MuPFiS, a new tool for characterisation of particulate material in water distribution systems

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RESUMEN

MuPFiS, a new tool for characterisation of particulate material in water distribution systems

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La materia en suspensión (MES) presente en la red de abastecimiento de agua potable es una de las causas principales de las quejas de los usuarios por problemas de coloración y sabor del agua. Además, la MES puede provocar problemas de salubridad de índole bacteriológico. La composición y la concentración de la MES varían a lo largo de una red de abastecimiento de agua potable.

Esta tesina se centra en el desarrollo de una nueva herramienta, el Multiple Particle Filtration System (MuPFiS), para la determinación y la mejor compresión del comportamiento de la MES en una red de abastecimiento de agua. Este trabajo ha permitido desarrollar y validar el MuPFiS y su protocolo operativo mediante ensayos de laboratorio y trabajos de campo.

Los ensayos preliminares han permitido desarrollar y validar el método analítico así como comparar diferentes tipos de filtros. En primer lugar, se han definido las condiciones estándares de filtración: filtración durante 2 horas, caudal superior a 20 l/h y un gradiente de presión de 2 bars. A continuación, se ha determinado la precisión del MuPFiS según las condiciones estándares anteriormente definidas para la medición del residuo seco y la fracción inorgánica de la MES retenida. Finalmente, se han elaborado unas guías para la elección del filtro.

Una vez definido el método analítico, se ha realizado una campaña de ensayos en diferentes emplazamientos de la red de abastecimiento de agua potable holandesa. Los ensayos se han realizado en Delft, Nieuwegein, Amstelveenseweg y Gouda entre los meses de mayo, junio y julio del 2008. En primer lugar, los ensayos realizados en Nieuwegein y el laboratorio de TUDelft han permitido registrar variaciones en la concentración de MES a lo largo del tiempo. Estas variaciones han sido atribuidas a la formación de partículas por post-floculación y corrosión de las canalizaciones durante episodios de bajo caudal. En segundo lugar, los ensayos realizados en la red de abastecimiento de Gouda han permitido registrar variaciones de la concentración de MES en distintos emplazamientos. Estas variaciones son causadas por la formación y desprendimiento de la biopelícula, la post-floculación y la corrosión de las canalizaciones.

En general, los resultados obtenidos en la red de abastecimiento de agua potable holandesa muestran concentraciones de MES bajas (6,6 a 15,5 µg/l) en comparación con los resultados obtenidos (10 a 44 µg/l) por investigaciones llevadas a cabo en otros países (Gauthier et al., 2001, Inoue et al., 2004, Matsui et al., 2007). Estas bajas concentraciones de MES registradas en Holanda son debidas a la aplicación de tratamientos de agua con múltiples etapas de filtración que permiten obtener un agua biológicamente estable.

Finalmente, el MuPFiS se ha comparado con otros métodos existentes como el contador de partículas y el Time Integrated Volume Sampler (TILVS). La comparación con el TILVS ha demostrado que el MuPFiS es un método más versátil. El MuPFiS permite realizar ensayos de menos duración y además sus múltiples líneas de filtración permiten mayor flexibilidad en los ensayos. La comparación con el contador de partículas ha permitido relacionar la masa de MES retenida (MuPFiS) con el número y el volumen de MES presente (contador de partículas) en el agua.

ABSTRACT

MuPFiS, a new tool for characterisation of particulate material in water distribution systems

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Suspended solids (SS) in drinking water distribution systems (DWDS) are one of the main causes of organoleptic and discoloration concerns affecting water customers. In addition they play an important role in the deterioration of the bacteriological quality of water. SS composition and concentration change along a DWDS.

This project focuses on the development of a new method, the Multiple Particle Filtration System (MuPFiS), for the characterization and better understanding of SS behaviour in DWDS. This project has served to develop and validate the MuPFiS and its analytical methodology using laboratory and field tests.

Preliminary tests have served to develop and validate the analytical method and to compare results with those obtained using different filter types. First of all, the MuPFiS' standard filtration conditions were defined: 2-hour filtration run, filtration rate higher than 20 l/h, and trans-membrane pressure of 2 bars. Then, MuPFiS' accuracy for the measurement of TSS and FSS concentration was identified according to standardized procedures. Finally, guidelines for selecting the most appropriate filter type were developed.

A testing program has been conducted at different times and locations of the Netherlands' DWDS following the analytical method previously defined. Tests have been carried out at Delft, Nieuwegein, Amstelveenseweg and Gouda in May, June and July 2008. Tests carried out at Nieuwegein and at the water laboratory of TUDelft have recorded variations of SS concentration with time. This time variations are caused by particle formation, post flocculation or pipe corrosion during low flow conditions in pipes. Tests carried out at the DWDS of Gouda have recorded variations of SS concentration along the network. SS concentration variations were caused by biofilm formation and sloughing, post-flocculation of particles, and pipelines corrosion.

In general, SS concentrations detected in Netherlands' DWDS were low (6.6 to 15.5 µg/l) compared to values obtained (10 to 44 µg/l) by international research (Gauthier et al., 2001, Inoue et al., 2004, Matsui et al., 2007). These low SS concentrations are the result of the systematic application of multiple filtration steps to obtain biologically stable water in the Netherlands.

Finally, the MuPFiS has been compared to other methods currently used for the characterisation of SS, such as the particle counter and the Time Integrated Volume Sampler (TILVS). TILVS' comparison has shown that the MuPFiS is a more versatile tool; filtration runs are shorter and multiple filtration lines offer larger analysis possibilities. On the other hand, a comparison with the particle counter method has allowed a preliminary evaluation of the SS mass retained (MuPFiS) with regard to the number and the volume of SS present in water (particle counter).

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1 INTRODUCTION

In developed countries, drinking water is produced and supplied to consumers using collective centralized systems. Centralized systems are preferable to individual systems because they enhance water quality and safety, and they reduce costs. Commonly, a drinking water supply system is divided in two parts; production and distribution (Figure 1.1).

The production part consists of an abstraction system and a treatment step of raw waters to satisfy drinking water quality standards. A drinking water distribution system (DWDS) is made of three parts: 1) a transport network that conveys water from the treatment facilities to the storage reservoir, 2) a storage reservoir that buffers the daily consumption variations and 3) a distribution network that delivers water to consumers.

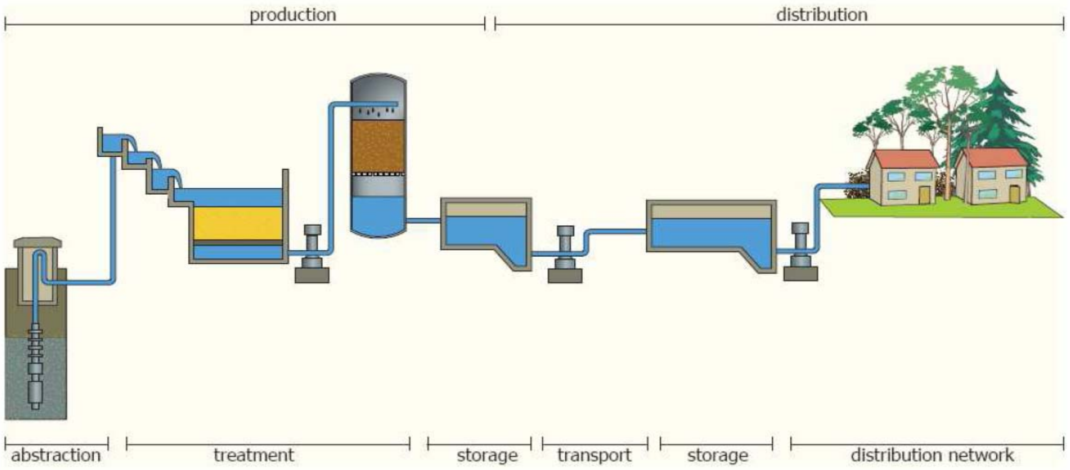


Figure 1.1. Drinking water production and distribution scheme.

The Netherlands are mainly constituted by the delta of the Rhine and Meuse rivers, therefore, the soil primarily consists of poorly permeable sedimentary clay and peat-soil. Fresh groundwater exists in areas where rainwater infiltration occurs. The average rainfall is 740 mm and the evaporation is about 540 mm per year. The Netherlands' flat surface promotes that almost the entire mean rainfall surplus, 235 mm a year, ends up infiltrated in the soil. About 2/3 of drinking water supplies are abstracted from groundwater and 1/3 is collected from surface water (Rhine and Meuse rivers). In the Netherlands, 99.8 % of households are connected to a DWDS (de Moel et al., 2006).

1.1 Problem definition

Water quality is controlled by treatment plants. Although water quality delivered by treatment plants is considered to be constant (or at least within drinking water quality specifications), water quality at consumer taps may vary (Matsui et al., 2007, Hamilton et al., 2006, Prevost et al., 2005). Usually, water at consumer taps has a lower quality than originally treated water, and in extreme cases may surpass non drinking water specifications.

Consequently, some of these water quality variations lead to consumer complaints. Typical consumers' complaints are water discolouration and organoleptic problems. For instance, a typical listing of customers contacts to a UK water company shows that discoloured water (turbid water) is the reason for 34 % of the complaints (Vreeburg and Boxall, 2007).

Previous research (Verberk et al., 2006, Vreeburg, 2007) has shown that suspended solids (SS) in drinking water are an important cause of discolouration episodes and that they play an important role in water organoleptic quality and bacteriological problems.

Undesirable amounts of SS present in drinking water can be traced back to different sources in the production and distribution systems. SS can escape from production processes, because of their incomplete removal during treatment or by their incorporation at treatment plants (Gauthier et al., 2003). Furthermore, SS can also appear in the DWDS. For a long time, distribution networks have been considered as an inert part of the drinking water supply process. However, water composition in DWDS usually changes due to biological, chemical or physical reaction processes taking place between pipes and flowing water (Vreeburg, 2007).

2 OBJECTIVES

The main objective of this study is to contribute to the development of a new analytical tool, called Multiple Particle Filtration System (MuPFiS), for better understanding the presence of SS and thus preventing water quality deterioration in DWDS.

The specific objectives of this study are:

1. To develop a new tool capable of characterising SS in DWDS.
2. To define a MuPFiS' protocol that provides accurate and comparable results by verifying the main parameters affecting filtration: type of filter, duration and pressure.
3. To carry out field filtration tests to associate MuPFiS' results with processes occurring in DWDS (post-flocculation, biofilm formation and sloughing, and corrosion) that affect the concentration and the composition of SS in DWDS.
4. To compare the MuPFiS method with currently used methods.

2.1 Outline of this report

Chapter 3 of this report presents an introduction to water quality in DWDS. Firstly, different compounds in water are presented. Then European, French, Spanish and Dutch quality standards for drinking water are compared. Afterwards, processes affecting water quality variations in the DWDS are summarized. The final section of this chapter summarizes previous research concerning water quality in DWDS.

Chapter 4 describes available methods for monitoring water quality in DWDS; the principles employed, the methods used in this project and other available techniques are also presented.

Chapter 5 presents and reviews laboratory tests carried out with the MuPFiS method, together with validation tests, first filtration tests and comparison tests with the particle counter and the TILVS.

Chapter 6 presents field tests performed with the MuPFiS method in transportation and distribution networks in the Netherlands.

The final chapter outlines the conclusions and recommendations of this study.

3 WATER QUALITY IN DISTRIBUTION SYSTEMS

This chapter offers first an introduction concerning compounds present in water, and then presents water quality parameters in drinking water, water quality evolution in DWDS, and previous research on the subject.

3.1 Compounds in water

Compounds present in water can be classified into: suspended solids, dissolved compounds, vegetable matter, higher organisms and pathogenic organisms.

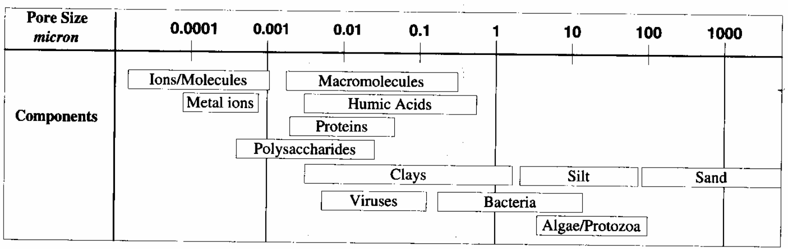


Figure 3.1. Size of typical particles in water (Ravazzini, 2008).

3.1.1 Suspended solids

Suspended solids (SS) consist of particles which have not been dissolved in the water. SS can be distinguished from dissolved and colloidal substances by means of the size of the particles (Figure 3.2).

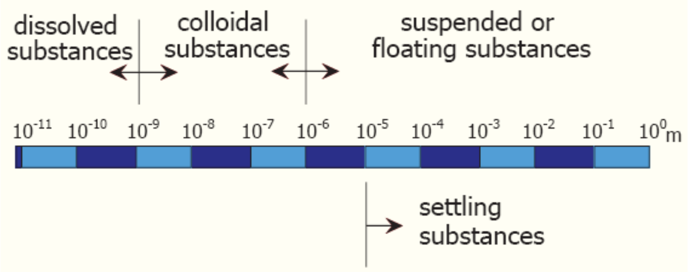


Figure 3.2. Dimension of compounds in water (de Moel et al., 2006).

SS are one of the main causes of discolouration and organoleptic problems in drinking water. In addition, SS are suspicious of transporting bacteria and protecting them from disinfectants.

SS originates, disappears or moves by different ways depending on the characteristics of particles. So far, aquatic SS are extremely heterogeneous with respect to size, density, shape, chemical composition, surface charge, etc (Inoue et al., 2004).

SS can be of mineral or organic origin. Typically, mineral SS originate from sand, clay, loam and other inorganic soil, while organic SS originate from the decay of vegetation and from the discharge of untreated domestic and industrial wastewater (de Moel et al., 2006).

The quantity of SS varies largely depending on the type of water. Drinking water in the Netherlands has a SS concentration of 20 to 60 µg/l, European rivers have a concentration of about 30 mg/l and tropic rivers may contain up to 10 000 mg/l.

The quantity of SS can be determined using different methods; turbidity, Total Suspended Solids (TSS) concentration and number of particles per size.

3.1.2 Dissolved compounds and colloids

Colloidal compounds are particles having a diameter between 10^{-9} and 10^{-6}m. They generally have a negative electrical charge and their density is similar to the one of water. Dissolved compounds have a diameter below 10^{-9}m. Dissolved compounds are classified in organic and mineral compounds. So far, they can be also classified in macro-pollutants if the concentration is over 1 mg/l or micro-pollutants if their concentration is under 1 mg/l. Dissolved solids can be referred as Total Dissolved Solids.

3.1.3 Vegetable matter

Different kinds of vegetables can be found in water, from large size (hyacinths) to small size (phytoplankton).In general they are not harmful to human health, but they impact the appearance of water (colour, taste and odour).

3.1.4 Higher Organisms

Higher organisms can be found in drinking water. They can grow in treatment plants or in DWDS when there are nutrients in water.

3.1.5 Pathogenic organisms

Pathogenic organisms are not present in water by nature, they are introduced by feces or urine from humans and animals.

3.2 Drinking water quality parameters

In France, the Netherlands and Spain the water quality is regulated following the parameters established by the European directive (Table 3.1).

The Drinking Water Directive (DWD) 98/83/EC concerns the quality of water intended for human consumption. The aims of the directive are to improve the quality of water systems and the sustainable us of water. The DWD 98/83/EC appeared on 1998 and replaced the DWD 80/778/EEC within a period of two years.

In France the directive 98/83/EC was transposed into the “décret 2001-1220”. France legislation has more stringent values for colour and turbidity.

In the Netherlands the Water Supply Act and the Decree on the Water Supply sets the standards for drinking water. This decree is within the parameters of the European directive

EU 98-83-EC. Compared to the European directive, the Netherlands Water Supply is stricter concerning dry residues, fluoride, sodium and sulphates.

In Spain drinking water quality is established by the “Real Decreto 140/2003”. This directive sets similar parameters compared to the European directive.

Table 3.1. European Union, Netherlands, France and Spain drinking water parameters.

	European Union	**Netherlands**	**France**	**Spain**
	(Drinking Water Directive 98/83/EC)	*(Decree on the Water Supply)*	*(Décret 2001-1220)*	*(Real Decreto 140/2003)*
Turbidity	Acceptable to consumers and no abnormal change, for surface water treatment a parametric value not exceeding 1.0 NTU	from pumping station FTU<1 from taps FTU<4	from taps NTU<1, for surface waters or karstic waters except an augmentation due to conditioning of softening treatment	from treatment plant or reservoir UNF<1 in distribution network<5 UNF
TOC	No abnormal change, does not need to be measured for supplies of less than 10 000 m³ a day.	No abnormal changes	No abnormal changes	No abnormal changes
Colour	Acceptable to consumers and no abnormal change	20 mg/l Pt/Co	15 mg/l Pt/Co	15 mg/l Pt/Co
Odour	Acceptable to consumers and no abnormal change	Tolerable level for consumers and no abnormal changes	No abnormal changes, no detected odour for a dilution of 3 at 25ºC	No abnormal changes, no detected odour for a dilution of 3 at 25ºC
Taste	Acceptable to consumers and no abnormal change	Tolerable level for consumers and no abnormal changes	No abnormal changes, no detected odour for a dilution of 3 at 25ºC	No abnormal changes, no detected odour for a dilution of 3 at 25ºC

Turbidity is the most commonly used parameter to control SS, however it has some limitations. Turbidity measures the amount of 90° light scatter from particles in a sample. Therefore, it does not offer information concerning particle size and when measuring very low concentrations can not be accurate enough.

3.3 Water quality evolution

Main processes occurring in DWDS that may vary the water quality (Figure 3.3):

- Sedimentation and resuspension of particles.
- Post treatment precipitation of small primary particles and flocculation to larger particles.
- Biofilm formation and sloughing.
- Corrosion and leaching of chemical components from cast-iron or cementous pipe walls.
- Incidental resuspension of old sediment and biomass.

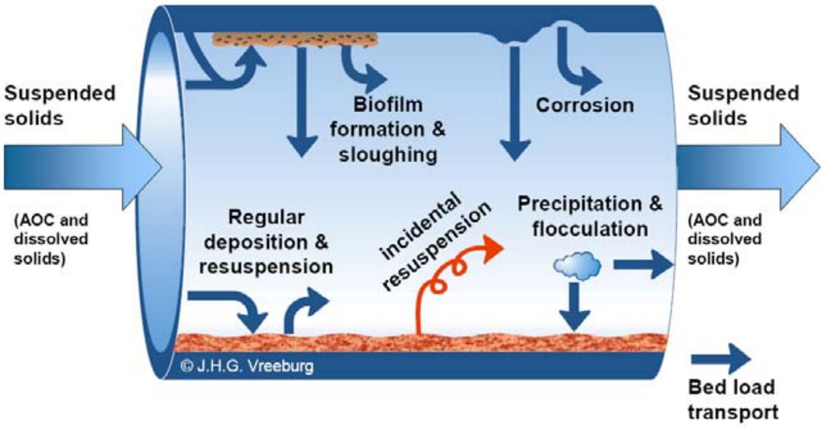


Figure 3.3. Overview of processes in DWDS influencing the water quality (Vreeburg, 2007).

3.3.1 Sedimentation and resuspension of particles

Sedimentation is the phenomenon where particles settle under the influence of the gravity force. Sedimentation in water depends on the characteristics of particles (grain size, density and shape) and on fluid proprieties (density, viscosity, flow rate and turbulence).

Particles are assumed to settle according to the Stokes' Law for low flows. Particles with larger density settle faster than particles with a lower density. Whereas, for turbulent flows sedimentation of small particles can not be successfully described. In addition, supplementary difficulties appear when using the sedimentation theory for particles in DWDS (Vreeburg, 2007).

(Vos, 2005) has shown that the theory of Stokes is difficult to apply for settling of sediment in drinking water. For instance, settling can be influenced by the drag force over the particle created by the water flow. Furthermore, temperatures in drinking water network can fluctuate from 0 to 22 ºC, thus viscosity of water is highly modified.

3.3.2 Post treatment precipitation of small particles and flocculation to larger particles

Flocculation of particles can occur due to different causes; particles coming from the treatment plant can flocculate due to chemical processes not in equilibrium yet, particles coming from different water mains due to different chemical compositions, etc.

For instance, unremoved manganese during water treatment can precipitate in DWDS. Manganese is not oxidized by air at neutral pH, thus is not removed during water treatment processes unless a chemical oxidation step is included. Afterwards manganese oxidizes and precipitates in DWDS due to both chemical and microbial processes (Sly et al., 1990).

3.3.3 Biofilm formation and sloughing

Biofilm occurs because of the presence of assimilable organic carbon (AOC) in water or on pipe walls.

Surfaces adsorb dissolved organics when they are in contact with water, micro organisms get fixed on surfaces and then biofilm is formed. Therefore, biofilm is the consequence of a complex interaction between the pipe material and water. That interaction is characterized by the nutrient level, disinfectant type and level, temperature and hydraulics.

Biofilm develops in all commonly materials used in DWDS (metals, polymers and cement).

The degree of bacterial attachment on surfaces depends on the physical and chemical characteristics of the materials. For instance, surface roughness has been identified as an important parameter affecting biofilm. High rough materials create more dense biofilms.

From experiments realized with different materials it has been shown that (Prevost et al., 2005):

- Plastic pipes sustain less attached biomass than iron-based materials.
- Attached bacteria densities are smaller on cement-lined pipes than on iron-based materials.
- It has been found that stainless steel and copper are the materials that generally sustain lower densities of attached bacteria.

So far, Biofilm growth is also related to disinfectant residual concentrations and flow conditions:

- Locations with no chlorine and stagnant water are subject to bacterial regrowth problems (Wolfaardt and Cloete, 1992).
- an abrupt change in flow conditions may lead to significant increase in effluent suspended bacteria concentration (Laurent, 1995).

3.3.4 Corrosion and leaching of chemical components from pipe walls

Corrosion consists on the release of pipe wall material due to chemical reactions. Pipe corrosion causes a weakening of the pipe and a release of particles into DWDS (Kivit, 2004).

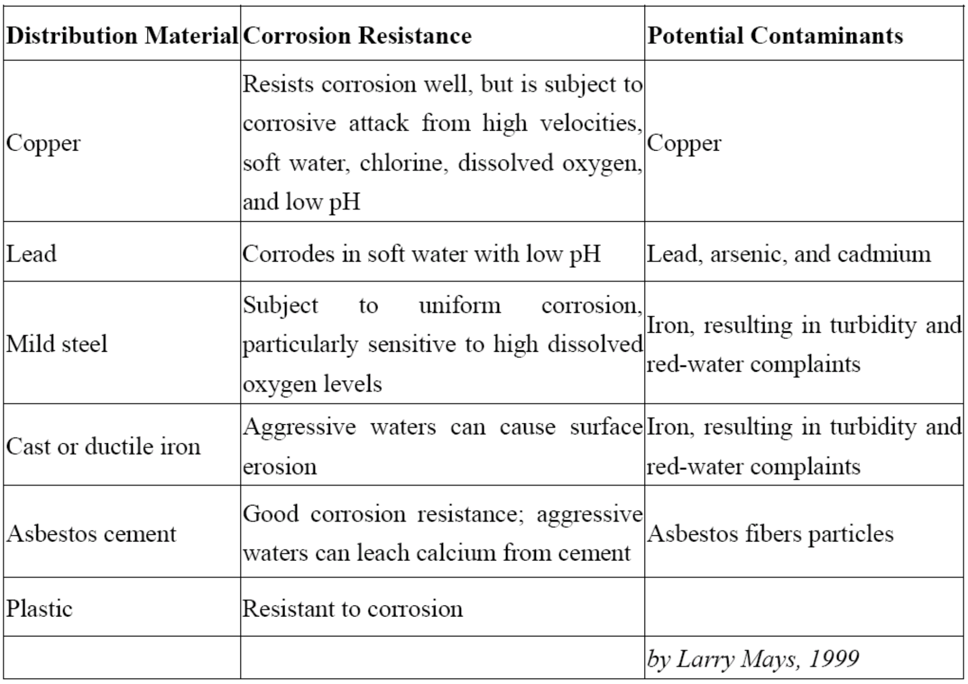
Corrosion in DWDS is affected by different factors (Benjamin et al., 1996):

- Water quality parameters such as pH, alkalinity, dissolved oxygen concentration, TDS, hardness, chloride, sulphate, ammonia, hydrogen sulphide, colour, NOM, copper and magnesium.
- Temperature, flow conditions and residence time.
- Pipe surface material (Table 3.2).

In addition, corrosion plays an important role in organic matter formation (Prevost et al., 2005):

- Metallic surfaces and corrosion deposits provide attachment sites for bacteria. Steel or iron surfaces corroded harbour more biofilm under the same operational conditions than do plastic or cement pipes.
- Corrosion particles are released into DWDS where they can accumulate and foster the development of aggregated bacteria in areas away from the corrosion sites such as plastic pipes or reservoirs.
- Biofilm on iron surfaces can survive in presence of chlorine and chloramines residuals (LeChevallier et al., 1990).

Table 3.2. Corrosion properties of pipe materials (Lahlou, 2002).



Distribution Material	Corrosion Resistance	Potential Contaminants
Copper	Resists corrosion well, but is subject to corrosive attack from high velocities, soft water, chlorine, dissolved oxygen, and low pH	Copper
Lead	Corrodes in soft water with low pH	Lead, arsenic, and cadmium
Mild steel	Subject to uniform corrosion, particularly sensitive to high dissolved oxygen levels	Iron, resulting in turbidity and red-water complaints
Cast or ductile iron	Aggressive waters can cause surface erosion	Iron, resulting in turbidity and red-water complaints
Asbestos cement	Good corrosion resistance; aggressive waters can leach calcium from cement	Asbestos fibers particles
Plastic	Resistant to corrosion	
		by Larry Mays, 1999

3.3.5 Incidental resuspension of old sediment and biomass

Resuspension occurs when particles settled in drinking water pipes are resuspended due to hydraulic changes. Hydraulic changes in DWDS can be caused by a pipe burst, a valve switch, a fire hydrant or even a fluctuation of the daily water demand.

