Equip	ment List			Instrument List							
Displayed Text Description		Pump type	Model	Material	Displayed Text	Description	Instrument type					
P 102	Sludge pump	Positive Displacement	500 L/h		1-03	pH control R 101	pH					
P 104	sample loop pump	Centrifugal	10 L/h		1-04	Temperature control R 101	301/15					
R 101	Anaerobic reactor		1000 L		1-06	Flow meter biogas	FR					
5 Albertwoods					1-07	Flow meter sludge	FR					
					1-09	flow detector in sampling loop	FE					

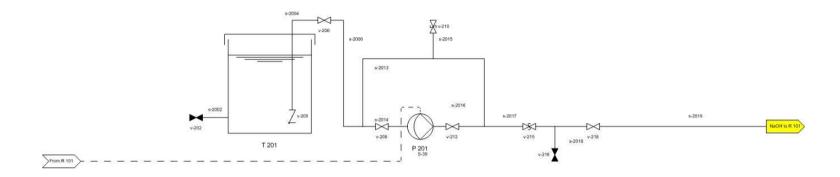
Figure 8.2 P&ID of the reactor

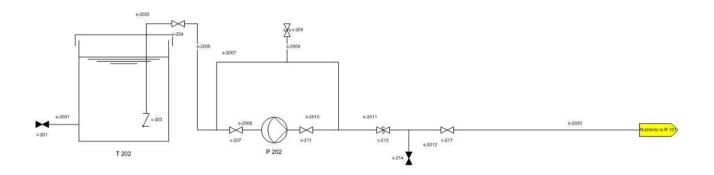


	Equipm	ent List		
Displayed Text	Description	Pump type	Model	Material
E-41	Effluent tank	(1-2-0) I		
P 103	Spray pump	Centrifugal	50 L/h	

	Instrum ent List	
Displayed Text	Description	Instrument type
1-05	On/off control of P 103 through floater ball	LC

Figure 8.3 P&ID of the effluent system





	Equipment List													
Displayed Text	Description	Pump type	Model	Material										
P 201	NaOH dosing pump	Diaphragm	25 L/h											
P 202	NaOH dosing pump	Diaphragm	25 L/h											
T 201	5% NaOH tank		50 L	HDPE										
T 202	Nutrients tank		50 L	HDPE										

Figure 8.4 P&ID of the dosing system

# Appendix 2: Technical drawings of the reactor

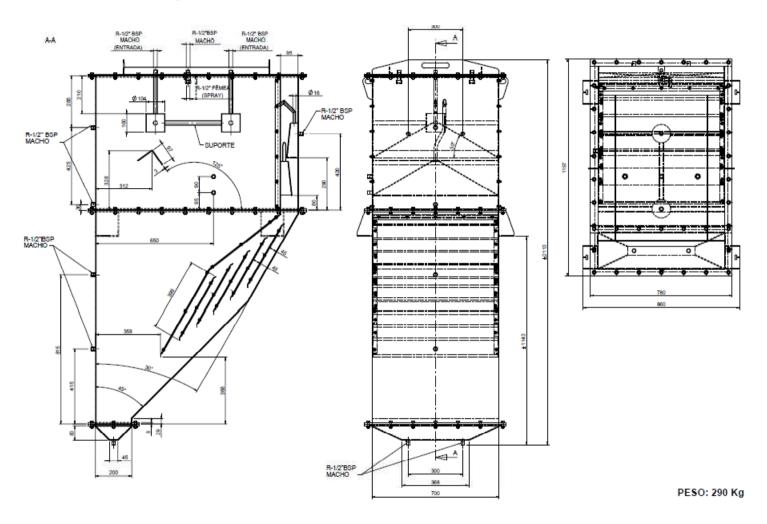


Figure 8.5 Technical drawings of the pilot reactor

# **Appendix 3: Pilot registration sheets**

<i>*</i>		Aı				Sen					ecto				
ambiryr		2ª F	eira	3ª F	eira	4ª F	eira	5ª F	eira	6ª F	eira	Sab	ado	Dom	ingo
	Data Hora														
Ponto de amostragem	Unidade	diluição	valor												
CQO <sub>total</sub> afluente															
CQO <sub>soluv el</sub> afluente															
CQO <sub>total</sub> reactor															
CQO <sub>soluv el</sub> reactor															
CQO <sub>total</sub> saída															
CQO <sub>soluv el</sub> saída															
SO <sub>4</sub> <sup>2-</sup> afluente															
SO <sub>4</sub> <sup>2-</sup> saída															
N <sub>total</sub> afluente															
N <sub>total</sub> saída															
NH <sub>4</sub> <sup>+</sup> afluente															
NH <sub>4</sub> <sup>+</sup> saída															
P afluente															
P saída															

Figure 8.6 Registration sheets of local analyses

		Ano		Semana		Projecto		
<b>ambiry</b> r		2ª Feira	3ª Feira	4ª Feira	5ª Feira	6ª Feira	Sabado	Domingo
Gillon 97	Data						,	
*** ***	Hora							
Ponto de amostragem	Unidade	Valor	Valor	Valor	Valor	Valor	Valor	Valor
TSS afluente								
TSS reactor								
TSS lamas								
TSS saída								
VSS afluente								
VSS reactor								
VSS lamas								
VSS saída								
VFA afluente								
VFA reactor								
VFA saída								
LCFA afluente								
LCFA reactor								
LCFA saída								
-								

Figure 8.7 Registration sheets of analyses done at the Laboratory for Environmental Biotechnology, University of Minho, Braga, Portugal

			no			Sen	nana			Proj	ecto				
<b>ambiry</b> r		2ª Feira		3ª Feira		4 <sup>a</sup> F	eira	5ª F	eira	6ª F	eira	Sak	ado	Dom	iingo
	Data Hora											<u> </u>			
Parámetro	Unidade	on-line	mão	on-line	mão	on-line	mão	on-line	mão	on-line	mão	on-line	mão	on-line	mão
pH afluente															
pH reactor															
T reactor															
Caudal de alimentação															
Recirculação de lamas															
NaOH doseado															
Nutrientes doeado															
Imhoff afluente															
Imhoff reactor															
Imhoff saída do reactor															
Imhoff lamas															
Caudal de biogás															
CH₄ biogás															
CO <sub>2</sub> biogás															

Figure 8.8 Pilot process parameter registration

			2ª Feira	1	3ª Feira		4ª Feira		5ª Feira		6ª Feira	Sabado	abado Domingo		
<u> </u>	Data		* *						* * *		* * *		******		. •
Equipamento	Hora	ОК	Observações	ОК	manutenção C	ЭК	manutenção Ok	<	manutenção	ОК	manutenção	ок	manutenção	ОК	manutenção
Bomba de afluente															
Bomba de recirculação de lamas															
Bomba de alimentação															
Bomba de spray															
Sonda pH on-line															
Sonda pH mão															
Tanque de NaOH															
Tanque de nutrientes															
Tanque de alimentação															
Tanque de saída															
Reactor															

Figure 8.9 Operations verification sheet