Resuspension happens when forces caused by the flow of a fluid are larger than the forces of the own weight of a particle captured inside a sediment bed under water.

Once a critical stress is reached particles begin to move, this critical stress, depends mainly on the size and density of the particle and secondly on their shape and the cohesive forces acting between particles.

Those particles have a complex movement, a component parallel to the flow and a component perpendicular to the flow.

A lot of studies have analyzed resuspension of particles. Mainly, theories confirm that for bigger particles the density and size of the particle are the main factors to consider, and for smaller particles resuspension depends on cohesion and adhesion forces.

Usually, in DWDS sediments are composed of organic matter and flocs that leads to small and light particles. These kinds of particles tend to settle very slow and resuspend easily.

As it was said for the sedimentation of particles in DWDS, nowadays, there is no widely accepted equation for sediment resuspension in DWDS (Grefte, 2005, Wang, 2006).

3.3.6 Conclusion of the different processes

All processes take place simultaneously. The dominating main process is depending on local circumstances and needs. Those can be determined by water quality measurements at different locations in a DWDS.

3.4 Previous research

Previous research on DWDS has been mainly focused on operational aspects and disinfection. Recently, some research has been started concerning the variations of water quality in DWDS and especially on the behaviour of SS.

Commonly, particulate water quality has been only monitored measuring turbidity. Turbidity is an indirect measurement and only gives indications about quality and quantity.

Nowadays, research has been carried using particle counters (Verberk et al., 2006) and has focused on mass concentrations and chemical compositions of SS in DWDS (Inoue et al., 2004, Matsui et al., 2007, Gauthier et al., 2001, Verberk et al., 2006, Nguyen et al., 2002).

(Gauthier et al., 2001) compared samples of two different locations, Montreal and Nancy using apparatus as shown in Figure 3.5. The sampling points were located after the treatment plant and within DWDS. Turbidity, TSS concentration, and chemical composition of SS were analyzed. Gauthier measured TSS concentrations of 34 µg/l after Nancy treatment plant and 39 µg/l after the Montreal treatment plant. TSS concentrations increased, 44 µg/l, in the DWDS of Nancy and decreased, 27 µg/l, in the DWDS of Montreal.

(Nguyen et al., 2002) analyzed TSS concentration at Highett, Australia. Samples were taken at a fire hydrant. Results showed concentrations of SS between 110 and 740 µg/l.

(Inoue et al., 2004) sampled two treatment plants and its distribution networks in Japan using apparatus as shown in Figure 3.4. Tests analyzed the particle size, TSS concentration and chemical composition of SS. Concentrations found after water treatment plant and in DWDS varied from 10 to 30 µg/l.

(Verberk et al., 2006) analyzed particle size, TSS concentration and chemical composition of SS in the transportation network of Amsterdam using apparatus as shown in Figure 3.6. Sampling points were settled after the treatment plant and at some pumping stations (PS). Results showed a TSS concentration of 13 µg/l after the treatment plant, whereas the concentration of TSS fluctuated between 33 and 71 µg/l at the pumping stations.

(Matsui et al., 2007) took samples at the DWDS of Nagoya city and Wakayama City in Japan using apparatus as shown in Figure 3.4. Tests analyzed TSS concentration, chemical composition of SS and residual chlorine concentrations. TSS concentrations between 10 and 40 µg/l where found in the DWDS.

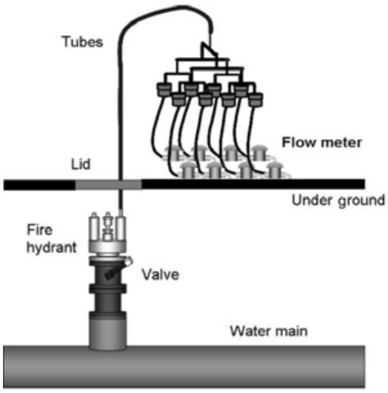


Figure 3.4. Sampling method with 8-line filtration system (Matsui et al., 2007, Inoue et al., 2004).

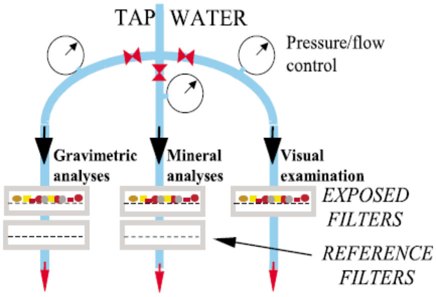


Figure 3.5. Principle of the particle sampling system (Gauthier et al., 2001).

Figure 3.6. Time Integrated Large Volume Sampler (TILVS) (Verberk et al., 2006).

4 EXPERIMENTAL SETUPS

This chapter describes apparatus and methods currently available for the analysis of SS in water. Available methods are classified into filtration methods, particle counting and other methods.

First of all, this chapter presents the filtration method and tools using that principle. Thereafter, describes the particle counting methodology and the particle counter used. Finally, presents other analysis methods such as the ICP-MS and the Image analysis. The MuPFiS, the TILVS and the particle counter are quantitative apparatus, while, the ICP-MS is a qualitative methodology.

4.1 Filtration methods

4.1.1 Practical definition of suspended and dissolved solids

The total amount of solids present in water is composed by a fraction of suspended and dissolved solids.

Total solids (TS) is the term used to define the material residue left in the vessel after evaporation of a sample. Samples are dried in an oven at a defined temperature of 103 °C.

Total solids is equivalent to the total suspended solids (TSS) the portion of total solids retained by a filter, and total dissolved solids (TDS) the portion that passes through the filter.

$$TS = TSS + TDS$$

Filtration methods are based on the well accepted principle by the environmental scientific community that when analyzing water, the term dissolved refers to the fraction of mater that pass through a 0.45 µm membrane filter. However as it is shown in the next points, the portion of retained matter depends in many factors such as the filter and the filtration conditions.

Total Suspended Solids (TSS) can be divided into Fixed Suspended Solids (FSS) and Volatile Suspended Solids (VSS). Fixed solids are the ones who remain after combustion of the sample at 550 ºC.

$$TSS = FSS + VSS$$

Standard methods previously defined are used for the analysis of solids in water. The experiments in this report are based on the Standard Methods for the examination of water and waste water, 20th Edition (Greenberg et al., 1999). For instance, this method establishes that for the analysis of SS a minimal mass retained of 2.5 mg must be achieved. The Appendix I describes the method in detail.

Filtration methods are used for all kinds of waters, however procedures slightly vary because of the different concentrations of particles in water.

For instance, to retain in the filter a representative mass of 5 to 10 mg a few centilitres need to be filtered for waste water, a few litres for natural waters while up to a few hundred litres are needed for drinking water. Compared to other kind of waters, drinking water needs big amounts of water to obtain the same amount of retained particles in a filter.

Indeed a filter has two functions, firstly separates SS from water and other compounds, and secondly concentrates SS to make possible the analysis.

4.1.2 Theory

Membrane filtration

A membrane is a perm-selective barrier that realizes the separation of components of a given solution.

The transport through a membrane is originated by a driving force. The driving force is usually the gradient of pressure for drinking water; however there exist other driving forces, such as, gradients of concentration, of potential electric or of temperature.

The Trans Membrane Pressure (TMP) measures the gradient of pressure, The TMP is the difference of pressure across the two sides of the membrane (Figure 4.1).

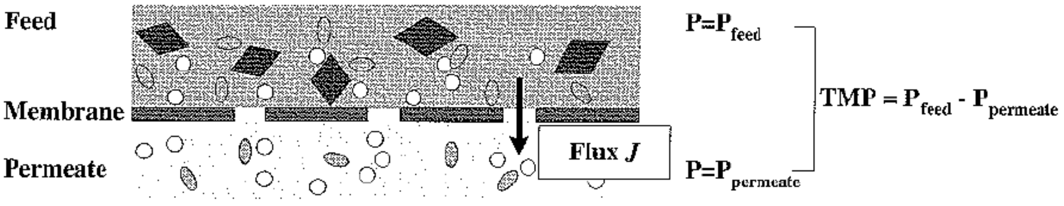


Figure 4.1. Schematic representation of membrane separation (Ravazzini, 2008).

The membrane pore size classifies the pressure driven membrane processes (Table 4.1). From bigger to smaller pore size, four membrane processes exist: microfiltration, ultrafiltration, nanofiltration and reverse osmosis. Indeed, the 0.45 μm membrane filters are within the microfiltration membranes group.

Table 4.1. Classification of membrane processes with pore size.

Process	Pore Size (nm)	Trans Membrane Pressure (bar)
Microfiltration	100-1000	0.1-2
Ultrafiltration	10-100	0.1-2
Nanofiltration	1-10	4-20
Reverse Osmosis	0.1-1	10-30

Process fundamentals

Membranes can be operated within two modes: dead-end or crossflow filtration (Figure 4.2).

In dead-end filtration the flow is perpendicular to the membrane and the entire filtration volume pass through the membrane. While, in crossflow filtration, the feed flow is tangential to the membrane surface and splits into two streams: the permeate and the retentate.

Dead-end filtrations accumulate all the filtrated solids on the surface of the membrane, whereas in crossflow filtration the retentate carries away most of the materials.

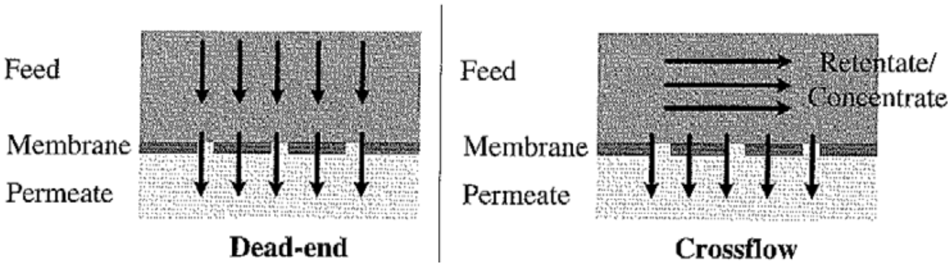


Figure 4.2. Schematic representation of dead-end and crossflow filtration (Ravazzini 2008).

The analysis of SS requires dead-end filtration because it retains all the solids in the filter.

Membrane fouling

Fouling is the loss of performance of a membrane due to deposition of suspended or dissolved substances on its external surface, at its pore openings, or within its pores.

For instance (Figure 4.3) shows the flow decrease of a membrane filtration run.

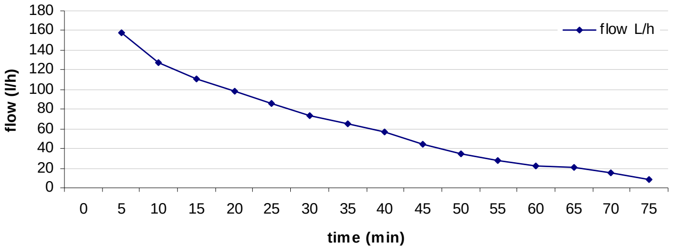


Figure 4.3. Flow variation during a membrane filtration test.

Fouling is a complex phenomenon that includes several mechanisms (Mulder, 1996), (Figure 4.4). Principal mechanisms associated with membrane fouling:

- Concentration polarization: the increase concentration of rejected solutes in the proximity of the membrane surface favours the increase of required transmembrane pressure for operation.
- Pore blocking: particles enter the pores and block them, thus, the number of channels available for permeation is reduced.
- Pore narrowing: particles, colloids and molecules that enter the membrane pores depose/absorb at the walls and reduce the cross section available for permeation.
- Cake layer formation: particles or other material that do not enter the membrane pores accumulate at the surface originating a layer. Depending on the constituents, this layer can be more or less permeable than the membrane.

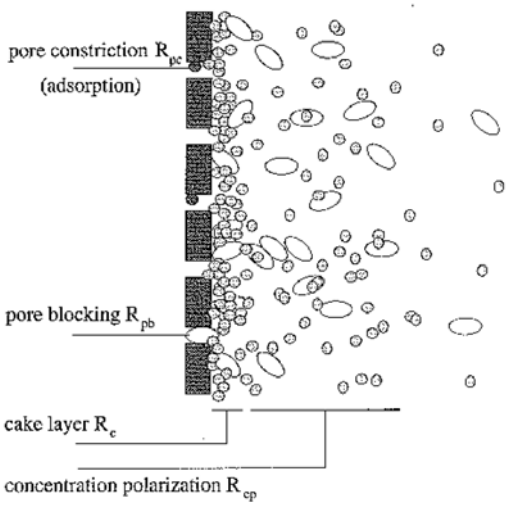


Figure 4.4. Fouling mechanisms (Ravazzini, 2008).

Fouling is mainly affected by three factors:

- Feed water characteristics: particle size distribution, chemical properties of the solutes and chemical properties of the solution.
- Membrane characteristics: pore size, roughness, porosity, structure and chemical properties.
- Operating conditions: permeation drag, pressure, and hydrodynamics.

Filters retain smaller particles when they start clogging because of the reduction of the nominal pore diameter and the formation of the cake layer.

Microfiltration characteristics

The fact that SS is the fraction of matter that passes through a 0.45 µm membrane filter. This is an operational definition and not scientifically based.

Studies concerning the detection of trace element concentrations (Horowitz et al., 1996, Hall et al., 1996) have shown that in special circumstances some dissolved metals such as Fe, Al can be retained in 0.45 µm membrane filters. To better understand these alterations of the universally accepted method some research has been done.

At a first stage, the filtration studies were classified in two categories: chemical and physical effects. Chemical studies looked into the potential contaminants present in certain filter brands and the sorptive capacity of filters that may prevent some matter finer than 0.45µm from passing through them. Whereas, physical studies gave special attention to retention of filters with different pore size.

(Horowitz et al., 1992) proposed a new classification of proprieties affecting filtration process, that new classification not only regarded the characteristics of the filter (pore size, structure, thickness, etc.) but also external factors (size distribution of particles, type of particles, pressure, etc.).

Major variables affecting microfiltration:

- Filter type: tortuous path and sieve filters.
- (Horowitz et al., 1992) illustrated that Fe is sorpt in higher quantities in tortuous path filters than in sieve filters. This is probably due to the length path the solution has to travel until it comes through the bottom of tortuous path filtration membranes. The sorption of dissolved matter may stop when all the potential sorption sites are filled, (Horowitz et al., 1996).
- Small sized particles tend to be trapped in depth filters, (Droppo et al., 1992).
- In general, sieve filters clog before than tortuous path filters (Horowitz et al., 1992, Laxen and Chandler, 1982).
- Finally, (Droppo et al., 1992), has showed that some fibrous materials are released from the tortuous path due to the filtration conditions, to avoid that situation, it is recommended to prewash the filters and avoid high trans-membrane pressures.
- Filter diameter. (Horowitz et al., 1992) illustrated that a same characteristics filter with a bigger diameter retains more dissolved particles because of the augment of the sorption capacity of the filter.
- Filtration method: pressure filtered or vacuum filtered. Greater pressures forces marginally-sized material through the filter, thus, when the pressure is higher the retention is lower, (Horowitz et al., 1992). (Droppo et al., 1992) stated that TMP higher than 5 psi (0.35 bars) might bind particles into and through the filter.
- Suspended sediment concentration. Clogging is determined by the suspended sediment concentration. High suspended sediment concentration accelerates the filter fouling.
- Suspended sediment grain-size distribution. Fine-grained material can accelerate the filter clogging, (Horowitz et al., 1992). Fine-grained material can easily enter and get adsorbed in pores paths compared to big material. As well, fine-grained material will form a cake layer denser with narrower paths through it.
- Concentration of colloids and colloidally associated trace elements. Colloids are capable of sorbing large concentrations of trace elements, thus these dissolved elements that otherwise would pass through the filter because of the colloid interaction now can be retained by the filter.

- Volume of sample processed. The total volume sampled influences the suspended sediment concentration, the suspended sediment grain size distribution and the concentration of colloids.
- Concentration of organic matter, the presence of organic matter in water accelerates the clogging process (Horowitz et al., 1992).

Therefore, it is recommended for pre-concentration methods such as the MuPFiS and the TILVS:

- To use of tortuous path filters to increase the particle retention
- To avoid high TMP during filtration tests to prevent the release of filter material and the escape of some particles through the filter.
- To adapt the choice of filter to the type of water analyzed to control the fouling of the filter. For instance, water with high concentration of organic compounds or water with high quantities of fine-grained material can accelerate fouling.

Types of microfiltration membranes:

Microfiltration membranes are made of organic (polymers) or inorganic materials (ceramics, metals, glasses).

- Organic membranes are made of a large range of materials, offer a low cost and are used for inorganic analysis. However, it is difficult to sterilize them, they are not resistant to solvent, chlorine and ozone and they are not suitable for some analysis procedures because they not resist high temperatures.
- Inorganic membranes have a long life, are very resistant to solvents, chemicals and high temperatures and are used for organic analysis. However they have a high cost.

Most commonly microfiltration membranes used are:

- Cylindrical pores membrane (Figure 4.5).

  Cylindrical pore membranes are composed of parallel cylindrically shaped pores of uniform dimension. This structure is created by track-etching.

  Pores are created by applying perpendicularly to the material a high energy particle radiation. The particles damage the material and create the tracks.

  Normally, this method is employed for polycarbonate membranes.

  Cylindrical pores membranes have a thickness of around 10 µm.
- Closed-cellular morphology membrane (Figure 4.6).

  Closed-cellular membranes are created by the phase inversion technique. This is a process where a polymer is transformed in a controlled manner from a liquid to a solid state. Porous membranes can be created controlling this process.

  Closed-cellular membranes are thicker than cylindrical pores membranes. For instance, 145 µm.
- Glass fiber membrane (Figure 4.7).

  Glass fiber membranes have a complex matrix. Porous paths are tortuous.

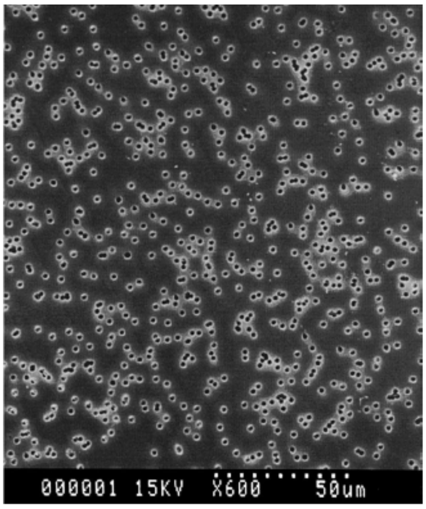


Figure 4.5. SEM image of a cylindrical pore membrane surface (Charcosset and Bernengo, 2000).

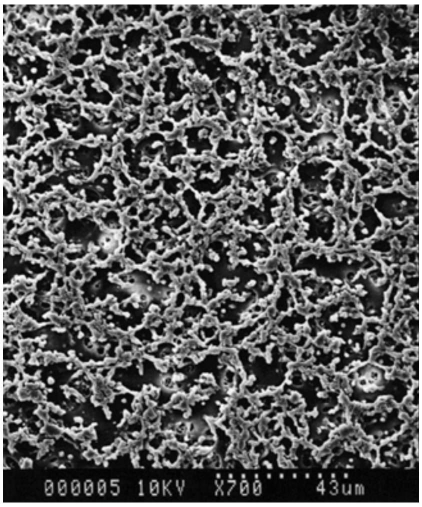


Figure 4.6. SEM image of the cellulose acetate membrane surface (Charcosset and Bernengo, 2000).

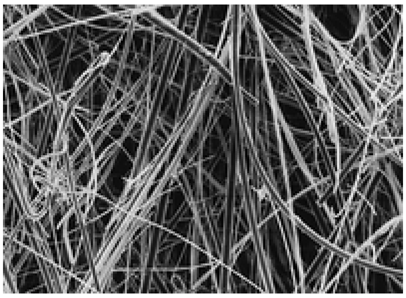


Figure 4.7 .Image of a glass fiber membrane surface (Millipore).

Micro glass fiber membranes have been used for the characterization of TSS and VSS because of their resistance to high temperatures. In Table 4.2 there is a list of the glass fiber filters that have been used in this report and by other authors.

Table 4.2. Types of filters, pore size and users.

Type	Pore size	Supplier	User
QM/A	2.2-µm	Whatman	(Matsui et al., 2007) & MuPFiS
GF/C	1.2-µm	Whatman	MuPFiS
GF/F	0.7-µm	Whatman	MuPFiS
AP40	0.7-µm	Millipore	(Gauthier et al., 2003)
APFD	2.7-µm	Millipore	(Gauthier et al., 2003)

The filter is chosen depending on the conditions of the experiment. Indeed, filters used with the MuPFiS have been selected considering water quality, water pressure and total amount of particles that are collected.

Filters with bigger porous size retain less small particles, thus they retard the fouling process, whereas filters with smaller porous retain smaller particles.

Big pores sized filters are recommended to be used with water of bad quality, high concentrations of SS, and also when the pressure is low, bigger pores filters offer less resistance to filtration. While, small pores sized filters are recommended to be used with waters with extremely good quality or networks with high pressures.

However, as it has been stated before, only results obtained by the same filters are comparable. For example, the GF/F has been used when doing comparison tests with the TILVS.

4.1.3 Time Integrated Large Volume Sampler (TILVS)

The Time Integrated Large Volume Sampler (TILVS) (Figure 3.6) has been one of the first devices developed by TUDelft to analyze the concentration of SS in drinking water.

Figure 4.8. Time Integrated Large Volume Sampler (TILVS).

The TILVS is an online filtration device. The TILVS uses a dosing displacement pump that feeds a constant flow onto a small filter membrane. The pump delivers a constant flow of water, within a range between 0.05 to 5 l/h. During the filtration process the filter fouls and resistance increases, thus, the pump varies the pressure to maintain a constant flow. The filtration unit can withstand up to 10 bars of pressure.

The unit does not sample directly from the pressurized system. The TILVS has a water overflow vessel that allows the pump passively sample from this vessel.

Depending on the flow and on the particle concentration the filtration period can vary between 19 and 168 hours. Filtration time has to be chosen to recover at least 2.5mg of SS (specified by the Standard Methods for Water and Wastewater) and to avoid a trans-membrane pressure (TMP) bigger than 10 bars. The TILVS disposes of a small metallic sieve placed inside the filter holder to avoid the filters damage due to high pressures, however, it is not guaranteed that particles will not traverse the filter because of the high TMP.

Finally, the total volume of water filtered is calculated by integrating the constant flow of water by the time of duration of the test.

The TILVS was developed in 2005 by researchers from TUDelft in the Netherlands and Griffith University in Australia (Verberk et al., 2007).

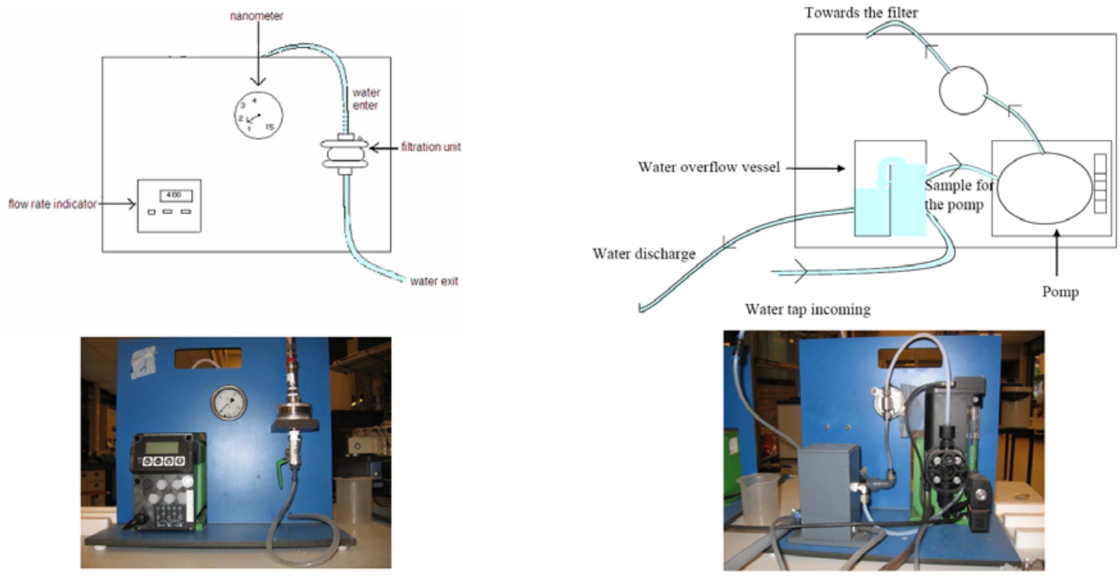


Figure 4.9. TILVS description (Sagel, 2007).

4.1.4 Multiple Particulate Filtration System (MuPFiS)

Description

The function of the Multiple Particulate Filtration System (MuPFiS, Figure 4.10) is to determine the composition of SS. Therefore, SS present in water are collected using membrane filters. These membrane filters are placed within filter-holders (Millipore Swinnex Holder 47mm) that are directly connected to the distribution system.

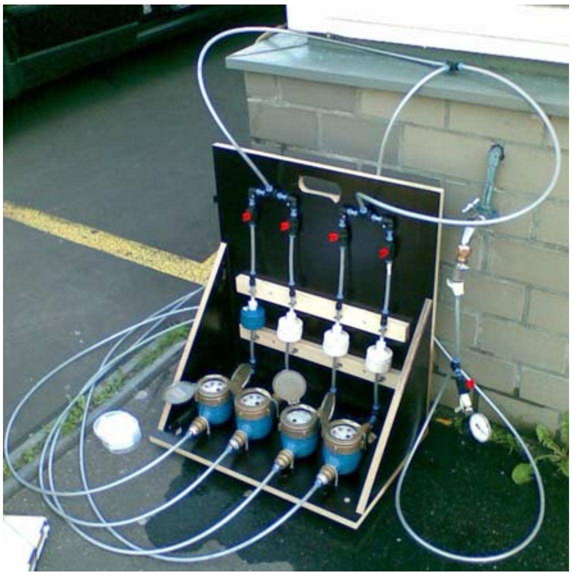


Figure 4.10. Image of the MuPFiS.

A main tube is directly connected to the sampling point of the distribution pipe (Figure 4.11). Then the main tube is connected to a "distribution system" that divides the main flow into four branches, in each of one is placed a filter. Each branch disposes of the necessary elements to operate independently from the other ones (valve, filter and water meter).

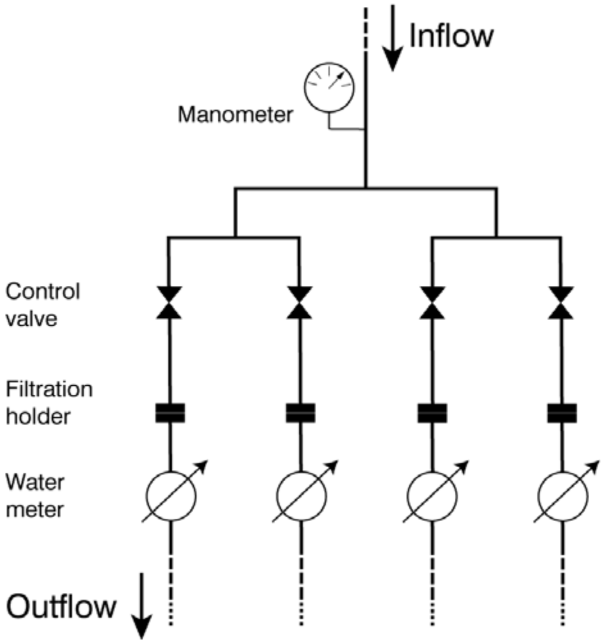


Figure 4.11. MuPFiS' scheme of principle.

So far, the system is constructed with interchangeable elements that offer to the system a flexibility to get adapted to specific requirements. The "distribution system" can be modified and be adapted to the requirements of the sampling point, new elements can be installed on the circuit (pressure meters, valves, T connections, etc).

Operation description of the MuPFiS

DWDS provide enough pressure to make possible the filtration. Usually, in DWDS the pressure is at least 2 bars. That is the starting pressure and during the test the pressure decreases because of the filter clogging. A run should be stopped before reaching a critical pressure.

The water meter used is an Elster MNR Qn 1.5. That water meter whose nominal flow is 1500 l/h has a minimal flow of 20 l/h. Tests might be stopped before attaining the minimal value.

Notice that in a filtration process pressure and flow are correlated, therefore, it is just necessary to control one of them because the other one is directly correlated.

Thanks to having four filters in parallel the system offers the following advantages:

- Running multiple tests in parallel at the same time permits to analyze different parameters of the same water sample.
- Performing tests one after another allows obtaining an on-line monitoring of the SS in water.

Filtration time of each test depends on the water SS concentration, the type of filter and the water pressure of the DWDS.

A standard filtration procedure is defined for the MuPFiS (Appendix I) to make different filtration tests comparable; 2-hour filtration run, filtration rate higher than 20 l/h and TMP of 2 bars.

Finally, the construction of the MuPFiS has been based on previous research (Gauthier et al., 2001, Matsui et al., 2007).

Initial constructions

At a first stage, a prototype was constructed in the laboratory to run the initial testing before constructing the definitive setup (Figure 4.12).

The prototype was constructed thinking of the final setup, which should have a compact size, should be transportable and easy to manipulate.

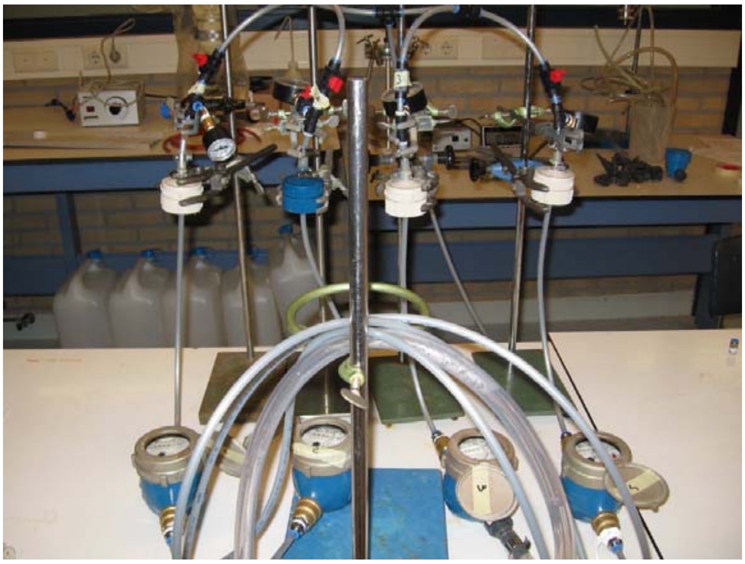


Figure 4.12. MuPFiS initial version

One of the mains objectives of the construction was to construct symmetric paths for each branch to obtain similar losses while running the experiments.

Final setup

The final setup has been constructed on a wood support. The used wood is water proof.

The final version is transportable and can be easily modified to be adapted to new requirements.

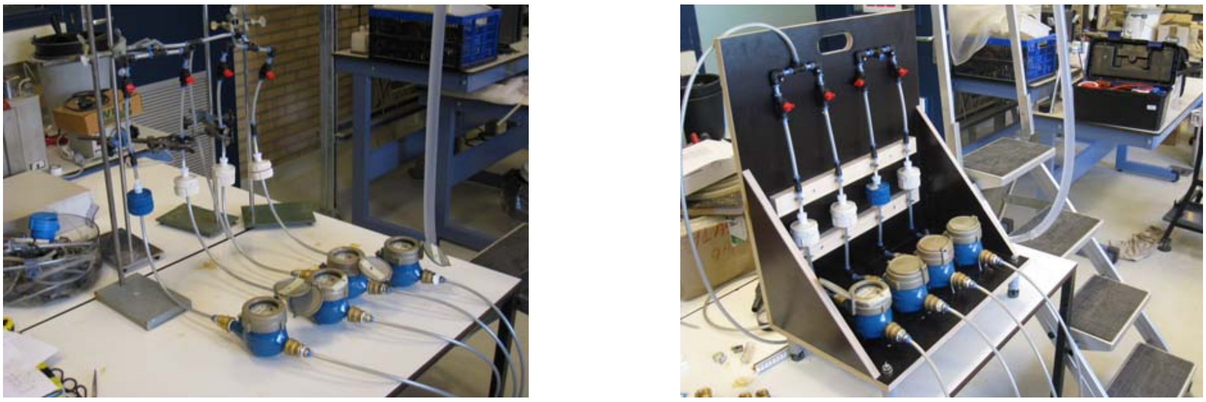


Figure 4.13. MuPFiS, from the prototype to the final setup.

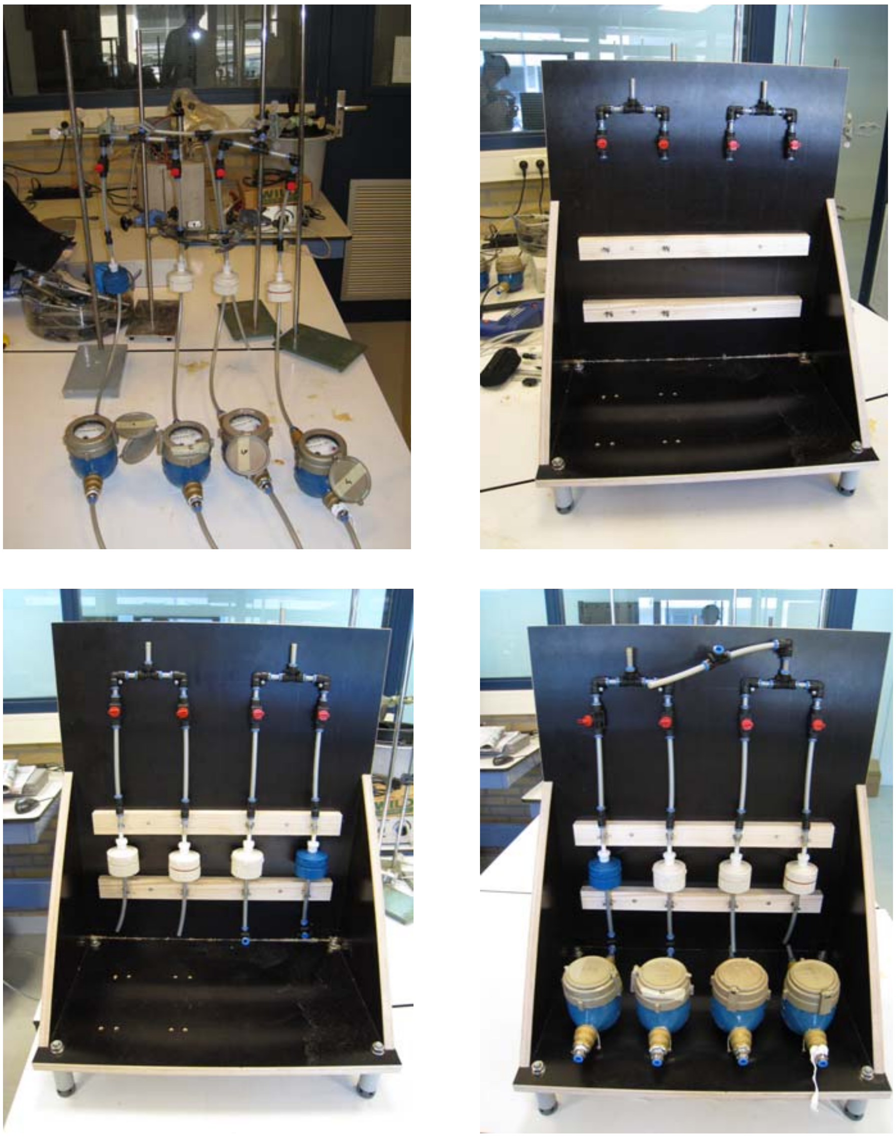


Figure 4.14. MuPFiS, construction of the final setup.

4.1.5 MuPFiS and TILVS comparison

MuPFiS and TILVS are preconcentrating apparatus using microfiltration membranes. TILVS sampling method is based on a fixed flow controlled by a dosing pump whereas MuPFiS principle is based on a constant pressure set by the DWDS. On one hand, MuPFiS' free flow system permits a higher flow compared to the TILVS, therefore the filtration run duration is shorter, but on the other hand the dosing displacement pump permits a better volume measurement accuracy of the TILVS compared to water meters used by the MuPFiS.

No need of electrical power, four filtration lines and shorter duration of filtration runs make of the MuPFiS a more fast and versatile tool.

Table 4.3. Comparison of MuPFiS and TILVS characteristics.

	MuPFiS	TILVS
Filtration lines	4 lines	1 line
Filtration flow	Variable 250 – 20 l/h	Fixed 0.05 – 5 l/h
Filtration pressure (TMP)	Fixed Pressure network (≈2bars)	Variable 0 – 10 bars
Filtration duration	20 min – 4 h	19 h – 1 week
Advantages and disadvantages	+ versatile (4 lines) + faster filtration runs - less precise (mechanical water meter) - filtration flow is not constant	+ precise (dosing displacement pump) - high TMP (until 10 bars) - long duration of filtration runs - needs electrical power - particles are damaged by the pump

4.2 Particle counting

4.2.1 Theory

A particle counter is an apparatus that detects and counts particles in water or air.

Particle counters use two different techniques to detect particles: Light Blocking and Light Scattering. The Light Blocking method has accuracy until one micrometer particles while the Light Scattering technique detects until the nanometer particles. The Light Blocking system is used for the detection of SS in water.

Light Blocking particle counters measure a change in light intensity as particles pass through a laser beam. The shadow cast by each particle is proportional to its size within a defined range (Figure 4.15).

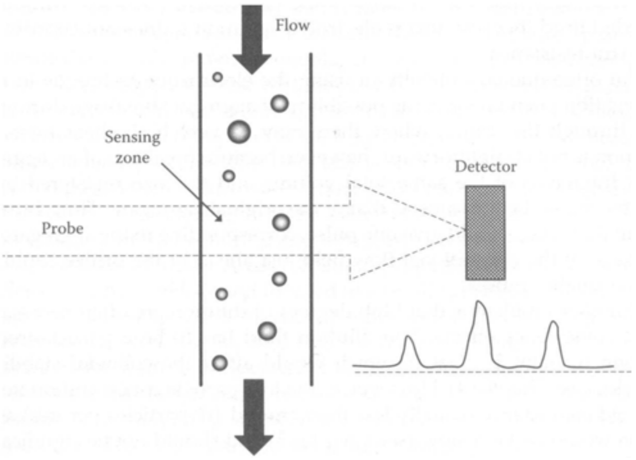


Figure 4.15. Schematic representation of a particle counter's sensor (Gregory, 2006).

4.2.2 Particle counter

Particle counters measure particle size distributions and permit to calculate particle volumes:

- The particle size distribution indicates how particles change along time.
- The particle volume concentration (PVC) is calculated considering that particles have a spherical form. PVC gives an indication of the amount of SS in water.

The analysis of data generated by a particle counter is complicated because of the big volumes of information. Indeed, when using a particle counter data must be treated to retain only representative and relevant information. Even though, after this step data is compressed to make the interpretation easier.

The particle counter used for the experiments is Met One PCX. The particle counter works online and samples a constant flow of 6 l/h.

The particle counter disposes of 224 channels with a resolution of 0.5 μm and gives a set of data every minute. During a minute 100 ml of water are analyzed (6l/h*1min=100ml) Data is given as average of number of particles per ml.

The PVC (0.1) has been calculated for each of the 224 ranges;

$$PVC = \frac{1}{6}\pi \sum_{Bin=1}^{Bin=i} (n_i d_g^3) \qquad (0.1)$$

n_i is the number of particles in a bin

i is the number of bins

d_g is the low diameter delimiting the bin (μm)

For instance, for the bin 12 to 12.5 μm, it has been considered a diameter of 12 μm for the volume calculation.

Particles have been packed into predefined particle size ranges of 2-5, 5-7, 7-10, 10-15, 15-20 and 20-114 µm. For instance, PVC of different ranges have been packed into the predefined particle size ranges.

Number of particles counted under or equal to 0.01 particles/ml have been discarded. Especially when 0.01particles/ml are not considered PVC calculations become more stable.

4.3 Other methods

4.3.1 ICP-MS

Inductively coupled plasma mass spectrometry (ICP-MS) is a mass spectrometry capable of the determination of a range of metals and several non-metals.

ICP-MS is used to analyze the chemical composition of mater retained in filtration membranes.

4.3.2 Image analysis

Image analysis has been used for characterization of particles.

(Lorenzo et al., 2006) have analyzed morphology and chemistry of particles by computer controlled scanning electron microscopy. Particle's volumes were calculated by pixel counting of images. The projected area and longest particle axis were measured by pixel counting. Afterwards, assuming that the particles lie on their largest side rather than on the tip volumes were calculated by approximation of prolate ellipsoids.

This method provides a more accurate estimation of particles volume than considering particles as being spherical.

5 DEVELOPMENT AND VALIDATION OF THE ANALYTICAL METHOD

This chapter describes the MuPFiS' analytical methodology. The new protocol, included in Appendix I, was derived from the experimental tests conducted at TUDelft's laboratory during April and May 2008.

First tests analyzed the MuPFiS filter's behaviour, then, tests were carried out to estimate the accuracy and to validate the MuPFiS. Different types of filters were tested to obtain functional information for future field tests.

Finally, the MuPFiS was tested in parallel with a particle counter. The particle counter comparison permitted to work on the relation between SS mass, number of particles and particle volume concentration (PVC).

TSS and VSS analysis were carried out following the guideline established by the Standard Methods for the Examination of Water and Wastewater, 20th Edition (Greenberg et al., 1999).

5.1 Previous test

Previous obtained a first approximation about the SS concentration in water and the filter behaviour during the filtration process.

First tests analyzed the behaviour of the filtration membranes when filtering drinking water directly from the DWDS. The water flow has been calculated during the filtration run of the 28th April 2008 with the flow meter, by recording every five minutes the accounted volume.

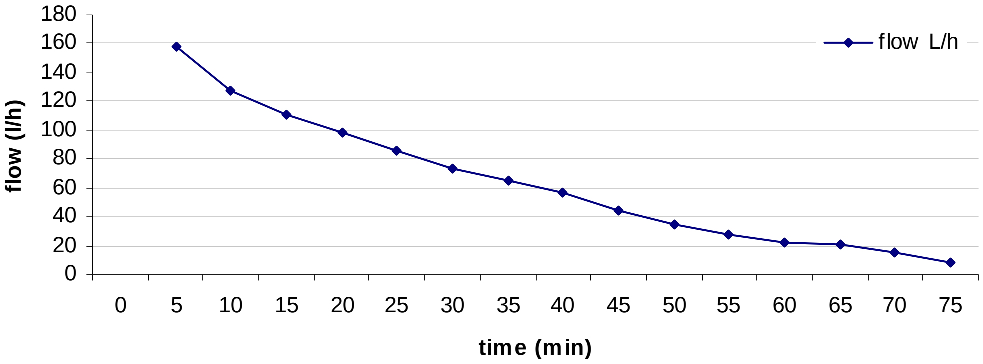


Figure 5.1. Flow variation along time – MuPFiS' test of 28th April 2008.

Results of the 28th April 2008 filtration run (Figure 5.1) show that the flow decreases rapidly from 160 l/h to 10 l/h in 75 minutes because of the filter fouling. Indeed, different filtration runs obtain similar flow/time curves; however, the exact curve depends on the type of filter, the type of water and the filtration conditions (tap pressure).

The MuPFiS filtration runs are limited to a minimal flow of 20 l/h or a minimal TMP of 0.6 bars because of the water meters accuracy limits. Hence, in the filtration run of the 28th April 2008 the filtration run should have been stopped at the minute 65.

When analyzing the previous filtration runs results (Table 5.7), the volume filtered varies significantly from 292 to 78.8 litres depending on the day and time of the filtration run.

Table 5.1. Volume filtered, mass retained and TSS concentration – MuPFiS' previous tests at the TUDelft's water laboratory.

	16/04 @ 11:40	22/04 @ 11:00	24/04 @ 10:00	28/04 @ 10:20	28/04 @ 12:00
Volume filtered (l)	292.7	158.3	134.6	78.8	92.8
Mass retained (mg)	3.0	-	4.3	3.7	4.0
TSS concentration (µg/l)	10	-	32	47	43

The objective of a filtration run is to retain the maximum possible amount of SS in the filter to increase the accuracy of the measurement; MuPFiS' accuracy is described in the point 4.2. Hence, the mass retained on a filter depends on the type of filter, the type of water filtered and filtration conditions. The relation between the type of filter, filtration conditions and the mass retained is studied in detail in the point 4.3.

TSS concentrations obtained during previous tests vary between 10 and 47 µg/l (Table 5.1). Previous results prove that SS concentration in drinking water does vary along time and the capability of the MuPFiS to measure SS variations.

5.2 Accuracy & Validation tests

Low retained masses may induce into errors. Especially when measuring VSS concentration some results can be altered by artefacts of the low masses retained.

First tests analyzed the accuracy of the TSS and VSS method for the MuPFiS and afterwards validation tests checked if obtained results are within the accuracy limits previously established for the MuPFiS.

5.2.1 Accuracy estimation of the TSS and VSS methods for the MuPFiS

The following paragraphs study the accuracy of each step of the analytical method for measuring TSS and VSS masses retained. Afterwards, every step error has been compared with the total retained mass to determine the influence of every measurement stage on the final results.

The process steps introducing incertitude during the analysis are: the balance, the mass variation of filters when heating at 550 ˚C, the mass variation of porcelain vessels, the mass variation of filters during filtration and the accuracy of water meters.

Balance

The precision balance used indicates when the weight is stable. The values given by this balance appear to be robust because they do not vary along time.

The balance from the water laboratory has been used. The balance used is a Mettler Toledo AE200 with a readability of ±0.1 mg, thus, the balance adds an error of ±0.1 mg each time it is used.

Mass variation of filters when heating at 550 ˚C

Previous tests (Sagel, 2007) have pointed that in an inorganic filter there is a small fraction of organic components. Thus, a small part of the filter is combusted when heating at 550 ˚C.

(Sagel, 2007) showed that Whatman’s Glass fibre filters type GF/C lose, during combustion, 1.3% ±0.6% of its mass.

Experiments carried with blank filters during the consecution of this report showed that Whatman’s Quartz filters QM-A lose, during combustion, 0.6% ±0.06% of its mass. Not enough blank tests have been done to determine the accuracy of that mass variation, hence, for QM-A filters is used the variance of GF/C filters, ±0.6% of the filter mass.

For instance, considering that a GF/C filter has in average a mass of 91 mg, a variation of 0.6% represents an inaccuracy of mass of 0.5 mg. Whereas a QM-A filter has an average mass of 150 mg, thus an inaccuracy of 0.09mg.

Mass variation of the porcelain vessels

It was not necessary to study the mass variation of the porcelain vessels because filters are weighted without the crucible. Filters do not get stuck to the porcelain vessels during combustion.

Mass variation of the filters during filtration

No relevant filter mass variation has been detected during filtration tests.

Accuracy of the water meters

The water meters employed are Elster D82 Qn 1.5m^3/h 6.131.42. These water meters overfill the ISO 4064 class C guidelines for cold water (Table 5.2).

Table 5.2. ISO 4064 definition of class C.

Flow	Maximum permissible error
Flow > 22.5 l/h	<2%
22.5 l/h > Flow > 15 l/h	<5%

The Maximum permissible error is related to the error of volume flow indication.

Indeed, if the maximum permissible error is 5%, the maximum difference between two measurements of the same volume must be at maximum 10%.

Conclusion

Accuracy of TSS' and VSS' concentration measurements have been calculated depending on the mass retained (Table 5.3 and Table 5.4).

Table 5.3. Calculation of TSS concentration accuracy depending on the mass retained.

			Mass retained (mg)		
times	type of error	accuracy (mg)	2.5	4	6
2	balance error	0.1	4%	3%	2%
1	water meter		10%	10%	10%
	TOTAL ERROR	0.2	18%	17%	17%

Table 5.4. Calculation of VSS concentration accuracy depending on the mass retained.

			Mass retained (mg)		
times	kind of error	accuracy (mg)	2	2.5	3
2	balance error	0.1	5%	4%	3%
1	loss of filter mass when combustion	0.54*	27%	22%	18%
			10%	10%	10%
	TOTAL ERROR	0.74	47%	40%	35%

* Loss of filter mass when combustion depends on the filter employed, 0.54 is for the Whatman GF/C filter

The mass of VSS is lower than the TSS, hence, the accuracy is lower. For the VSS measurement the incertitude is higher because of the additional incertitude related to the loss of filter mass during combustion.

Table 5.5. TSS and VSS accuracy for the MuPFiS.

Volume accuracy	±10%
TSS concentration accuracy	±0.2 mg ±10% of volume
VSS concentration accuracy	±0.74 mg ± 10% of volume

* VSS concentration accuracy has been calculated for the Whatman GF/C filter

5.2.2 Validation of MuPFiS

The MuPFiS has been tested and validated before being used on the field.

Hereby has been checked if the results obtained during validation tests are within the accuracy limits established. Volume, TSS and VSS concentration have been checked.

Three validation tests have been done; 13th, 27th and 30th May. During 13th and 27th May tests, two branches have been run in parallel, while the 30th May test four branches have been run in parallel.

Stock graphs show average, high and low values of the volumes, TSS and VSS concentrations of the data obtained during the test (Figure 5.2).

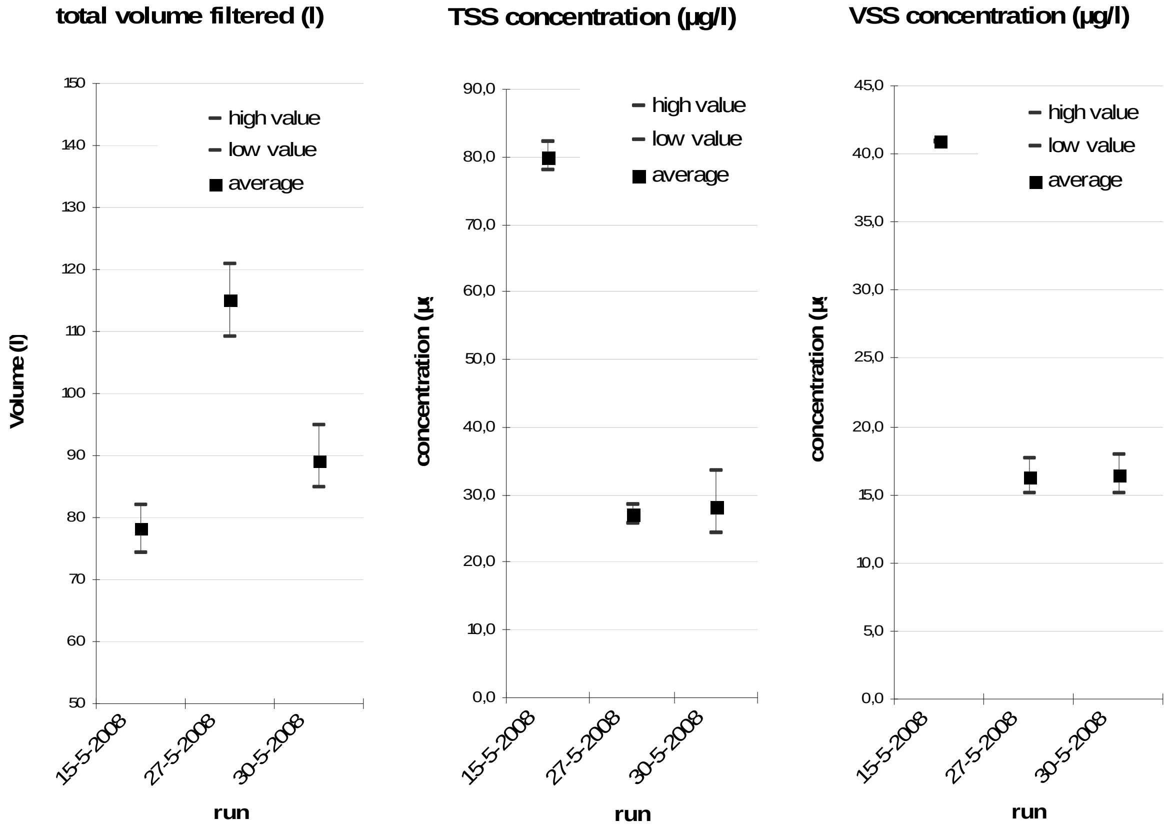


Figure 5.2. Stock graphics for volume filtered, TSS and VSS concentration - validation tests.

Table 5.6. Comparison of results and defined accuracy - validation test.

		Measured			Accuracy			
		low value	average	high value		low limit	high limit	accordance
Volume (l)	15-may	82	78	74	±10%	70	86	yes
	27-may	121	115	109		104	127	yes
	30-may	95	89	85		80	98	yes
TSS (µm/l)	15-may	78	80	82	±0.2 mg ±10% of volume	69	91	yes
	27-may	26	27	28		23	31	yes
	30-may	24	28	*33*		23	33	*no*
VSS (µm/l)	15-may	41	41	41	±0.74 mg ± 10% of volume	28	54	yes
	27-may	15	16	18		9	24	yes
	30-may	15	17	18		7	26	yes

The comparison between the accuracy results (Table 5.6) and the accuracy calculated (Table 5.5) showed that all the values obtained during the validation test are within the accuracy limits previously established, thus, the apparatus is set to be used following the accuracy indications previously described (Table 5.5).

5.3 Filters validation & comparison

(Gauthier et al., 2001) pointed that discrepancies in results appear when the same sample is analyzed with different types of filters, thus, the comparison of results using different kinds of filters must be done cautiously.

This point compares different types of filters that have been used for the MuPFiS experiences. This study enables to compare filters characteristics and to set some basic directives to choice the most appropriate filter for a filtration run.

5.3.1 Comparison of used filters for MuPFiS' tests

Three types of micro membrane filters were used for the MuPFiS filtration runs; the Whatman GF/F, the Whatman GF/C and the Whatman QM-A. GF/F is commonly used for the TILVS, while the GF/C and QM-A are generally used with the MuPFiS.

Description of the micro membrane filters used

Whatman GF/F, GF/C QM-A are glass fibre free-binder filters produced by Whatman (Table 5.7). The GF/F has a pores size of 0.7-µm, the GF/C has a pores size of 1.2-µm and the QM-A has a pore size of 2.2-µm. All the filters have a diameter of 47 mm.

Whatman GF/F and GF/C are made of pure borosilicate glass, whereas Whatman QM-A is made of high-purity quartz (SiO_2). The organic composition enables filters to resist to high temperatures.

Table 5.7. Employed filters' characteristics (Whatman).

Grade	Description	Particle Retention in Liquid (µm)	Typical Thickness (µm)	Basis Weight (g/m²)
GF/C	Borosilicate Glass	1.2*	260	53
GF/F	Borosilicate Glass	0.7*	420	75
QM-A	Quartz	2.2*	450	85

*Particle Retention Rating at 98% efficiency

Test 1: Comparison between GF/C and GF/F filters

Whatman GF/C and GF/F glass fibre filters have been tested in parallel with the MuPFiS in the TUDelft's water laboratory.

A GF/C filter (1.2-µm pore size) was installed in the first filtration line and a GF/F filter (0.7-µm pores size) was installed in the second filtration line. Two runs were done in parallel, starting at 10h00 and ending at 11h30 the 19th June 2008. The system was flushed during five minutes before filtration tests.

Table 5.8. Comparison between GF/C and GF/F filters – MuPFiS' filters comparison test.

Filter type	GF/C (1.2 µm)	GF/F (0.7 µm)	Variation
Run	10:00 - 11:30	10:00 - 11:30	
Volume filtered (l)	162	116	39%
Mass retained (mg)	2.6	2.1	24%
TSS concentration (µg/l)	16	18	-11%
VSS concentration (µg/l)	15	17	-14%

The volume and the mass retained are bigger with the GF/C than with the GF/F filter (Table 5.8). TSS concentration is slightly superior with the GF/F filter. Indeed, when comparing volume and mass retained between GF/C and GF/F, the GF/C has filtered more volume (+39%) but did not retain proportionally the same amount of mass (only +24%) .

Both filters present a similar discolouration (Figure 5.3 and Figure 5.4).

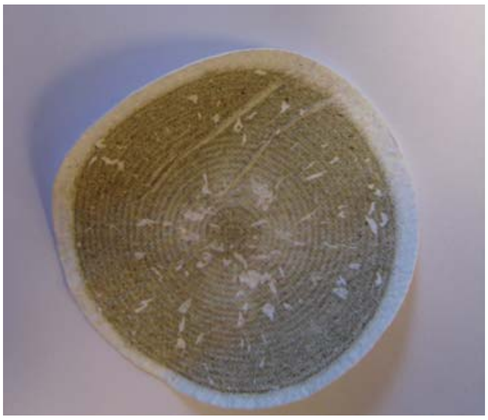


Figure 5.3. GF/C filter image – MuPFiS' filters comparison tests.

Figure 5.4. GF/F filter image - MuPFiS' filters comparison tests.

Test 2: Comparison between GF/C and QM-A filters

Whatman GF/C and GF/C glass fibre filters have been tested in parallel with the MuPFiS in the TUDelft's water laboratory.

A GF/C filter (1.2-µm pores size) was installed in the first filtration line and a QM-A filter (2.1 µm pores size) was installed in the second filtration line. Two runs were done in parallel, starting at 10h00 and ending at 11h15 the 21st July 2008. The system was flushed during five minutes before filtration tests.

Table 5.9. Comparison between GF/C and QM-A filters – MuPFiS' filters comparison test.

Filter type	QM/A (2.1 µm)	GF/C (1.2 µm)	Variation
Run	10:00 - 11:15	10:00 - 11:15	
Volume filtered (l)	187	118	59%
Mass retained (mg)	5.4	3.8	42%
TSS concentration (µg/l)	28	32	-11%
VSS concentration (µg/l)	23	24	-4%

The volume and the mass retained are bigger with the QM-A than with the GF/C filter (Table 5.9). TSS concentration is slightly superior with the GF/C filter. Indeed, as happened with GF/F and GF/C filters, when comparing volume and mass retained between QM-A and GF/C, the QM-A has filtered more volume (+59%) but did not retain proportionally the same amount of mass (only +42%) .

The QM-A filter (Figure 5.5) presents a more intense colouration than the GF/C filter (Figure 5.6).

The filters mass loss when combustion at 550 ˚C is different for the QM-A and GF/C filters.

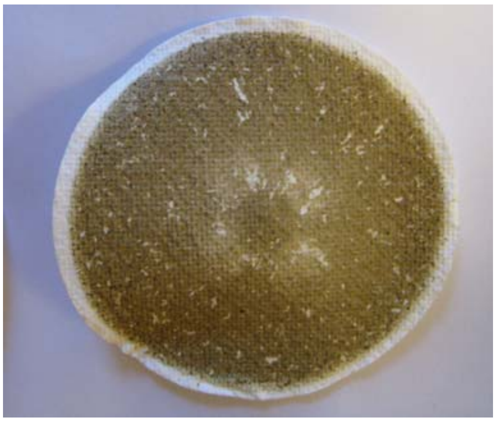


Figure 5.5. QM-A filter image - MuPFiS' filters comparison tests.

Figure 5.6. GF/C filter image - MuPFiS' filters comparison tests.

Test 3: Comparison between GF/C and QM-A filters

Whatman GF/C and GF/C glass fibre filters have been tested in parallel with the MuPFiS in the TUDelft's water laboratory. In this test 3 is also reported the flow variation of both filters.

A GF/C filter (1.2 µm pores size) was installed in the first filtration line and a QM-A filter (2.1 µm pores size) was installed in the second filtration line. Two runs were done in parallel, starting at 11h05 and ending at 12h00 the 6th August 2008. The system was not flushed before filtration tests.

Table 5.10. Comparison between GF/C and QM-A filters – MuPFiS' filters comparison test.

Filter type	QM/A (2.1 µm)	GF/C (1.2 µm)	Variation
Run	11:05 - 12:00	11:05 - 12:00	
Volume filtered (l)	157	44	257%
Mass retained (mg)	10	4,6	117%
TSS concentration (µg/l)	64	105	-39%
VSS concentration (µg/l)	26	42	-40%

The volume and the mass retained are much bigger with the QM-A than with the GF/C filter (Table 5.10) compared to results from test 2. TSS concentration is largely superior with the GF/C filter. As was observed in test 1 and 2, the bigger pore size filter filters more volume (+257%) but did not retain proportionally the same amount of mass (only +117%).

QM-A filter's flow is higher than GF/C filter's flow (Figure 5.7). In addition, QM-A flow decreases linearly along time while GF/C decreases exponentially. GF/C flow variation is caused by an accelerated fouling and finally leads to the clogging of the filter. The GF/C filter reaches the flow limit of 20 l/h at the 40th minute of filtration.

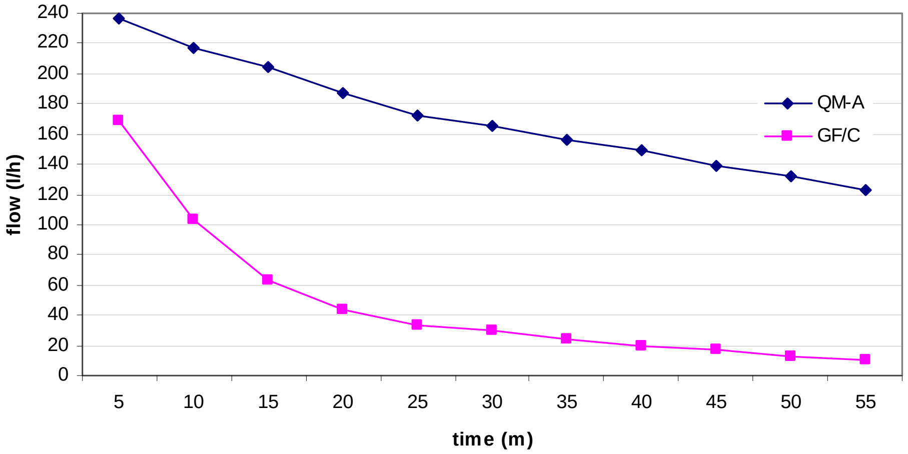


Figure 5.7. Filtration flow – comparison test between GF/C and QM-A filters.

In test 3 the clogging of the filter has accentuated the difference between TSS concentration obtained by the bigger and the smaller pores size filter.

Figure 5.8. QM-A filter image - MuPFiS' filters comparison tests.

Figure 5.9. GF/C filter image - MuPFiS' filters comparison tests.

The QM-A filter (Figure 5.8) presents a darker colouration than the GF/C filter (Figure 5.9).

Filters comparison results

The filter with bigger pores' size has filtered a bigger volume of water in the three tests. Both filters were tested in parallel with the same TMP during comparison tests (from network's pressure to zero). Thus, as the filter with the bigger pores size offers a lower resistance more water flows through it.

In addition the filter with the bigger pores' size has retained more mass during comparison tests. However, when looking into the TSS concentrations retained by each filter (Figure 5.10, Figure 5.11 and Figure 5.12), it can be seen that in both cases filters with smaller pores size retained bigger concentrations of TSS. The TSS concentration variation for experiments 1 and 2 is 11 %, while for the experiment 3 is 39% (Table 5.8, Table 5.9 and Table 5.10).

That difference of TSS concentration is due to the range of smaller particles that is retained in the small pore size filter and not in the big pore size filter, in the 0.7 to 1.2 µm size range and in the 1.2 to 2.1 µm size range, and the clogging of the filters.

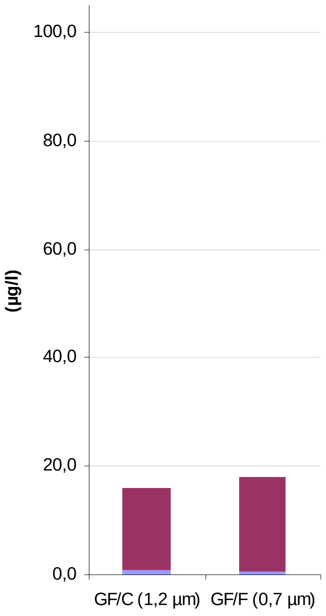


Figure 5.10. TSS and VSS concentration (µg/l) - comparison test between GF/F and GF/C filters.

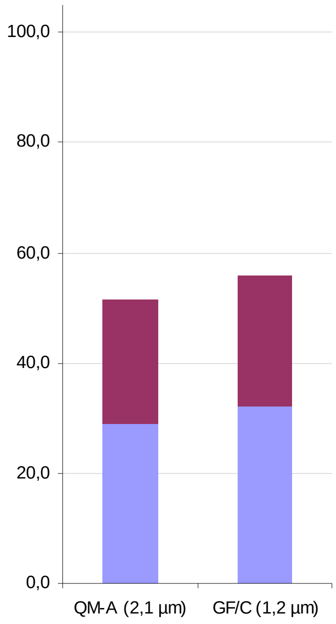


Figure 5.11. TSS and VSS concentration (µg/l) - comparison test between QM-A and GF/C filters.

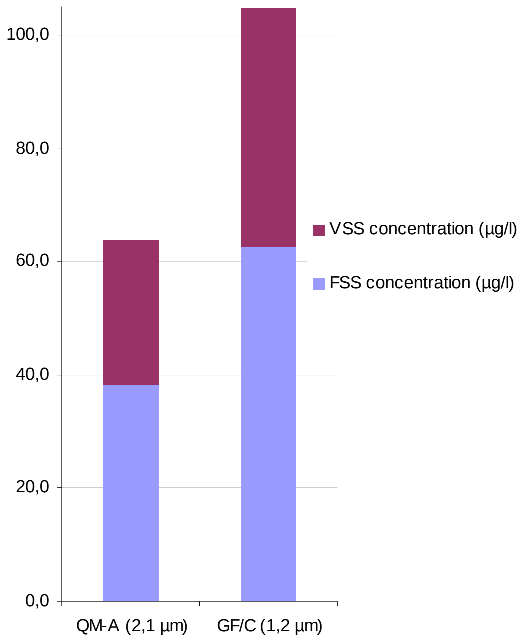


Figure 5.12. TSS and VSS concentration (µg/l) - comparison test between QM-A and GF/C filters.

The filter comparison test concluded that when sampling drinking water from the TUDelft's water laboratory either with a 1.2 or 0.7-µm pores size filter or a 1.2 or 2.1-µm pores size filter the TSS concentration varies of 11% in both cases. Thus, it is not recommended to compare results coming from different pores size filters.

Furthermore, for a fixed run time a bigger pores size filter retains more mass compared to a smaller pores size filter. A bigger pore size filter filtrates bigger volumes of water for a given time period than a smaller pores size filter, thus, usually retains bigger amounts of SS. As showed before, when the mass retained is bigger the accuracy is also bigger.

Besides, images from filters show that higher TSS mass retained imply more intense colourations when sampling the same types of water (Figure 5.3, Figure 5.4, Figure 5.5, Figure 5.6, Figure 5.8 and Figure 5.9).

5.3.2 Guidelines for filters choice

From these comparison tests with drinking water from the water laboratory of TUDelft it can be stated that:

- Different types of filters measure different TSS concentrations.
- Bigger pores size filters filter bigger volumes, thus, retain bigger amounts of particles.
- Clogging of filters can disturb results.
- Bigger pores size filters permit to retard fouling, however do not capture small sized particles.

Indeed, when doing filtration tests it is recommended to:

- Use always the same type of membrane filter to obtain comparable results.
- Chose the pore size of the filter to control the mass retained and the filtration run time.

However, (Horowitz et al., 1996) stated that comparative results from different kinds of filters depend on the type of water analyzed. Indeed, these guidelines are obtained after experimental tests with drinking water from the TUDelft’s water laboratory, thus, these results should be used as an indication for other types of water.

5.4 Tests with the particle counter

5.4.1 Description of the tests

Three tests have been performed in the water laboratory of TUDelft with the MuPFiS and the particle counter Met One PCX installed in parallel (Figure 5.14 and Figure 5.13). Both apparatus were connected to the same tap using a "T" connection.

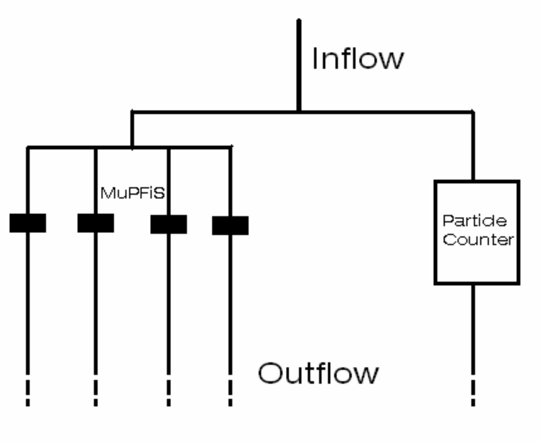


Figure 5.13. Schematic representation of the MuPFiS and particle counter installation.

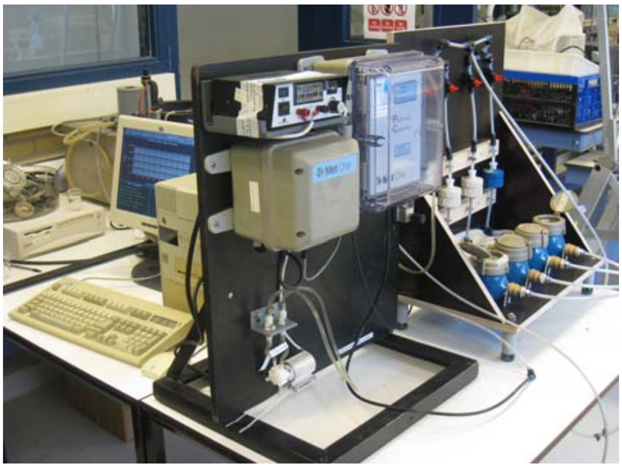


Figure 5.14. Installation of the MuPFiS and particle counter in parallel.

Both apparatus functioned simultaneously during each test. The particle counter sampled the water continuously offering a set of data every minute, while the MuPFiS did consecutives filtration runs of one hour on average. Filtration runs were shortened, when the critical flow of 20 l/h was reached before the end of the test.

Three tests were done on different dates:

- Test of 4th June. The test started at 9:05 and finished at 15:10. Six filtration runs were done with the MuPFiS. The tap was flushed[1] during ten minutes before starting the test.
- Test of 6th June. The test started at 8:55 and finished at 12:55. Four filtration runs were done with the MuPFiS. The tap was not flushed before starting the test.
- Test of 16th June. The test started at 8:45 and finished at 13:15. Five runs were done with the MuPFiS. The tap was not flushed before starting the test.

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[1] When the water flow is low, in a pipe, suspended solids are formed (chapter 2.3). During the night there is an almost zero flow situation inside the DWDS of the TUDelft's laboratory. Thus, the system is flushed a few minutes to discard this stagnant water and obtain a representative sample of drinking water from the network.

5.4.2 Analysis of results

The analysis of the particle counter data shows that there are three different phases for each test: transitory state, steady state and peaks due to big changes on the flow (Figure 5.15).

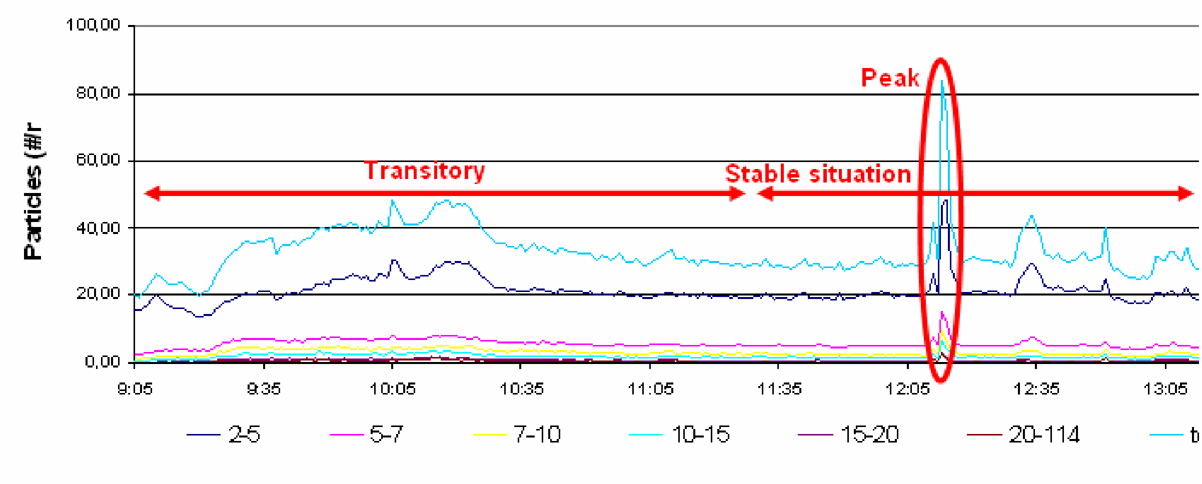


Figure 5.15. Transitory, steady state and peak on particle distribution - 4th June 2008 test.

Definition of phases:

- The transitory state occurs at the beginning of each test until the number of particles and the particle volume concentration (PVC) reaches the steady state. This period can be associated to internal production of particles due to corrosion and biofilm detachment (Vreeburg et al.).
- The steady state occurs when either there is no variation of the total number of particles or no variation of size distribution of particles during a test.

  The steady state takes into account the total number of particles counted from the analyzed point until the end of the test. Note that the steady state starts when the total number of particles at one point is ±10% the average of total number of particles from that point until the end of the test.
- Two indicators identified the peaks; firstly, when the number of particles augments (number of particles peak, Figure 5.16) and secondly when the PVC increases (volume peak, Figure 5.17) compared to the average of the period. Peaks are associated to hydraulics events that create resuspension of particles (Vreeburg et al.).

  For this study have been analyzed only the biggest peaks, the peaks appeared when the number of particles, number of particles peak, or the PVC, volume peak, increase more than 30% compared to the respective value of the steady state.

  Two kinds of peaks were analyzed on particle counts, a volume peak on the test of 16th June at 11h13 (Figure 5.17) and a number of particles peak during the test of the 4th June at 12h13 (Figure 5.16).

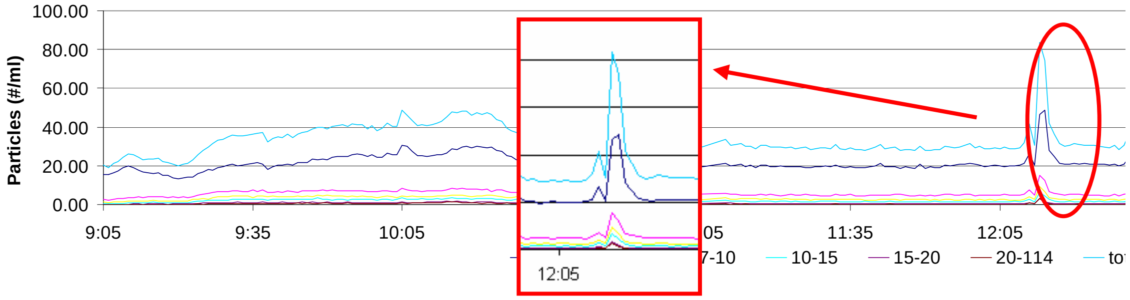


Figure 5.16. Number of particles peak – 4th June 2008 test.

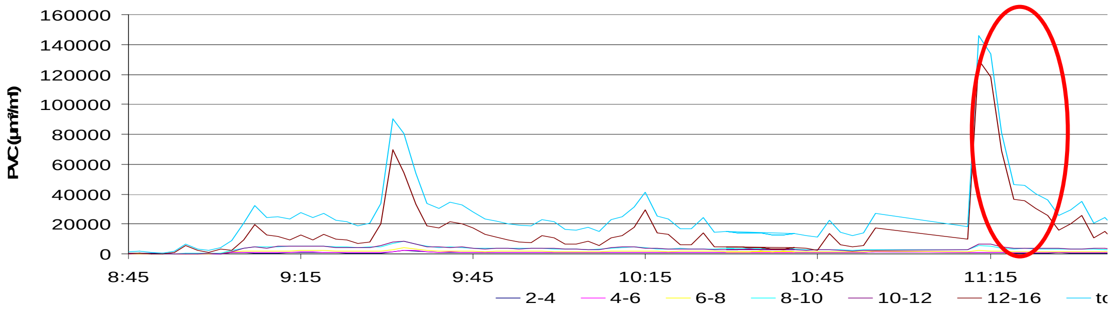


Figure 5.17. Volume of particles peak - 16th June 2008 test.

There are three phases in each test. Once the phases have been defined (Table 5.11) a comparative test has been run between all the experiments. The different phases are compared between them and with the results obtained with the MuPFiS.

Table 5.11. Identification of the three phases.

	4th June	6th June	16th June
Total	9:05 – 15:10	8:55 – 12:54	8:45 – 13:15
Transitory	9:05 - 11:30	8:55 - 10:55	8:45 - 11:35
Steady state	11:30 - 15:10	10:55 - 12:54	11:35 - 13:15
Peaks	10:15, 11:10, 12:13, 13:10 and 14:13	11:55	9:31, 10:13, 11:13 and 12:13

This chapter shows a synthesis of the relevant data obtained during the experiments. Appendix II presents further information regarding the experiment.

Steady state

The steady state period has been isolated and the peaks have been taken out for the analysis. Peaks could disturb conclusions because they are not part of the steady state.

Table 5.12. Nº of particles and PVC during the steady state.

	4th June	6th June	16th June
nº of particles (#/ml)	29.23	29.97	51.61
PVC (µm³/ml)	5867	8834	15107

The total number of particles is about 30 particles/ml during tests of the 4th and 6th June and about 50 particles/ml during tests of the 16th June (Table 5.12).

First of all, the results obtained the 4th and 6th June are compared because of the similar amount of total number of particles, and afterwards these results are compared with the ones from the 16th June.

The comparison of the 4th and the 6th June results points that:

- The total number of particles is similar, about 30 particles/ml, however the PVC is different, 5900 µm³/ml the 4th June and 8800 µm³/ml the 6th June (Table 5.12).
- Different values for volumes are due to a different size distribution of particles, although the distribution of particles within the ranges is almost the same (Figure 5.18), in the 6th June run it has been noticed a bigger amount of particles in the bigger ranges compared to the 4th June test (Figure 5.19).

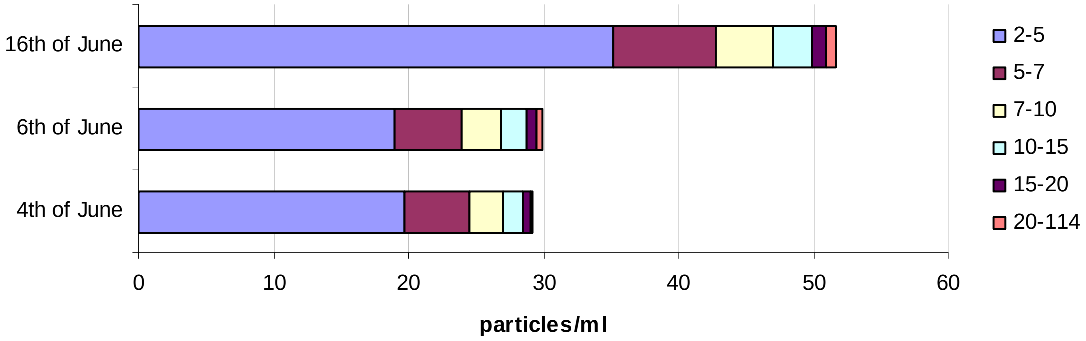


Figure 5.18. Particle size distribution by ranges (#/ml) – particle counter tests.

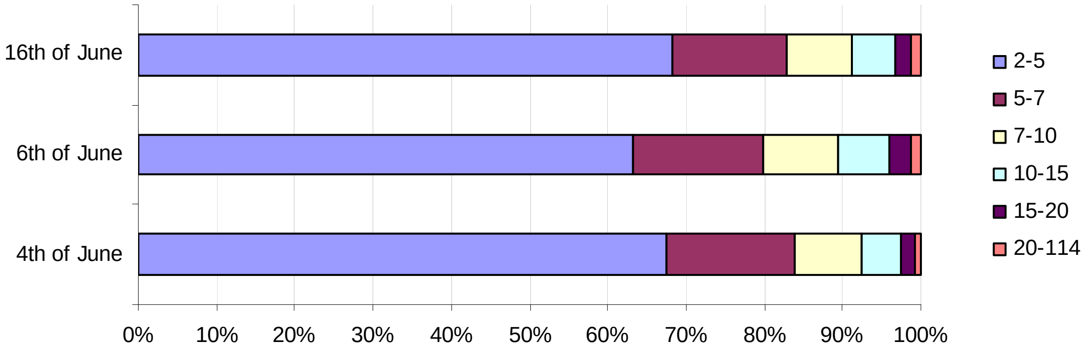


Figure 5.19. Size distribution of particles (%)– particle counter tests.

The analysis of the 4th and 6th June MuPFiS' test results (Table 5.13) notes that:

- The volume filtered during 6th June tests is bigger than during 4th June test, this can be due to a minor presence of smaller ranges of particles, thus there is less clogging.
- The TSS concentration is lower in the test done the 6th June (20 to 27 µg/l) compared to the test done the 4th June (33 to 41 µg/l). This higher concentration can be caused by the higher presence of smaller particles.

Table 5.13. Steady state – MuPFiS' tests results of the 4th, 6th and 16th June.

	4th June				6th June		16th June	
	run 3	run 4	run 5	run 6	run 3	run 4	run 4	run 5
Duration test	11:10 - 12:10	12:10 - 13:10	13:10 - 14:10	14:10 - 15:10	10:55 - 11:55	11:55 - 12:55	11:15 - 12:15	12:15 - 13:15
Volume filtered (l)	77.3	78.8	95.7	87.2	108.5	126.5	87.2	92.5
TSS concentration (µg/l)	40	41	29	33	27	20	46	41
FSS concentration (µg/l)	16	16	10	11	9	4	13	16
VSS concentration (µg/l)	24	24	19	22	17	16	33	25

Smaller particles accelerate filter clogging, thus reduce volume filtered. Despite their small volume, small particles have shown to have an important influence on the mass retained. This can be interpreted as small particles are denser than bigger particles.

The results from the 16th June test show a bigger number of particles (Table 5.12). The size distribution of the 16th June (Figure 5.19) is comparable to the size distribution of the 4th June for the smaller ranges but the 16th June has a higher portion of big sized particles. Hence, the PVC for the 16th June is bigger (Figure 5.20).

PVC is a good indicator to notice variations of number of particles within the big ranges. Volume is proportional to the cubic of the diameter of the particle; in fact a double diameter means an eight times bigger volume.

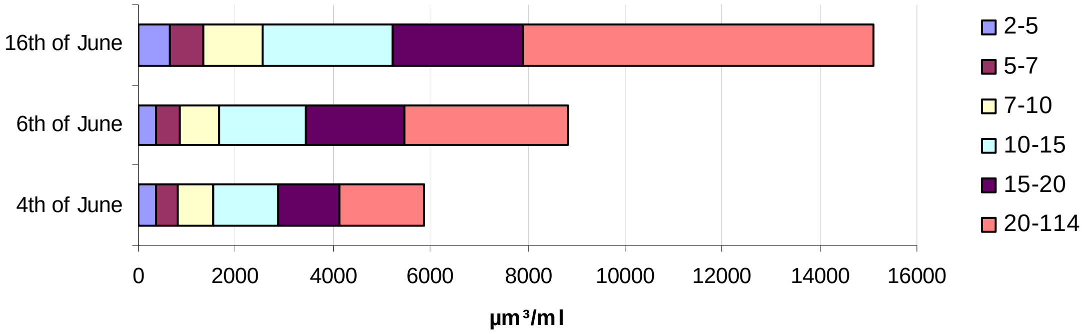


Figure 5.20. PVC by ranges (µm³/ml) – particle counter tests.

The comparison of the volume of water filtered during each run (Table 5.13) of the steady state points that the water volumes filtered the 16th June are similar to the water volumes filtered the 4th June. As said before, this smaller volume can be due to the presence of smaller particles that clog the filter.

Furthermore, TSS concentrations on the 16th and 4th June during the steady state are similar (Table 5.13). The 4th June during the steady period the TSS concentration was between 30 and 40 µg/l and the 16th June between 40 and 45 µg/l. As stated before, smaller particles have a big importance on the total mass retained.

5.4.3 Transitory state

The transitory period of the 16th June test has been analyzed and compared to the steady state.

First of all, despite being a big variation of the number of particles the PVC does not fluctuate considerably. Indeed, when analyzing the particle size distribution, it is noticed that while smaller ranges of particles increase during the transitory period, bigger ranges of particles do not fluctuate significantly (Table 5.14).

Table 5.14. Particle size distribution, number per range and run - 16th June test.

Period	Run	size (µm)	2-5	5-7	7-10	10-15	15-20	20-114	total
transitory	Run 1	8:45 - 9:35	28.97	6.04	3.60	2.54	0.88	0.53	42.57
transitory	Run 2	9:35 - 10:15	58.41	12.39	6.88	4.53	1.62	1.18	84.99
transitory	Run 3	10:15 - 11:15	56.20	11.17	5.86	3.57	1.20	0.70	78.70
steady	Run 4	11:15 - 12:15	37.07	8.57	4.95	3.48	1.40	1.21	56.68
steady	Run 5	12:15 - 13:15	35.10	7.61	4.40	3.12	1.19	0.86	52.28

It can be assumed that there is a high concentration of smaller ranges of particles during the transitory because of the internal production of particles as a result of corrosion, post flocculation and detachment of particles from bio films (Vreeburg et al.). Results show that internal production processes increase the smaller ranges of SS.

Table 5.15. TSS, VSS and FSS concentration evolution – 16th June test.

	run 1	run 2	run 3	run 4	run 5
Duration test	8:45 - 9:35	9:35 - 10:15	10:15 - 11:15	11:15 - 12:15	12:15 - 13:15
TSS concentration (µg/l)	51	59	44	46	41
FSS concentration (µg/l)	*25*	*23*	*15*	*14*	*15*
VSS concentration (µg/l)	*26*	*37*	*29*	*32*	*26*

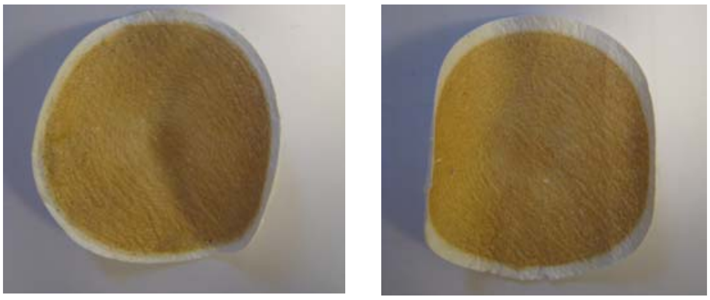
The analysis of the FSS and VSS composition of particles retained with the MuPFiS (Table 5.15) notices that during the transitional period there is a higher amount of TSS than during the steady phase. This difference is due to a higher concentration of FSS during the temporary state compared to the steady state. The concentration of VSS stays constant during all the experiment. The inorganic composition of particles indicates these internal produced particles are caused by pipe corrosion and post flocculation (Vreeburg et al.).

So far, pictures taken from filters (Figure 5.21) point that filters from first runs (transitory state) are more red coloured than the last filters (steady state). The red coloration is probably due to a higher presence of iron.

Run 1, 8:55 to 9:35
(transitory)

Run 2, 9:35 to 10:15
(transitory)

Run 3, 10:15 to 11:15
(transitory)



Run 4, 11:15 to 12:15
(steady state)

Run 5, 12:15 to 13:15
(steady state)

Figure 5.21. Filters images - 16th June test.

Filtration tests done the 4th and 6th June have shown similar results. When water stagnation occurs there is a formation of small particles, particles counters show bigger amounts of small ranges of particles during the transitory periods. In addition, TSS concentrations are bigger during the transitional periods compared to the steady states because of a higher presence of TSS while the VSS fraction stays constant.

5.4.4 Peaks

Peaks are big variations of total number of particles detected by the particle counter. They are normally caused by a suddenly flow augmentation. During the test this variations are caused by the opening and closing of valves between two runs. At the end of a filtration run the flow is low (30 l/h) because of the clogging of the filter and at the beginning of the next filtration run the flow is bigger (180 l/h) because the filter is not clogged and then offers no resistance to water.

The peaks have been analyzed considering an average of number of particles before, during the peak and after. That shows how does vary the total number and distribution of particles during a suddenly flow variation. Afterwards, TSS variations caused by peaks have been analyzed. This has been possible because particle counter data permits to localize the peaks in time.

Two peaks have been analyzed:

- Number of particles peak at 14:13 the 4th June 2008.
- PVC peak of 11:13 the 16th June 2008.

Number of particles peak at 14:13 the 4th June 2008.

The number of particles peak of the 4th June at 12h13 shows a big augmentation of total particles, from 30 to 65 particles per ml (Figure 5.22). The particle size distribution stays slightly the same; however there is an increase of the bigger ranges of particles.

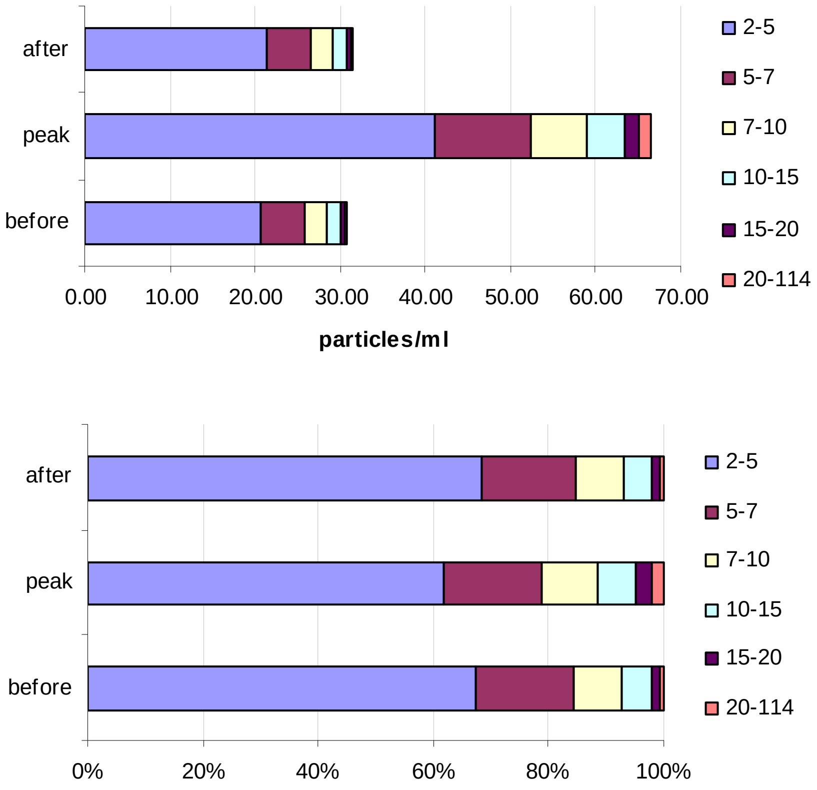


Figure 5.22. Evolution of number of particles before, during and after the peak - 4th June 2008 at 14:13 test.

Finally, it has been analyzed the relation between the presence of the peak and the augmentation of TSS concentration between the run 5 and 6 (Figure 5.23).

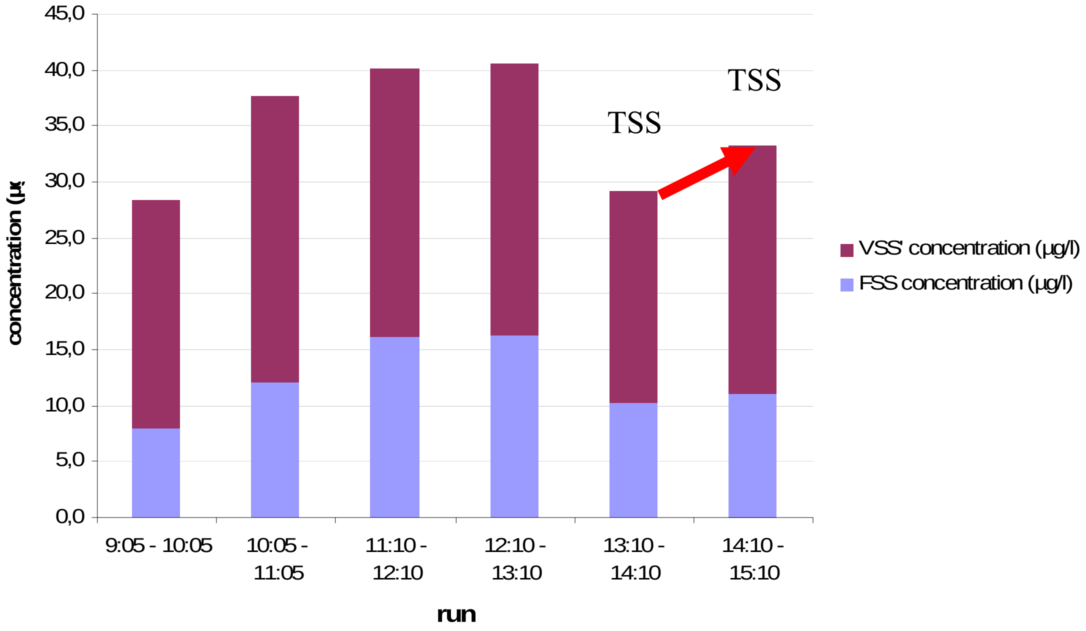


Figure 5.23. TSS and VSS concentration (µg/l) - 4th June 2008 test.

In this specific case, the TSS concentration variation can be associated to the peak because:

- The 14:13's peak is located at the beginning of the run 6.
- There is no peak during the run 5 (Table 5.11).
- The particle size distribution does not change before and after the peak (Figure 5.22).

Supposing that the 4 µg/l TSS variation is only caused by the peak and the volume filtered during the run 6 is 87 litres (Table 5.16) it can be assumed that mass released during the peak is 0.35 mg (0.35 mg = 4 µm/l * 87 l).

Table 5.16. Volume filtered and TSS concentration (µg/l) - run 5 and 6, 4th June 2008 test.

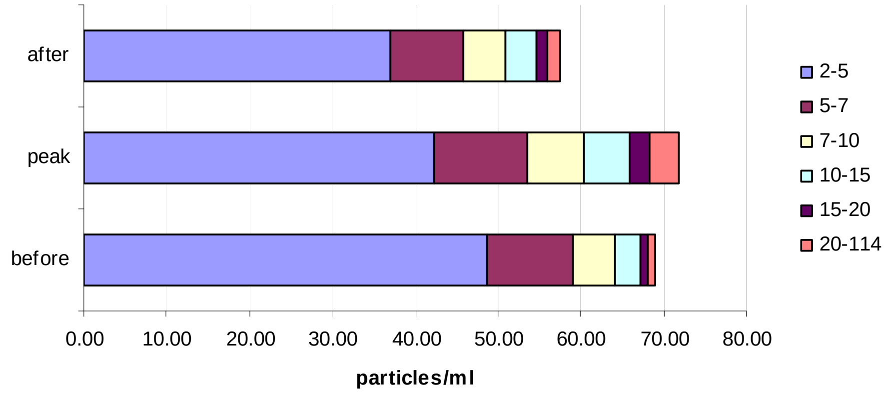
	run 5	run 6	variation between run 6 and run 5
Duration test	13:10 - 14:10	14:10 - 15:10	
Volume filtered (l)	96	87	
TSS concentration (µg/l)	29	33	4

PVC peak at 11:13 the 16th June 2008.

Looking into the peak of the 16th June at 11h13 it has been noticed a small increase of the total number of particles between before and during the peak, from about 69 to 71 particles per ml (Figure 5.24).

Despite this small augmentation, when regarding into the particle size distribution (Figure 5.24) it has been observed an augmentation of the larger ranges of particles compared to the smaller ranges. That augmentation of larges ranges of particles had been also noticed in the peak of 4th June 2008 at 14:13.

In the PVC peak the TSS augmentation caused by the peak can not be analyzed because the number of particles is not totally steady before and after the peak (Figure 5.24) about 69 particles/ml before the peak and about 58 particles/ml after the peak.



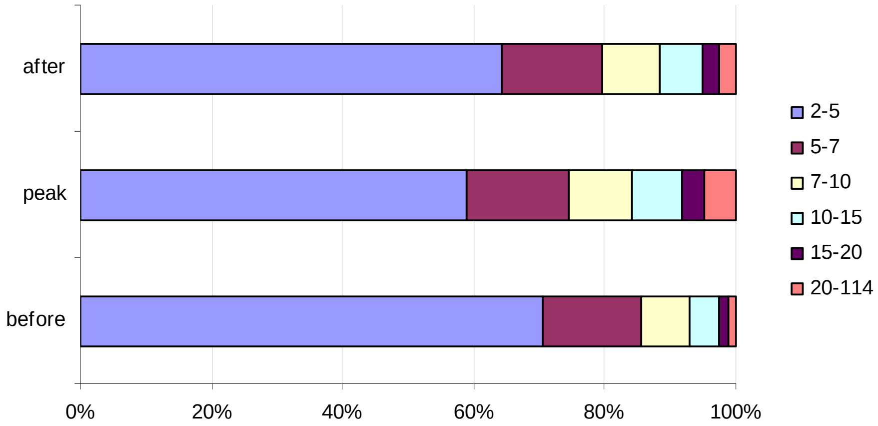


Figure 5.24. Evolution of number of particles before, during and after the peak of 16th June at 11h13.

From these results it can be concluded that sudden augmentation of water velocities in pipelines increase the number of SS, especially the bigger ranges of particles.

Changes in water flow have shown that mainly affect the bigger ranges of particles. When the flow is increased suddenly bigger ranges of particles resuspend while smaller ranges resuspend with the normal flow.

6 TESTING OF THE ANALYTICAL METHOD

This chapter reports field tests carried out in May, June and July 2008 with the MuPFiS. Tests were carried out at four different locations: at TUDelft's water laboratory and a at consumer's house in Delft, at Kiwa's installations in Nieuwegein, at the pumping station of Amstelveenseweg for Amsterdam and finally at the DWDS of Gouda. Gouda and Nieuwegein are supplied from groundwater while Delft and Amstelveenseweg from surface water. Tests were done following the standard filtration procedure defined for the MuPFiS included in the Appendix I.

Field tests enable the analysis of different facets (Table 6.1); the comparison of the MuPFiS with the TILVS, the comparison of SS concentrations at different locations, the analysis of SS concentrations variations within one DWDS, the study of SS concentrations variations along time, and finally the comparison of field results using different pore size filters.

Table 6.1. MuPFiS' field tests description.

Location		1st objective	2nd objective
Delft laboratory	Industrial area	Analysis of TSS and VSS variations	Comparison of different pore size filters
Nieuwegein	Industrial area	Comparison with TILVS	Analysis of TSS variations during day
Amstelveenseweg	Transportation network	Analysis of TSS and VSS	Comparison of different pore size filters under low pressure conditions
Gouda	Treatment plant, consumer's house and industrial area.	Analysis of TSS and VSS variations within the DWDS	Comparison of different pore size filters
Delft house	Consumers house	Analysis of TSS and VSS variations during the day	Comparison of different pore size filters

6.1 Delft (TUDelft's laboratory)

6.1.1 Test description

Several filtration tests were carried out at the TUDelft's water laboratory (Figure 6.1). For the consecution of these tests the MuPFiS was connected to a drinking water tap in the laboratory. Tests were carried out between the 24th April and the 6th August 2008.

Figure 6.1. Installation of the MuPFiS at the TUDelft's water laboratory

Sampled water is produced at the treatment plant of Kralingen. Surface water is first self purificated in a big open natural basin situated in Biesbosch, then water is conveyed to the treatment plant where is flocculated and precipitated, ozonated, rapid sand filtered, carbon activated filtered, post disinfected, and then distributed.

6.1.2 Results and discussion

Obtained data has permitted the analysis of SS concentrations variations along a day, a week and during several months. These first tests done in the laboratory did not respect the two hours run filtration duration defined previously as the standard procedure.

Variations along a day

The test done the 6th June 2008 between 8:55 and 12:55 was composed of four runs. Results show a decrease of TSS concentration, from 51 to 29 µg/l (Figure 6.2). Higher concentrations of SS at the beginning of the test can be associated to the particle formation that occurs during water stagnation along the night (chapter 2.3).

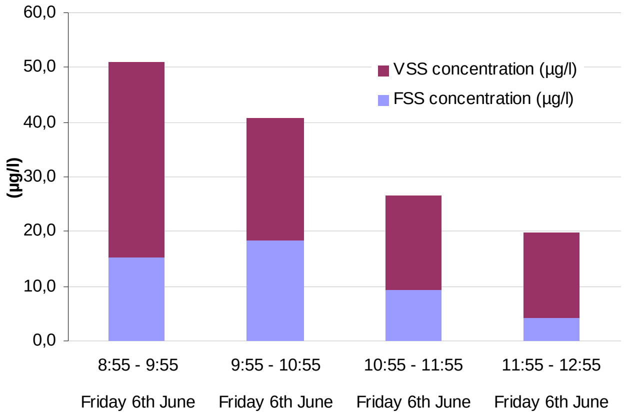


Figure 6.2. TSS and VSS concentration (µg/l) - 6th June 2008 test.

Variations along a week

SS concentrations variations along a week have been measured after flushing the system several minutes. The system has been flushed to discard particles produced during the night (previous point).

SS concentrations decrease during the week, on Monday 16th June the SS concentration is 43 µg/l whereas on Thursday 19th June is 16 µg/l (Figure 6.3). Higher concentrations of SS at the beginning of the test can be associated to the particle formation that occurs during water stagnation along the weekend (chapter 2.3).

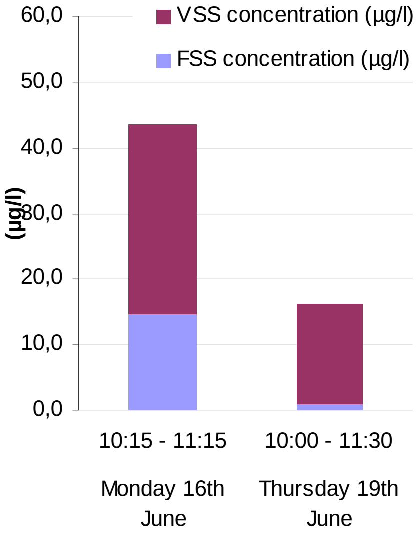


Figure 6.3. TSS and VSS concentration (µg/l) - 16th and 19th June 2008 test.

Variations along months

SS concentrations have changed during the test period; 16 µg/l the 19th June while 42 µg/l the 16th June. Moreover, organic and inorganic fraction of SS has also varied; 94% of inorganic fraction the 19th June whereas 7% of inorganic fraction the 24th April. The system was flushed before carrying out the tests.

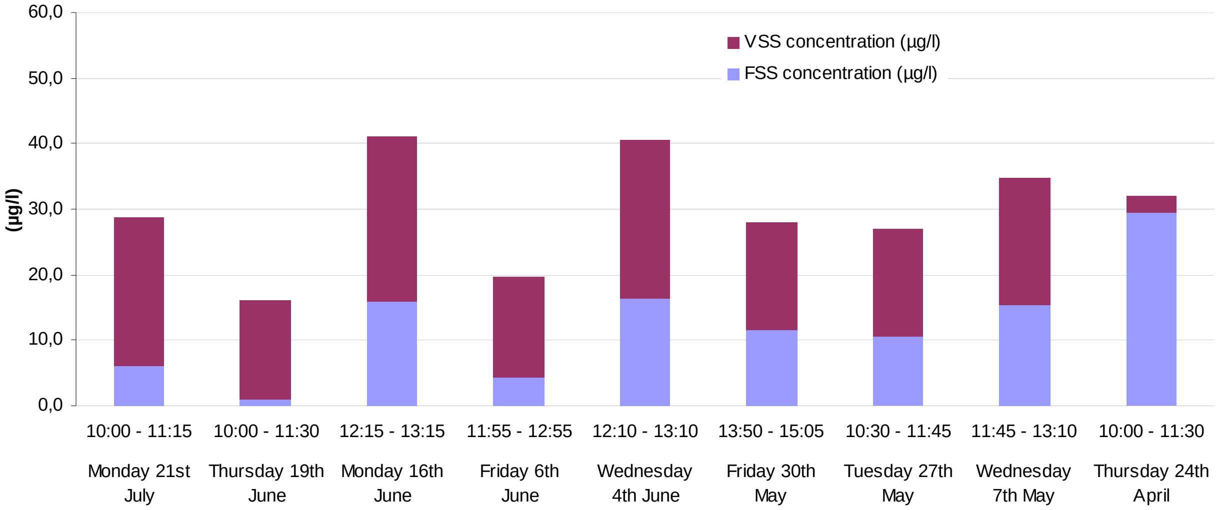


Figure 6.4. TSS and VSS concentration (µg/l) – 2008 tests.

Results show an augmentation of SS concentration during low flow periods generally caused by the variation of fixed SS. To avoid this FSS formation it is recommended to reduce low flow conditions in the network and to use materials that may not release inorganic particles.

6.2 Nieuwegein

6.2.1 Test description

The MuPFiS and the TILVS were compared in the Kiwa's water research laboratory of Nieuwegein the 15th July 2008 (Figure 6.6). Both apparatus were connected to the same tap and filtration experiments were carried out in parallel (Figure 6.5). A 2-branch version of the MuPFiS was used for tests (Figure 6.6).

Drinking water from Kiwa's water research laboratory is produced at Tull en't Wall. Ground water is first aerated, rapid sand filtered and then distributed.

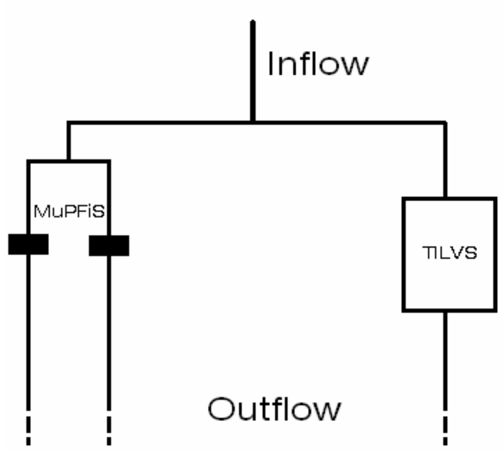


Figure 6.5. Schematic representation of the MuPFiS and TILVS installation.

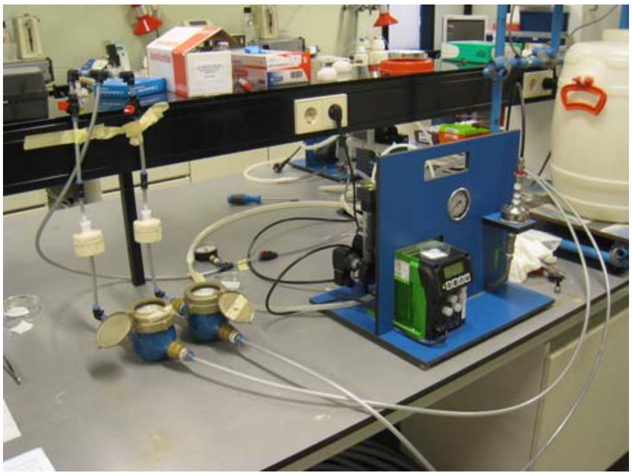


Figure 6.6. Installation of the MuPFiS and TILVS at the Kiwa's water research laboratory.

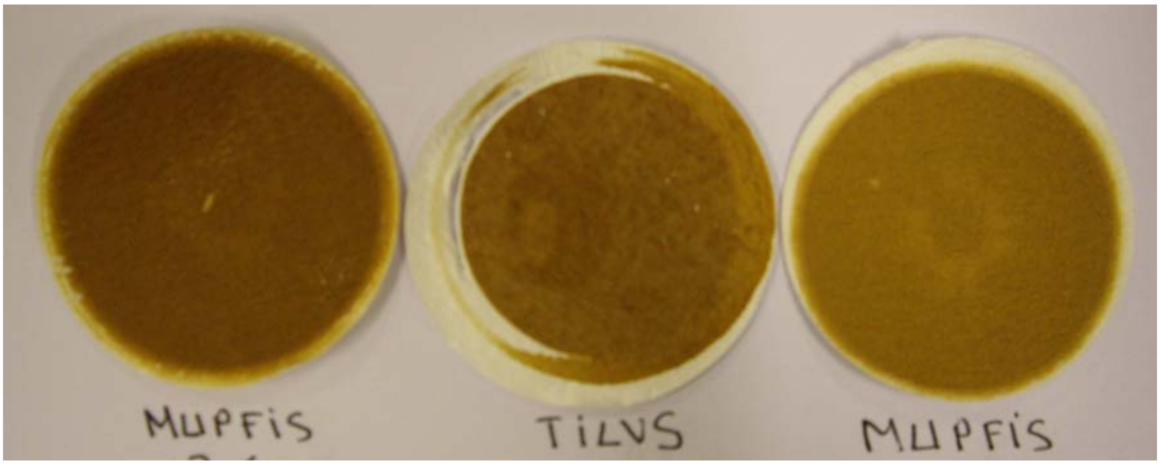
One 5-hour run was done for the TILVS while fifteen consecutive 20-minutes runs were done with the MuPFiS. Both experiences lasted the same time making the results comparable. The TILVS' pump was set at a flow of 4 l/h.

A Whatman GF/F glass fiber filter of 0.7-µm pore size was used for the MuPFiS and TILVS. The Whatman GF/F is a commonly glass fibre filter used for the TILVS. Therefore, it was also used for the MuPFiS to comparable results between both apparatus.

Afterwards, the TSS analyses of the filters were carried at Kiwa's water research facilities in Nieuwegein.

6.2.2 Results and discussion

After the TILVS' filtration test the Whatman GF/F filter was damaged (Figure 6.7), however, the filter used was analyzed. Occasionally, filters get damaged when filter holders are opened or closed, hence results are not affected.



MuPFiS TILVS MuPFiS

Run: 10:40 – 11:05 Run: 10:40 - 15:45 Run: 15:25 – 15:45

Figure 6.7. Filters image - 15th July 2008 test at Nieuwegein.

The TILVS measured a TSS concentration of 223 µg/l while the MuPFiS measured TSS' concentrations from 560 to 150 µg/l (Table 6.2). The TSS concentration obtained with the MuPFiS decreased along time, at the first run TSS concentration was 560 µg/l while at the last run TSS' concentration was 150 µg/l. On average the MuPFiS measured higher TSS concentrations than the TILVS.

So far, the TILVS sampled a total volume of water of 20 litres (4 l/s * 5 hours test = 20 litres). While the MuPFiS sampled volumes from 17 to 35 litres, the filtrated volume increased along time; the first MuPFiS' filtration run filtered 17 litres and the last filtration run filtered 35 litres.

In the first place, it has been noticed that the TSS concentration measured by the MuPFiS varies considerably, from 560 to 150 µg/l. This TSS concentration diminution has also been observed in tests done in the water laboratory at TUDelft. The presence of higher SS concentrations at the beginning of tests can be associated to the particles formation that occurs during water stagnation (chapter 2.3) or to a SS peak.

This high TSS concentration has induced to a fast clogging of the MuPFiS' filters; first runs filtered around 17 litres while last runs filtered 35 litres.

When looking into filters colouration, it can be noticed that the MuPFiS' first filtration run filter has a more intense colouration than the last MuPFiS' filtration run filter (Figure 6.7). The TILVS filter colouration is slightly more intense than the colouration of the last MuPFiS run filter. Indeed, filters colouration intensity is correlated with the TSS concentration of the filtration run; more intense colouration, higher TSS concentration.

An average TSS concentration has been calculated for the MuPFiS. Each run concentration has been multiplied by the specific volume filtered of the filtration run and then divided by the total volume filtered during all the MuPFiS experiment. The value obtained, 385 µg/l of SS, is higher than the value obtained by the TILVS, 223 µg/l.

That difference between TILVS' measured TSS concentration and MuPFiS' TSS calculated average concentration can be caused by:

- The filter used for the TILVS' filtration test was damaged, thus some particles could have passed through the filter.
- High TSS concentrations accelerate fouling, thus filtration flows where small. MuPFiS' water meters underestimate volumes when measuring low water flows, hence, the TSS concentrations could be overestimated.
- Sampling methods are different, thus, values found could be not comparable.

This experiment has proved that the MuPFiS can complete a filtration run in a shorter time than the TILVS, therefore the MuPFiS permits to track SS concentration variations along time more regularly. For instance, in this experiment while the TILVS carried out one filtration run the MuPFiS did 15 successful filtration runs.

However, this experiment has not been able to prove the comparability of results obtained by both methods; more tests in parallel should be carried out to analyze the differences.

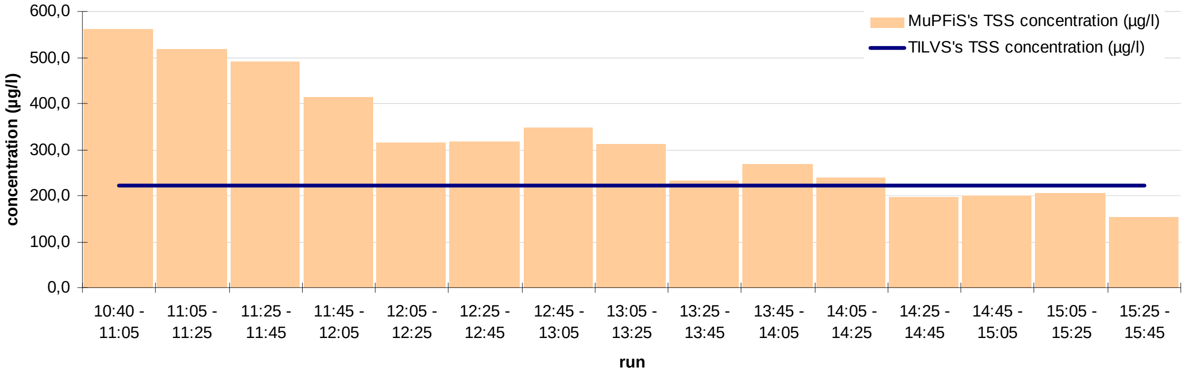


Figure 6.8. TSS concentration (µg/l) obtained with the MuPFiS and the TILVS during the comparison test at Nieuwegein the 15th June 2008.

Table 6.2. TSS concentration (µg/l) obtained with the MuPFiS and the TILVS and MuPFiS' sampled volume during the comparison test at Nieuwegein the 15th June 2008.

Run	10:40 11:05	11:05 11:25	11:25 11:45	11:45 12:05	12:05 12:25	12:25 12:45	12:45 13:05	13:05 13:25
TILVS' TSS concentration (µg/l)	223	223	223	223	223	223	223	223
MuPFiS' TSS concentration (µg/l)	562	518	491	415	316	317	348	312
MuPFiS' sampled volume (l)	17,7	16,9	17,6	18,8	24,4	24,3	22,4	22,9

Run	13:25 13:45	13:45 14:05	14:05 14:25	14:25 14:45	14:45 15:05	15:05 15:25	15:25 15:45
TILVS' TSS concentration (µg/l)	223	223	223	223	223	223	223
MuPFiS' TSS concentration (µg/l)	232	268	239	196	199	206	155
MuPFiS' sampled volume (l)	29,3	26,5	31	33,6	32,4	33	34,6

6.3 Amstelveenseweg

6.3.1 Test description

MuPFiS runs were conducted at the pumping station of Amstelveenseweg the 20th June 2008. The pumping station of Amstelveenseweg is situated at the end of the transportation network and supplies Amsterdam.

Two filtration runs were done with the MuPFiS. The first experiment was done between 9:50 and 12:45 and the second run between 12:45 and 14:25. In each of the filtration runs, two different filters were used in parallel. In one filter unit was installed a Whatman GF/C 1.2-µm pore size glass fiber filter, and in the other branch a Whatman GF/F 0.7-µm pore size glass fiber filter.

Experiments were carried out at the Amstelveenseweg North line. The sampling point was placed at the end of the transportation line, upstream the underground distribution reservoirs, thus the pressure was of 0.6 bars.

The source water is collected at the Rhine river, raw water is pretreated by flocculation, settled in basins and then rapid sand filtered. The pretreated water is infiltrated in a dune area and abstracted to be treated in the Leiduin treatment plant. At the Leiduin treatment plant the water is aerated, rapid sand filtered, ozonated, softened, activated carbon filtered and finally low sand filtered. The transport mains from the Leiduin treatment plant to the pumping station of Amstelveenseweg are made of concrete and cement mortar lined steel.

Figure 6.9. Installation of the MuPFiS and TILVS at the Amstelveensweg North line.

6.3.2 Results and discussion

Mass retained in both filters (Table 6.3) is under de minimal prescriptions of 2,5 mg established by the Standard Methods for the Examination of Water and Wastewater, (Greenberg et al., 1999). Therefore, when mass retained is under the minimal limit, TSS concentration has been calculated considering a mass retained of 2.5 mg.

For instance, the first run done with the GF/C filter has filtered 270 l and retained 1,8 mg, thus the TSS concentration calculated is <9.3 µg/l (9.3 µg/l = 2.5 mg / 270 l). Indeed, the calculated concentration is an upper limit and does not reflect the real concentration of TSS.

Table 6.3. MuPFiS' results - 16th June 2008 test at Amstelveenseweg.

Location	Amstelveenseweg - Pumping Station			
Run	9:50 - 12:45 (2h 55 min)		12:45 - 14:25 (1h 40min)	
Filter type	GF/C	GF/F	GF/C	GF/F
Mass retained (mg)	1,8	1,3	1,8	0,9
Volume filtered (l)	269	180	211	131
TSS concentration (µg/l)	<9,3	<14	<11,9	<19

Results point that GF/C filters have filtered bigger volumes of water and retained bigger amounts of mass compared to GF/F filters. The bigger pores size filter induces a smaller resistance to water filtration, thus, the water volume filtered under low pressure conditions is 50% bigger with the GF/C filter than with the GF/F filter.

It can be observed a red coloration of the filters (Figure 6.10), especially at the GF/C filters because they have retained bigger masses of SS.

9:50 - 12:45
GF/C filter

9:50 - 12:45
GF/F filter

12:45 - 14:25
GF/C filter

12:45 - 14:25
GF/F filter

Figure 6.10. GF/C and GF/F filters image - 20th June 2008 test at Amstelveenseweg.

So far, retained particles were placed on the filters and could be easily moved by the contact of the tweezers because of the low pressure conditions, 0.6 bars. Furthermore, some particles stayed in the filter holder because of the low pressure. It is recommended to avoid low pressure sampling points to minimize sample alteration while manipulation.

Indeed, TSS concentrations obtained at the Amstelveenseweg North line are low, <9.3 and <11.9 µg/l, compared to previous tests done with TILVS in 2006 (Verberk et al., 2006), TSS concentration of 37 – 49 µg/l.

Further analysis (not considering the 2.5 mg mass retention limitation).

These results have to be taken into account cautiously because the 2.5 mg mass retention limitation is not satisfied.

TSS and VSS variation along time have been studied from the results obtained by the Whatman GF/C filter because of the bigger mass retained, nevertheless below the retention limits of 2.5 mg.

Results show a small augmentation of TSS concentration between the first and second test, this augmentation (Table 6.4 and Figure 6.5) could be caused by the lower accuracy when sampling with small masses. So far, when looking into the composition of SS, it is noticed that the major fraction is composed of inorganic SS in both runs.

Table 6.4. TSS, VSS and FSS concentration (µg/l) (not considering the 2.5 mg limitation) - 20th June 2008 test at Amstelveenseweg.

	9:50 - 12:45	12:45 - 14:25
TSS concentration (µg/l)	6,7	8,5
FSS concentration (µg/l)	4,7	7,8
VSS concentration (µg/l)	2,0	0,7

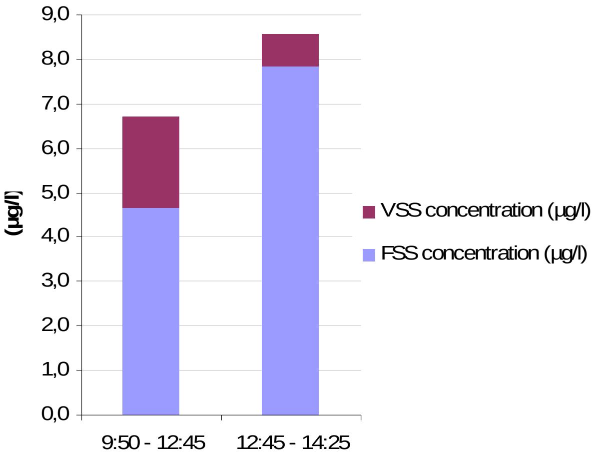


Figure 6.11. FSS and VSS concentration (µg/l) (not considering the 2.5 mg limitation) - 20th June 2008 test at Amstelveenseweg.

The transport mains from the treatment plant to the pumping station of Amstelveenseweg are made of concrete and cement mortar lined steel and they have a total distance of about 23km.

The high content of inorganic SS could be caused by the release of particles from the concrete and cement mortar lined steel transportation line, however, no conclusions can be obtained about the evolution of water quality from the treatment station to the pumping station of Amstelveenseweg because there is no available data concerning the SS concentration at the treatment plant.

6.4 DWDS of Gouda

6.4.1 Tests description

This test aims to characterize SS concentration at different points of the DWDS of the water company Oasen in Gouda using the MuPFiS.

Three different locations (Figure 6.12) have been selected to determine the SS evolution in the DWDS; at the treatment plant, and at two locations in the distribution network.

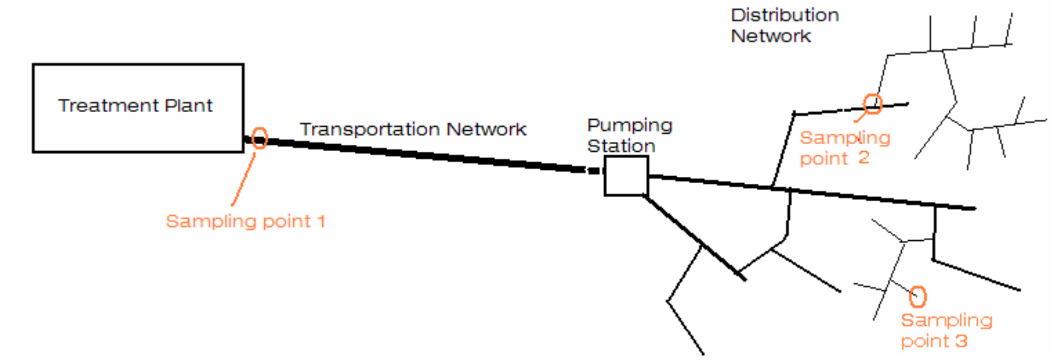


Figure 6.12. Schematic representation of the network and placement of the sampling points.

Location of the sampling points:

- At the treatment plant: downstream the Rodenhuis pumping station that is situated at the treatment plant (Figure 6.13).
- In the distribution network: Oasen's office at Gouda (Figure 6.14).
- In the distribution network: consumer's house (Figure 6.15).

Rodenhuis water treatment plant treats ground water. Water is aerated and rapid sand filtered, softened, rapid sand filtered, then activated carbon filtered, disinfected by UV, and finally pumped to the DWDS.

A run of two hours has been done at each location and pressure upstream the MuPFiS has been adjusted to 2.5 bars with the connection tap. During each run four filtrations experiments have been done in parallel:

- Two filtrations experiments using Whatman GF/C 1.2-µm pore size glass fibre filters.
- Two filtrations experiments using Whatman QM-A 2.1-µm pore size quartz filters.

Four simultaneous filtration experiments permit to obtain double values (duplicate experiment with both filters) and to compare retained SS and volume filtered between two different pore size filters.

Figure 6.13. Sampling point 1 at the treatment station: Rodenhuis treatment plant.

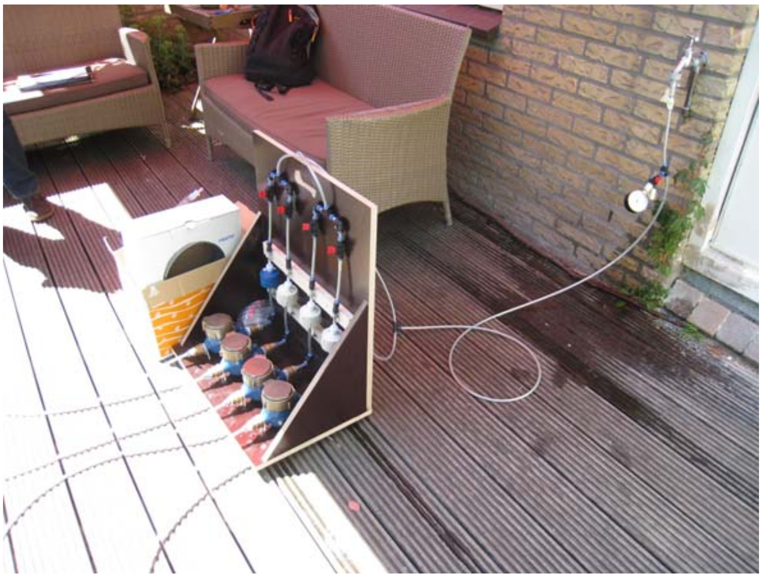


Figure 6.14. Sampling point 3 inside the distribution network: consumer house.

Figure 6.15. Sampling point 2 at the distribution network, Oasen's office at Gouda.

6.4.2 Results and discussion

Results show (Table 6.5) that masses retained on filters are low at the pumping station and consumer’s house, between 1 and 1.6 mg, and higher at the Oasen’s office, between 2.2 and 2.7 mg. Indeed, the minimal required mass retention established by The Standard Methods for the Examination of Water and Wastewater, 20th Edition, (Greenberg et al., 1999), has not been reached neither at the pumping station nor at the consumer’s house.

Volumes filtered with different pores size filters have been almost equivalent at locations with low concentration of SS, the pumping station and the consumer’s house, whereas there has been a bigger difference in volumes filtered at the Oasen’s office where there is a bigger SS concentration.

Table 6.5. MuPFiS’ results - 24th July 2008 tests at Gouda’s DWDS.

Location	Pumping station				Consumer's house				Oasen's office			
Run	9:20 - 11:20 (2h)				12:45 - 14:45 (2h)				15:30 - 17:30 (2h)			
Filter type	QM-A	QM-A	GF/C	GF/C	QM-A	QM-A	GF/C	GF/C	QM-A	QM-A	GF/C	GF/C
Mass retained (mg)	filter broken	1	1.6	1.5	1.3	1	1.3	1.4	2.7	2.6	2.4	2.2
Volume filtered (l)		290	254	220	262	261	221	220	269	249	205	197

Although at the pumping station and consumer’s SS concentration is low, under the 2.5 mg established criteria, a variation of the filters colour can be observed when looking into filters (Figure 6.16). The filter from the pumping station shows a beige coloration with presence of big black particles, whereas the filter from the consumer’s house has a brownish coloration and no black particles are present.

The big black particles present at the pumping station filter could be released from the activated carbon filtration step. These particles may precipitate in the DWDS and do not appear on the filters from tests carried out at the consumer’s house and at the Oasen’s office.

Pumping station	Consumers house	Oasen’s office
9:20 – 11:20	12:45 – 14:45	15:30 – 17:30
GF/C filter	GF/C filter	GF/C filter

Figure 6.16. GF/C filters images - 24th July 2008 tests at Gouda’s DWDS.

Filters from the consumer's house and Oasen's office present a more intense brown coloration compared to the pumping station filter. This can be due to a possible augmentation of suspended iron compounds. Iron compounds can be originated from corrosion of pipes and precipitation of iron that has not been removed at the treatment plant. Elemental analyses of the filters can help in determining the composition of TSS.

In addition, it is noticed that the brownish coloration of the Oasen's office filter is more intense compared to the filter from the consumer's house. This water quality diminution can be related to the fact that the Oasen's office is placed in an industrial area. Industrial areas have usually over sized drinking water distribution networks, big diameter pipe lines and low consumptions, thus low water velocities can lead to water quality diminution.

TSS and VSS analysis

TSS and VSS analysis have been done with all filters; however, the obtained results have to be taken into account cautiously because the 2.5 mg mass retention limitation is not respected.

In a first stage, it has been compared the results from the three locations obtained with the same filter, the GF/C filter. Indeed, the GF/C is the most commonly filter used with MuPFiS, thus, it is chosen to obtain comparable results. In a second stage, it has been compared the results obtained by the QM-A filter and the GF/C filter in three locations.

For this analysis an average has been calculated from the runs done with the same type of filter in each location.

Comparison between locations

Results (Table 6.6 and Figure 6.17) obtained with GF/C filters point that there is a water quality diminution between the treatment plant and Oasen's office but not between the pumping station and the consumer's house.

Table 6.6. TSS, VSS and FSS concentration (µg/l) (not considering the 2.5 mg limitation) - 24th July 2008 tests at Gouda's DWDS.

	Pumping station	Consumer's house	Oasen's office
TSS concentration (µg/l)	6.6*	6.1*	11.5
VSS concentration (µg/l)	2.8*	3.1*	6.3
FSS concentration (µg/l)	3.8*	3.0*	5.1

* These results have to be taken into account cautiously, the minimal 2.5 mg mass retention was not reached

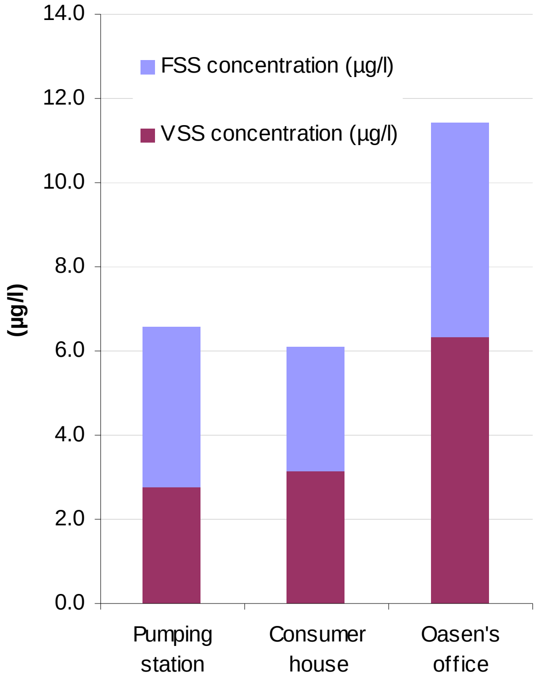


Figure 6.17. FSS and VSS concentration (µg/l) (not considering the 2.5 mg limitation) - 24th July 2008 tests at Gouda's DWDS using GF/C filters.

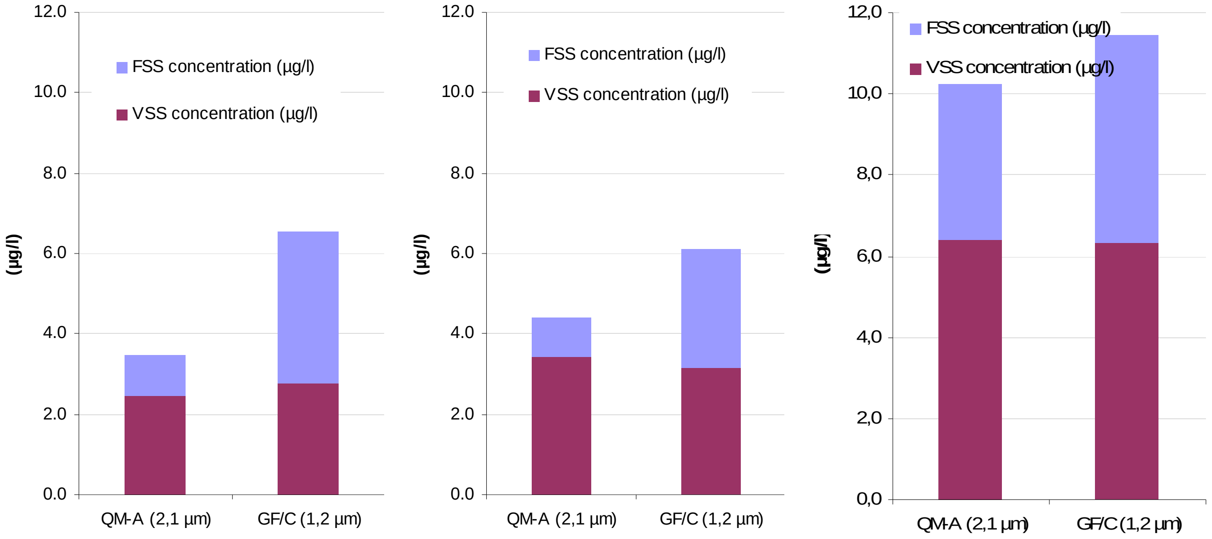
Comparison between filters

Bigger pore size filters have permitted a bigger filtration volume but have not lead to a higher retention of mass on the filters (Table 6.5). This is due to:

- Volumes filtered during parallel QM-A and GF/C filtration runs are similar (Table 6.5) especially at locations with low concentration of SS. That can be caused by the high pressure in the DWDS, and low concentrations of SS in water.
- Particles in the 1.2 to 2.1 µm size range have contributed to the mass retained, explained here above.

When looking into the FSS and VSS concentrations measured by QM-A and GF/C filters (Figure 6.18) it has been noticed:

- The TSS concentration is bigger using GF/C filters than QM-A filters, thus particles between 1.2 and 2.1 µm contribute to the TSS concentration.
- Comparing results from both filters, VSS concentrations are the same whereas FSS concentrations vary significantly. Therefore, it can be assumed that particles between the ranges of 1.2 and 2.1 µm are mainly inorganic.



Pumping station Consumer’s house Oasen’s office

Figure 6.18. Comparison of FSS and VSS concentration (µg/l) between QM-A and GF/C (not considering the 2.5 mg limitation) - 24th July 2008 tests at Gouda’s DWDS.

Carbon activated filters can be better operated to avoid the release of carbon particles into the DWDS. In addition, industrial areas should be designed correctly to avoid low flow conditions that lead into particles formation.

6.5 Delft

6.5.1 Test description

A MuPFiS test was done at a consumer's house at Delft the 6th July 2008. A reduced version of the MuPFiS with only two branches was used for tests (Figure 6.19). Sampled water in Delft's consumer house is treated at the same treatment plant as TUDelft's drinking water.

Figure 6.19. Installation of the MuPFiS at the consumer's house in Delft.

Four filtration runs of about two hours were done between 20h30 and 02h30. In each filtration run two filters were used in parallel. On one filter unit was installed a Whatman GF/C 1.2-µm pore size glass fiber filter and on the other branch a Whatman QM-A 2.1-µm pore size quartz filter. The TMP was set at 2 bars.

6.5.2 Results and discussion

Results show (Table 6.7) that when comparing results from the same type of filter mass retained and volume filtered are stable during the three runs. Moreover, QM-A filters have retained more mass and filtered bigger volumes compared to the GF/C filters.

Table 6.7. MuPFiS' results – 6th August 2008 test at Delft.

Location	Delft - consumer's house					
Run	20:30 - 22:30 (2h)		22:30 - 00:30 (2h)		00:30 - 02:30 (2h)	
Filter type	QM-A	GF/C	QM-A	GF/C	QM-A	GF/C
Mass retained (mg)	3,5	2,9	3,6	3,1	3,7	2,6
Volume filtered (l)	319	187	314	200	323	184

Both filters have retained sufficient mass; therefore the analysis of SS evolution along time has been done with the GF/C filter to compare the results with other tests.

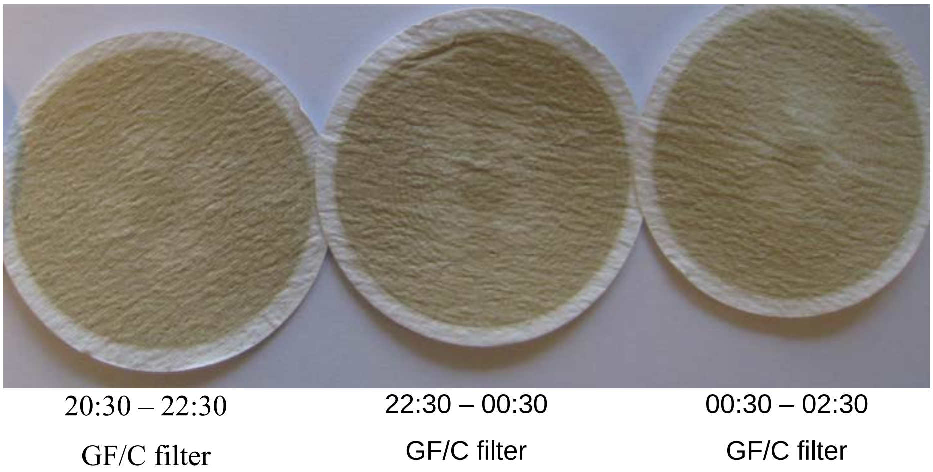


Figure 6.20. GF/C filters image – 6th August 2008 test at Delft.

TSS concentration is constant along time (Table 6.8 and Figure 6.21), between 14.2 and 15.4 µg/l. SS solids are principally composed of organic matter, 70-75% of VSS. No colouration variations are observed on filters (Figure 6.20).

Table 6.8. TSS, VSS and FSS concentration (µg/l) – 6th August 2008 test at consumer's house using GF/C filters.

	20:30 - 22:30	22:30 - 00:30	00:30 - 02:30
TSS concentration (µg/l)	15,5	15,5	14,2
FSS concentration (µg/l)	4,2	3,8	4,1
VSS concentration (µg/l)	11,3	11,8	10,1

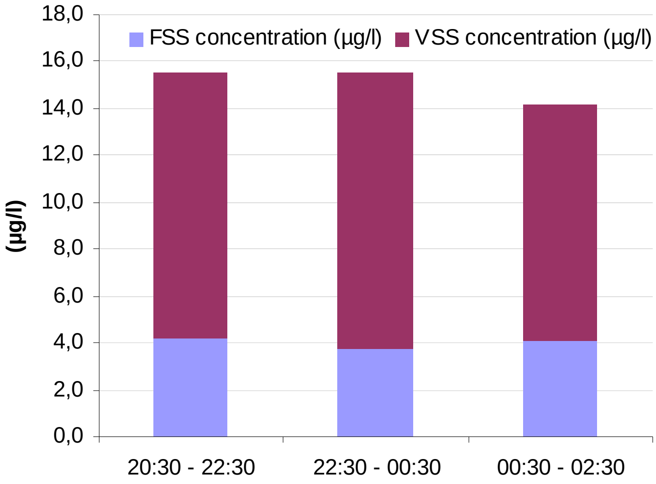


Figure 6.21. VSS and FSS concentration (µg/l) – 6th August 2008 test at consumer's house using GF/C filters.

Comparison between filters

QM-A filters have filtered bigger volumes of water and retained bigger amounts of mass, similar results were found during laboratory results. GF/C filters have measured major TSS concentrations, between 20-40%, than QM-A filters (Table 6.9).

Table 6.9. TSS, VSS and FSS concentration (µg/l) – 6th August 2008 test at consumer's house.

Run	20:30 - 22:30		22:30 - 00:30		00:30 - 02:30	
Type of filter	QM-A	GF/C	QM-A	GF/C	QM-A	GF/C
TSS concentration (µg/l)	11,0	15,5	11,5	15,5	11,5	14,2
FSS concentration (µg/l)	2,5	4,2	2,5	3,8	3,4	4,1
VSS concentration (µg/l)	8,4	11,3	8,9	11,8	8,1	10,1

No SS concentration variation occurs during the night (low flow condition). It is recommended to carry out more filtration tests during the day to determine if there are variations of SS concentration during normal and low flow conditions.

6.6 Overall discussion

6.6.1 Suspended Solids concentration

SS concentrations measured on the field were, in generally, very low compared to filtration tests carried at the laboratory (Table 6.10). This leads either to the retention of small SS masses, sometimes under the limits, or long filtration runs.

Table 6.10. TSS concentration (µg/l) in different points in the Netherlands.

	TSS concentration (µg/l)			
	Sampling point			
Localization	treatment plant	transportation network	distribution network, consumer house	distribution network, industrial area
Gouda	6.6*(1)		6.1*(1)	11.5(1)
Amsterdam		6.7 - 8.5*(1)		
Delft			14.2-15.5(1)	16-64(1)
Nieuwegein				150-560(2)

* The minimal 2.5 mg mass retention was not reached

(1) Results obtained employing a Whatman GF/C filter

(2) Results obtained employing a Whatman GF/F filter

As well, SS concentrations obtained in DWDS in Netherlands are low compared to values obtained by international research; (Inoue et al., 2004) measured 20 – 30 µg/l, (Matsui et al., 2007) measured 10 – 40 µg/l and (Gauthier et al., 2003) measured 44 µg/l. Lower concentrations in the Netherlands are obtained thanks to the multi barrier filtration approach. Indeed, in the Netherlands no chlorine disinfection is applied thus many filtration steps are implemented to obtain biological stable drinking water.

Field tests were performed at different locations during a day. Tests lasted different durations and had different number of runs:

- Test at Nieuwegein: 5-hour test and 15 runs.
- Test at Amstelveenseweg: 4-hour and 30 minutes test and 2 runs.
- Test at Gouda: three tests, 2-hour test at different locations.
- Test at Delft: 6-hour test and 3 runs.

Delft's, Gouda's (no comparable results along time) and Amstelveenseweg's tests did not record significant TSS variations. While Nieuwegein's test registered TSS variations. Nieuwegein's variations can be associated with steady state variations, variations observed at the beginning of tests caused by low flow conditions.

6.6.2 Volatile Suspended Solids concentration

VSS concentration at Amstelveenseweg (PS) and Gouda (PS) are very low (Figure 6.22), 2 and 2.8 µg/l, and represent a small portion of the TSS retained, 30 to 40%, thus proving the efficiency of the multiple barrier treatment systems.

High and low VSS concentrations have been found in DWDS:

- Different locations have different values, Gouda's consumer house 40% while Delft's consumers house 70% of VSS concentration.
- Different sampling periods offer different values, TUDelft's water laboratory the 6th August 40% and the 19th August 90% of VSS concentration.

Those variations in time and space prove that different degradation processes occur at different places and in different periods of time.

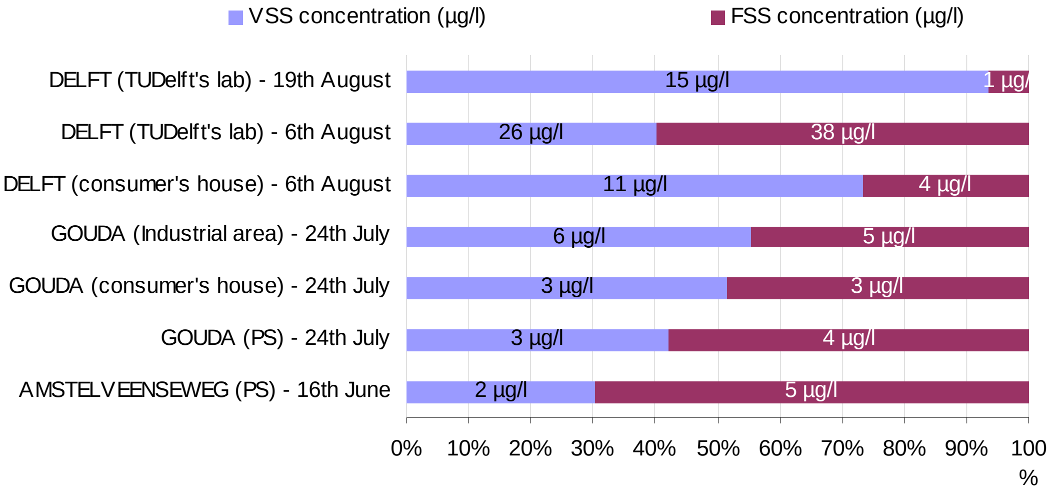


Figure 6.22. FSS and VSS concentration (% and µg/l) – field tests.

6.6.3 Type of filter

Field experiments have confirmed part of the results obtained during the tests done in the water laboratory of TUDelft.

Amstelveenseweg's, Gouda's (Oasen's office) and Delft's tests proved that bigger pores' size filters filter bigger volumes of water thus retain bigger amounts of TSS. Therefore, filters' pore size can be used as a tool to influence the filtration time and retention mass.

However, bigger pores size filters can misinterpret results in some special situations were small size ranged particles are the dominant fraction of SS. For instance at the consumer's house and at the pumping station of Gouda.

6.6.4 Filter selection

A filter selection procedure is defined for the selection of the filter (Figure 6.23). The procedure consists on the realisation of a previous filtration run to determine which the most adequate filter is. Until four different types of filters can be tested in parallel.

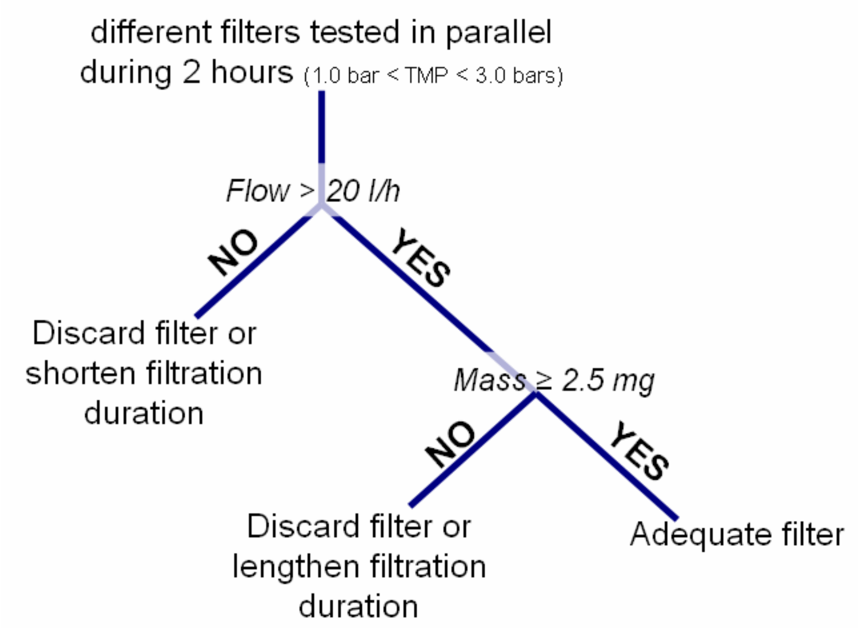


Figure 6.23. Filter decision procedure.

7 CONCLUSION AND RECOMMENDATIONS

Suspended solids (SS) in drinking water are one of the main causes of water quality deterioration occurring in drinking water distribution systems (DWDS).

The main objective of this project has been to contribute to the development of a new analytical method for characterizing and better understanding the behaviour of SS in DWDS. Particularly, the focus of this project has been the development and experimentation of a new method called Multiple Particle Filtration System (MuPFiS) for its application in the Netherlands' DWDS.

The experimental results obtained during this research allow the formulation of the following conclusions and recommendations.

7.1 Conclusions

This project has provided a new tool, the MuPFiS, capable of measuring and evaluating SS concentrations in DWDS.

Laboratory tests have been carried out to determine the influence of parameters affecting the filtration process and to develop and validate the proposed analytical method. MuPFiS' standard filtration conditions have been defined: trans-membrane pressure (TMP) of 2 bars, filtration rate higher than 20 l/h and 2-hour filtration runs.

A TMP of 2 bars is available in most DWDS. Water meters accuracy do decrease when their flow through is under 20 l/h. A 2-hour filtration period has been chosen to optimize the shortest filtration duration and to ensure enough mass retention on the filters.

The filter used may influence the volume filtered, the mass retained and the SS concentration measured under similar filtration conditions. Whatman QM-A 2.1-µm pore size filters allow filtration of larger water volumes (+20 to +75%) and generally retain bigger amounts of solids (+15% to +42%) as compared to Whatman GF/C 1.2-µm pore size filters. However, QM-A filters retain smaller TSS concentrations (-10% to -30%) because of differences in the particle sizes retained. GF/C filters retain particles in the 1.2 to 2.1 µm size range in addition to particles retained by QM-A filters.

MuPFiS' detection accuracy was assessed according to defined standard procedures. TSS' concentration accuracy is ±0.2 mg of the mass retained, plus ±10% of the volume filtered; FSS' concentration accuracy is ±0.7 mg of the mass retained, plus ±10% of the volume filtered with GF/C Whatman filters.

Field testing of the analytical method has proved the high quality of Netherlands' drinking water; low SS concentrations have been found in Netherlands' DWDS (6.6 to 15.5 µg/l) compared to values (10 to 44 µg/l) obtained by international research (Gauthier et al., 2001, Inoue et al., 2004, Matsui et al., 2007). In addition, SS degradation processes occurring in DWDS have been characterized. Some special locations (industrial areas with oversized networks) or specific time periods (after low flow episodes) have lead to higher SS concentrations (15.5 to 550 µg/l) because of low flow conditions.

Finally, a comparison of TILVS and MuPFiS has shown that the MuPFiS is a more versatile and practical tool: filtration runs are shorter, more filtration runs can be done during a given time period, its multiple filtration lines offer larger analytical possibilities, and it does not need an electrical power supply.

7.2 Recommendations

It is recommended that future research is focused on carrying out additional field tests with the MuPFiS. Field tests should focus on water distribution areas with water quality problems and especially on locations were these deteriorations do occur. Several MuPFiS units can be simultaneously used to sample different points.

Tests carried out during this project have identified the variation of SS concentration as well as that of SS organic and inorganic fractions. It is recommended that future research includes elemental analyses of the filters to evaluate the chemical composition of the SS retained.

The MuPFiS and the particle counter have been compared using laboratory tests. Combining quantitative mass measurements with particle volume measurements would allow calculation of SS densities. It is recommended that further research be carried out on the potential of linking results from both tools. Laboratory experiments done with both apparatus in parallel could be completed by field tests.

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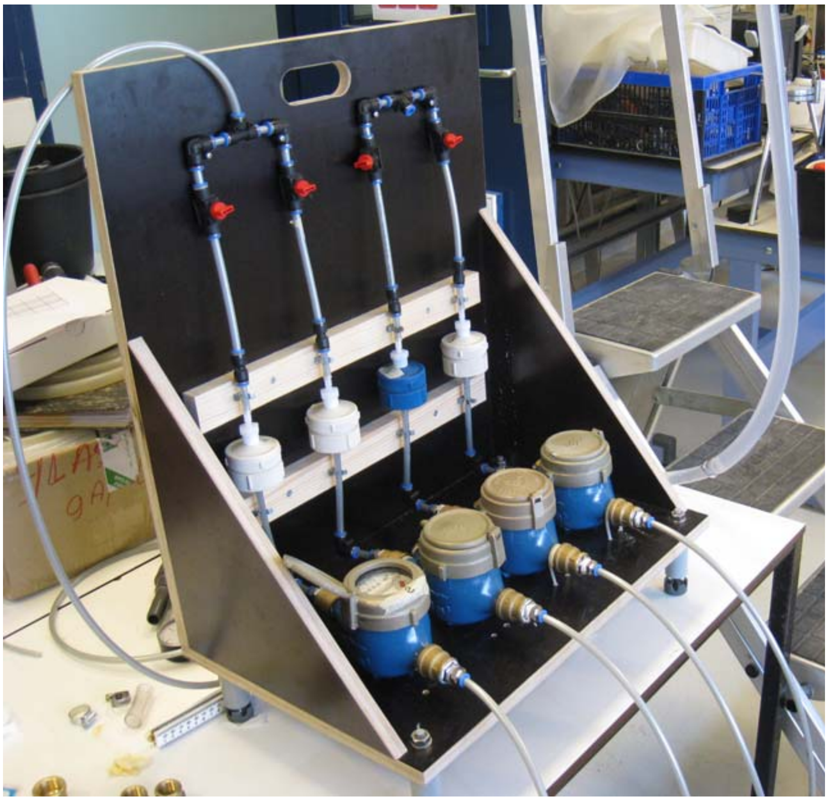
APPENDIX I:

MuPFiS test description



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July 2008

Introduction

This manual describes the operational methodology used for the realization of tests with the new developed tool MuPFiS.

This manual has been developed based on the Standard Methods for the Examination of Water and Wastewater (Greenberg et al., 1999) 20th Edition, as well as, the experiences obtained during the test realization at the TUDelft water laboratory.

General considerations

Weighting

The same valance must be used during the entire process. This will avoid possible errors due to different calibration of the different balances.

For this test we are interested in the mass variation of the filter before and after the filtration. As the mass is very small the measurements need to be very accurate.

Recommendations for weighting:

1. follow the balance instructions furnished by the fabricant;
2. after each measurement check if the balance value comes back to zero, if not repeat the measurement after setting up a new zero;
3. after each measurement re-zero the balance;
4. place the samples with care onto the balance, do not let them fall;
5. close the room doors during the measurements, in order to create the most stable environment;
6. manipulate the filters with tweezers and the recipients with the forceps as much as it is possible;
7. wait until the appearing value is stable;
8. do not use the support where the balance is placed while the measurements, otherwise, the results could be modified.

Filter supports

The filters are manipulated during all the process using Petri dishes (Figure 1).

Filters are placed over an absorbent towel inside the Petri dish. The absorbent towel can be a Kimberly-Clark Kimtech absorbent towel or equivalent.

Petri dishes and absorbent towels are numbered.

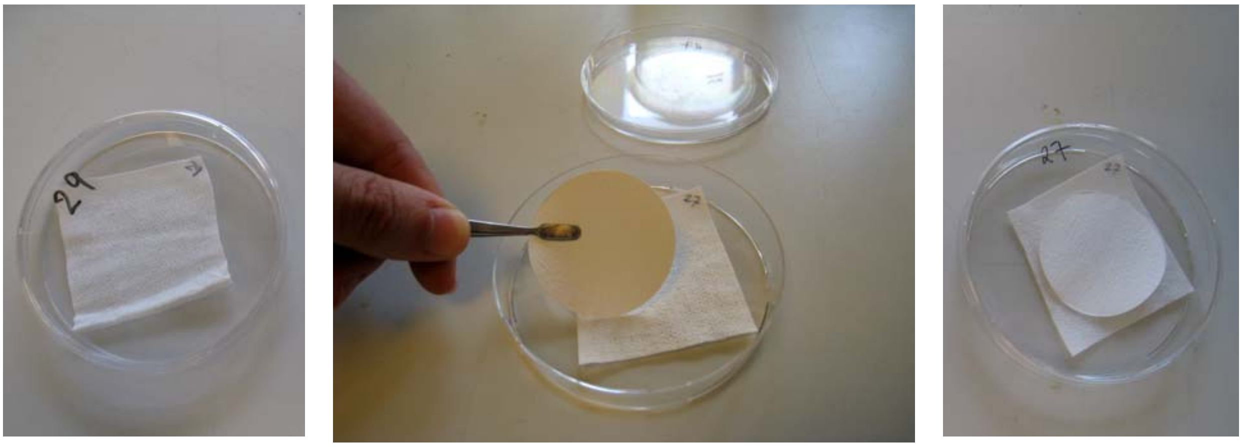


Figure 1. Petri dish before placing a filter, during manipulation and with a filter.

Biological growth

To avoid biological growth or interference, all glass and plastic used for the preparation and handling of the filters will have be bathed in a dilution of AR grade nitric acid before use.

When the 8mm plastic tubes used for the connections present biological growth they can be replaced by new ones of the same characteristics.

Filtration procedure

Preparation of filters

Filters must be cleaned before use, this is the process used to clean a filter:

1. The filter is washed with demineralized water using a vacuum (Figure 2) the wrinkled side is put in the bottom, and approximately 100 ml of demineralized water are used for each flushing;

Figure 2. Flushing of the filter with a vacuum.

2. the flushed filter is placed in a numbered Petri dish, the number of the Petri dish is assigned to the filter;
3. the filter is taken out of the Petri dish with the adsorbent towel and placed into the oven, the filter is dried in an oven at 103 °C during two hours. Petri dishes can not be placed in the oven. Filters with absorbent towels can be placed in other recipients to make easier the manipulations, for instance in an aluminum support (Figure 3);

Figure 3.Aluminium support used for filter manipulation in the oven and desiccator.

4. then the filter must be cooled in a desiccator (Figure 4) during one hour, in order to remove the steam and let it recover to the ambiant temperature;

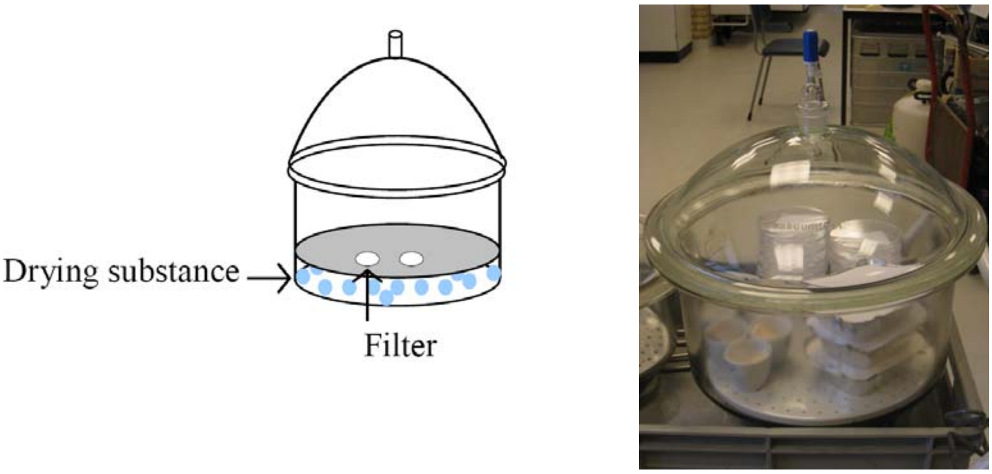


Figure 4. Desiccator.

5. finally, the filter is weighted (W_0) and placed in the correspondent Petri dish.

W_0 = weight of the filter cleaned

Filters must be stored in a desiccator until they are going to be used.

Filtration process

The main purpose of the filtration process is to obtain representative samples of the analyzed water. In order to achieve that objective the following process is recommended to be used.

1. Before the utilization of the MuPFiS the apparatus must be flushed to renew water present in the MuPFiS;
2. Installation of a filters:
    - Place the discharge tubes in a point of the same height as the filters, this will avoid air enter the system when the filter holders will be opened;
    - Install the filter in the filter holder, the wrinkled side is put in the bottom, if necessary; rinse the filter with demineralized water;
    - Close the filter holders and place again the discharge tubes in the evacuation water system;
3. Before starting the filtration check if the upstream pressure is 2.5 bars (Table 1), the pressure can be modified by a controlling valve or the used tap. Downstream pressure is zero because water is discharged when filtered;
4. To start the filtration, open slowly the valves.

When installing the filters it is important to avoid air entering the system, air will influence water volumes measurements.

The filtration run duration must be of 2 hours (Table 1). The filtration run duration can be modified if:

1. the filtration flow decreases under 20 l/h before the finalisation of the run, then the filtration duration will be reduced;
2. the retained mass is not sufficient for the analysis after 2 hours of filtration, then the filtration run can be prolonged.

If the filtration time is modified the new duration time will have to be applied to all the runs during the same MuPFiS' experience.

Table 1. MuPFiS’ filtration parameters.

Filtration parameter	Value
Trans membrane pressure (TMP)	2.5 bars
Filtration duration	2 h*

The filter holder must be closed to ensure that there will be no leakage, it is recommended not to use a very high force when closing the filters, otherwise, the filter could be damaged.

Measurement of TSS

Analysis of Total Suspended Solids at 103-105 ˚C

Process

1. the water is filtered through a weighed standard microfiltration filter until the weight of the dried retained matter is between 2.5 and 200 mg;
2. after the filtration the filter is placed in the correspondent Petri dish (Figure 6);
3. the filter is dried in an oven at 103 ˚C during 2 hours, the filter is placed inside the oven with the absorbent towel, if necessary use some recipient for easy manipulation of filters, for instance an aluminum container;
4. afterwards, the filter must be cooled in a desiccator during one hour, in order to remove the steam and let it recover de ambiance temperature;
5. the filter is weighted together (W_1).

W_1 = weight of the filter after filtration

$TSS = W_1 - W_0$

Figure 5. Removal of a filtered filter from the filter holder.

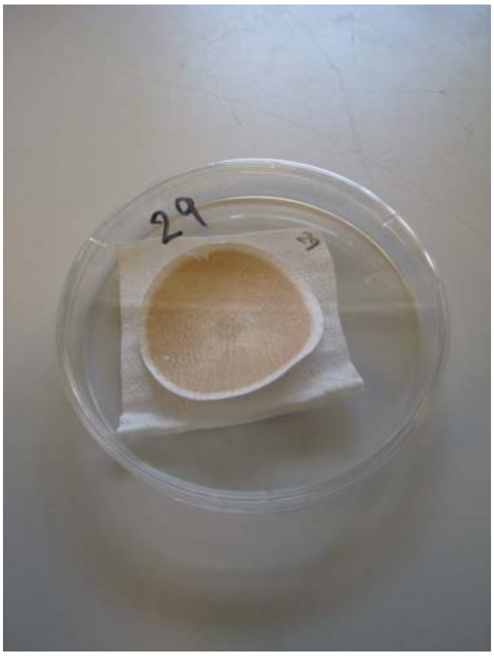


Figure 6.Filter after filtration run placed in the Petri dish.

Measurement of VSS

Analysis of Fixed and Volatile Solids Ignited at 550 ˚C

Process:

1. the filter from the analysis of Total Suspended Solids is weighted (W_1) and then placed in a numbered ceramic crucible, the ceramic crucible is covered with a lid to avoid matter enter or exit the recipient (Figure 7);

Figure 7. Ceramic crucible with a filter and ceramic crucible covered.

2. the crucible and filter are combusted at 550 ˚C during two hours (Figure 8) the filter is covered to avoid matter enter or exit the crucible;

Figure 8. Oven used for combustion at 550 ºC.

3. then the filter and crucible are cooled in a desiccator during one hour, in order to recover de ambiance temperature. The crucibles and filters should not be placed directly from the oven to the desiccator. It is recommended to let them cool a few minutes before placing them into the desiccator;
4. finally, the filter is weighted (W_2) to determine the amount of fixed solids remaining in it.

Mass lost of the filter when combustion

Previous tests have showed that in an inorganic filter (e.g. glass fiber filter) there is a small fraction of organic components (Sagel, 2007). Thus, when heating at 550 ˚C a small part of the filter will be combusted.

(Sagel, 2007) measured that the mass loss of a GC/C filter during combustion ($filterloss_{GF/C}$) is of 1.3% ±0.6% of its mass.

Test realized with blanks in the water laboratory of TUDelft have shown that the mass loss when combusting a QM-A filter ($filterloss_{QM-A}$) is of 0.6% ±0.06% of its mass.

$$filterloss_{GF/C} = 0.013 \pm 0.006$$

$$filterloss_{QM-A} = 0.006 \pm 0.0006$$

$$W_L = W_0 \times filterloss$$

W_1 = weight of the filter before combustion

W_2 = weight of the filter after combustion

W_L = mass lost of the filter when during combustion (calculation)

$$VSS = W_2 - W_1 - W_L$$

Storage of filtration papers, clean and used

Every recipient must be cleaned and dried after being used. Clean recipients are stored in the desiccators until they need to be used.

Filter choice

A filter selection procedure is defined for the selection of the filter (Figure 9). The procedure consists on the realisation of a previous filtration run to determine which the most adequate filter is. Until four different types of filters can be tested in parallel.

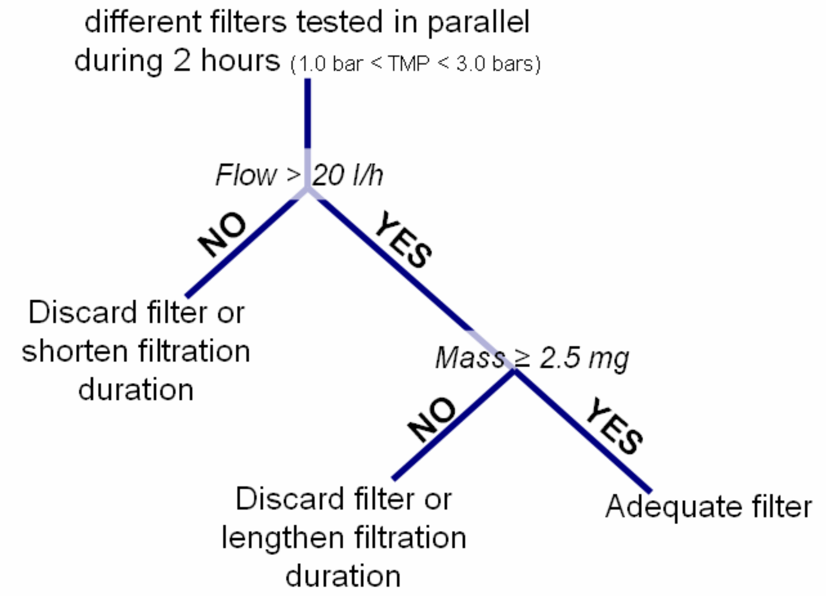


Figure 9. Filter decision procedure.

References

GREENBERG, A. E., CLESCERI, L. S. & EATON, A. D. (1999) Standard Methods for the Examination of Water and Wastewater, American Water Works Association.

SAGEL, L. (2007) Suspended solids in drinking water systems: concentration and composition.

Filtration form

Date	place
Commentaries	

	Branch Type of filter				
Filter cleaning	Nº of crystal recipient				
	Weight filter cleaned				
Filtration	Time start filtration				
	Initial volume				
	Final volume				
	Time end filtration/ duration				
After filtration	Weight filter				
Combustion	Nº of porcelain vessel				
	weight filter before combustion				
	weight filter after combustion				

APPENDIX II

Particle counter and MuPFiS results

4th of June 2008 test

Test done in the TUDelft laboratory the 4th of June 2008 with the MuPFiS and particle counter working in parallel.

Table 1. MuPFiS' results from the test done the 4th of June 2008.

	run 1	run 2	run 3	run 4	run 5	run 6
Duration test	9:05 - 10:05	10:05 - 11:05	11:10 - 12:10	12:10 - 13:10	13:10 - 14:10	14:10 - 15:10
Filtered volume (l)	109,2	87,5	77,3	78,8	95,7	87,2
TSS concentration (µg/l)	28	38	40	41	29	33
FSS concentration (µg/l)	8	12	16	17	11	11
VSS concentration (µg/l)	21	26	24	24	19	22

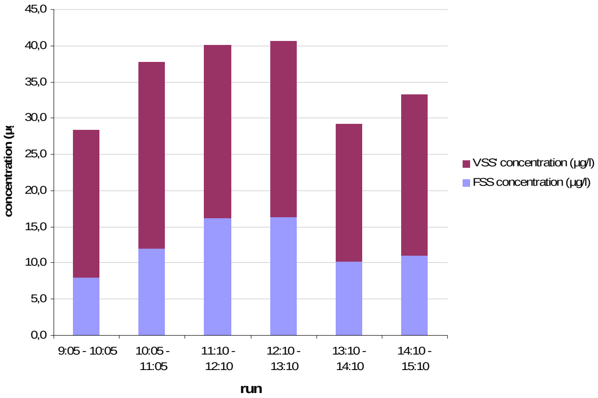


Figure 1. FSS and VSS concentration (µg/l) obtained the 4th of June 2008 with the MuPFiS.

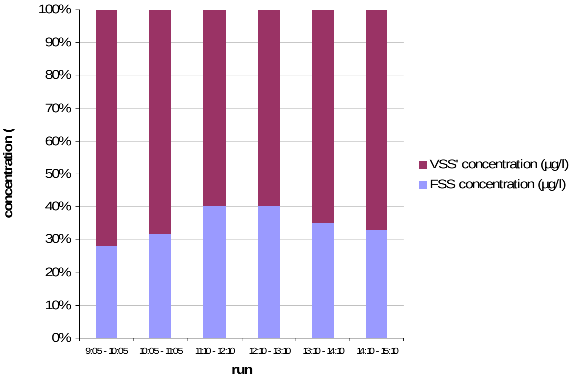
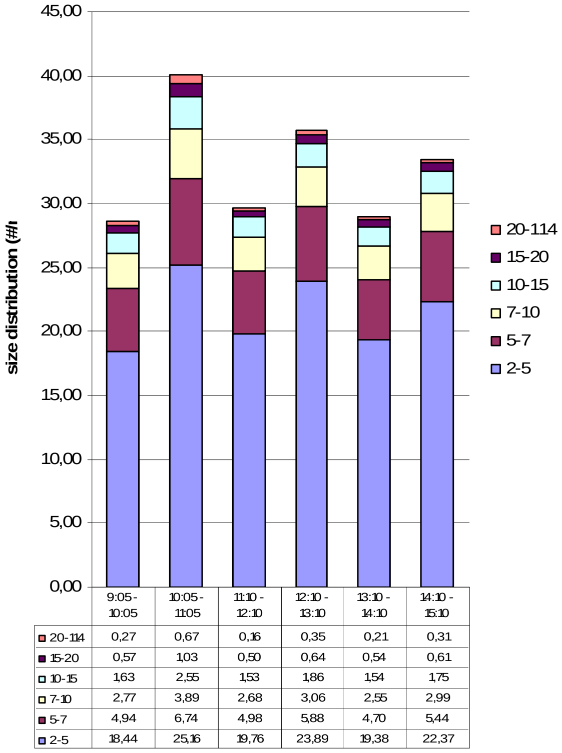


Figure 2. FSS and VSS concentration (%) obtained the 4th of June 2008 with the MuPFiS.



	9:05 - 10:05	10:05 - 11:05	11:10 - 12:10	12:10 - 13:10	13:10 - 14:10	14:10 - 15:10
■ 20-114	0,27	0,67	0,16	0,35	0,21	0,31
■ 15-20	0,57	1,03	0,50	0,64	0,54	0,61
■ 10-15	1,63	2,55	1,53	1,86	1,54	1,75
■ 7-10	2,77	3,89	2,68	3,06	2,55	2,99
■ 5-7	4,94	6,74	4,98	5,88	4,70	5,44
■ 2-5	18,44	25,16	19,76	23,89	19,38	22,37

Figure 3. Particle size distribution (#/ml) per ranges, particle counter test of 4th of June 2008.

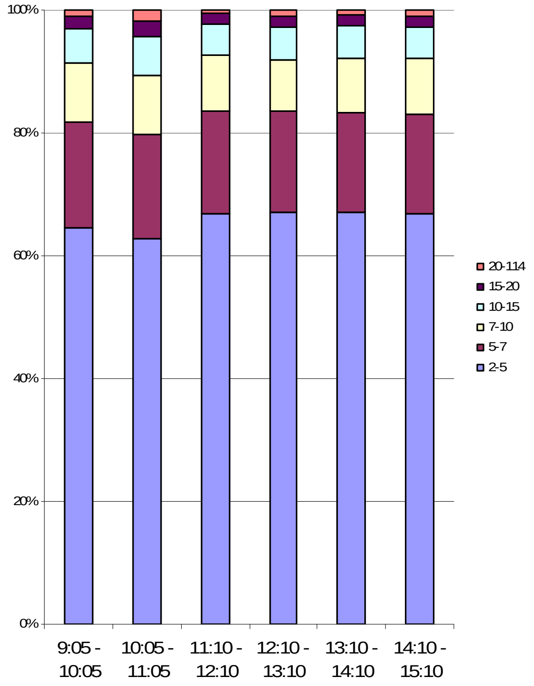
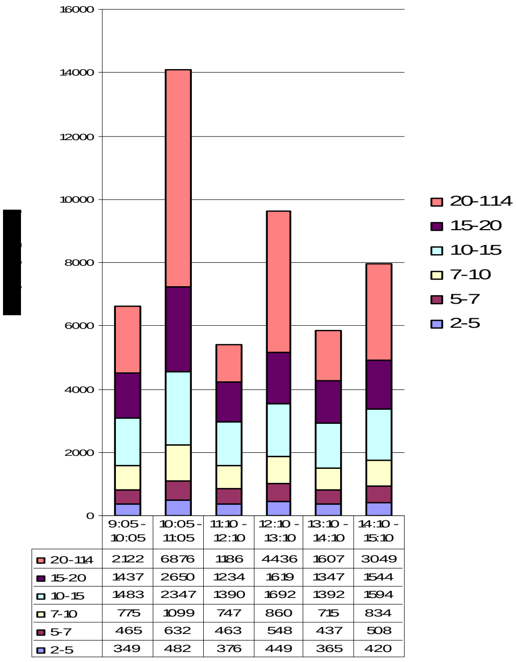


Figure 4. Particle size distribution (%) per ranges, particle counter test of 4th of June 2008.

[redacted]



	9:05 - 10:05	10:05 - 11:05	11:10 - 12:10	12:10 - 13:10	13:10 - 14:10	14:10 - 15:10
■ 20-114	2122	6876	1186	4436	1607	3049
■ 15-20	1437	2650	1234	1619	1347	1544
■ 10-15	1483	2347	1390	1692	1392	1594
■ 7-10	775	1099	747	860	715	834
■ 5-7	465	632	463	548	437	508
■ 2-5	349	482	376	449	365	420

Figure 5. PVC (µm³/ml) per ranges, particle counter test of 4th of June 2008.

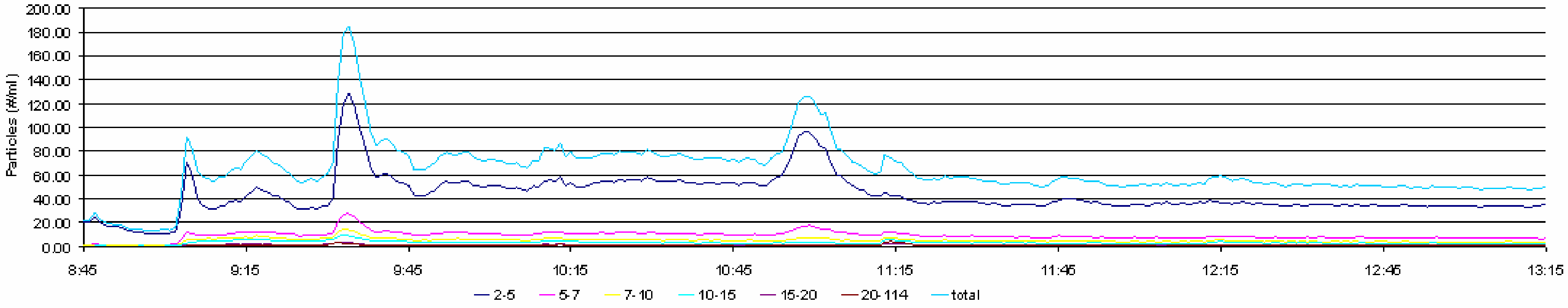


Figure 22. Size distribution (#/ml) per ranges, particle counter test of 16th of June 2008.

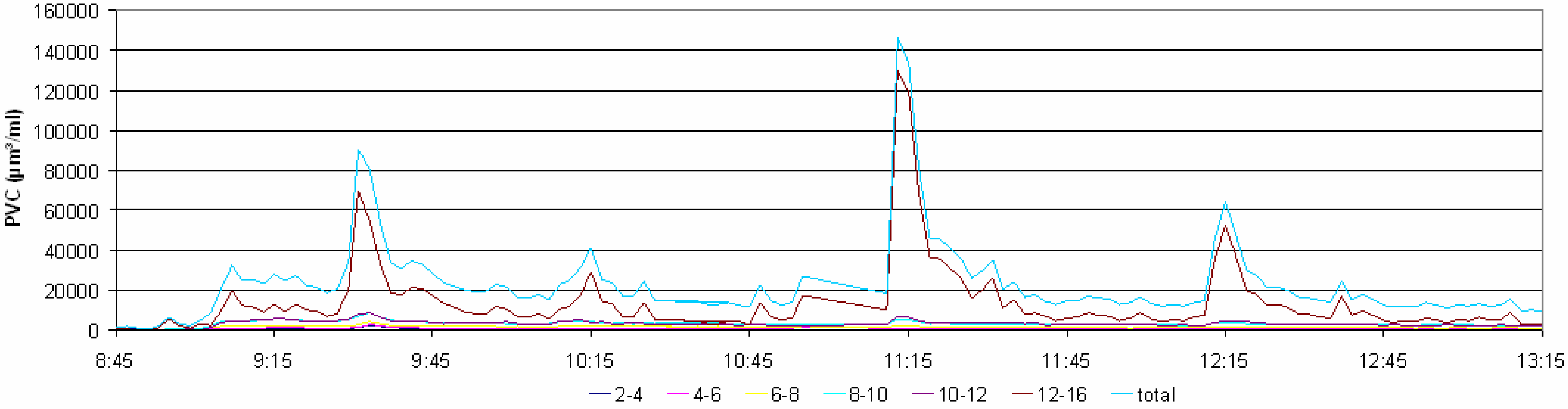


Figure 23. PVC (µm³/ml) per ranges, particle counter test of 16th of June 2008.

6th of June 2008 test

Test done in the TUDelft laboratory the 6th of June 2008 with the MuPFiS and particle counter working in parallel.

Table 2. MuPFiS' results from the test done the 6th of June 2008.

	run 1	run 2	run 3	run 4
Duration test	8:55 - 9:55	9:55 - 10:55	10:55 - 11:55	11:55 - 12:55
Filtered volume (l)	41,3	61,3	108,5	126,5
TSS concentration (µg/l)	51	41	27	20
FSS concentration (µg/l)	16	19	9	4
VSS concentration (µg/l)	35	22	18	16

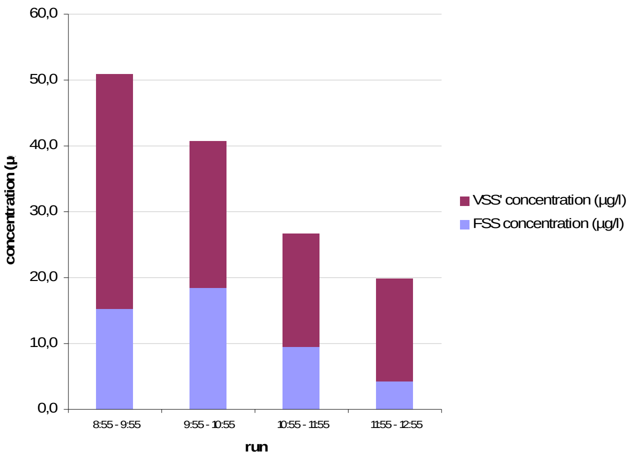


Figure 6. FSS and VSS concentration (µg/l) obtained the 6th of June 2008 with the MuPFiS.

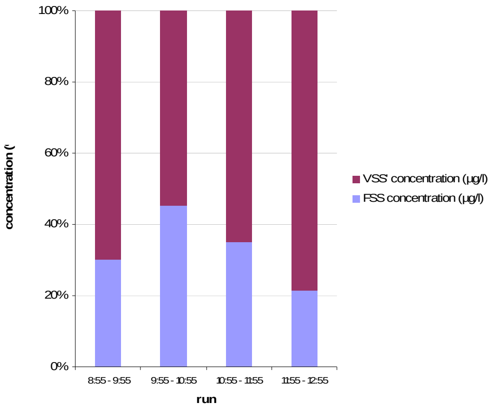
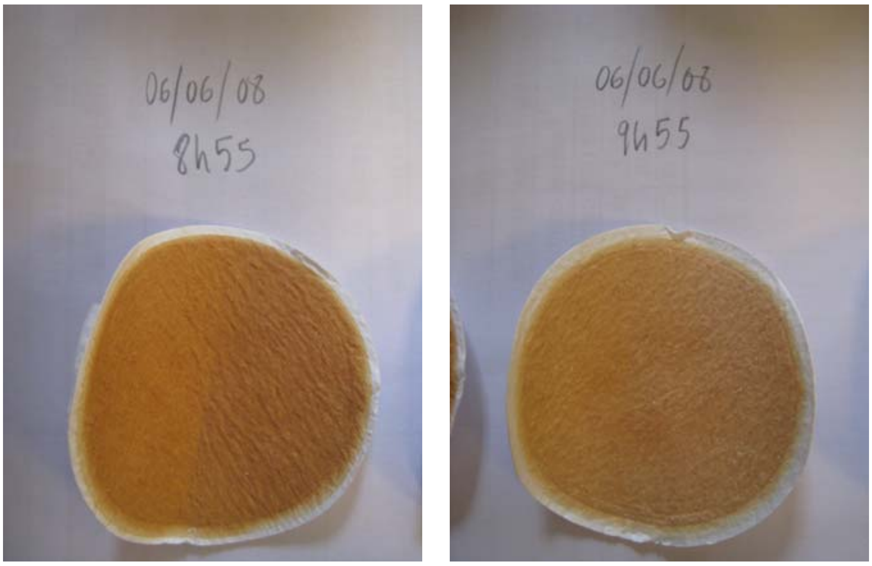
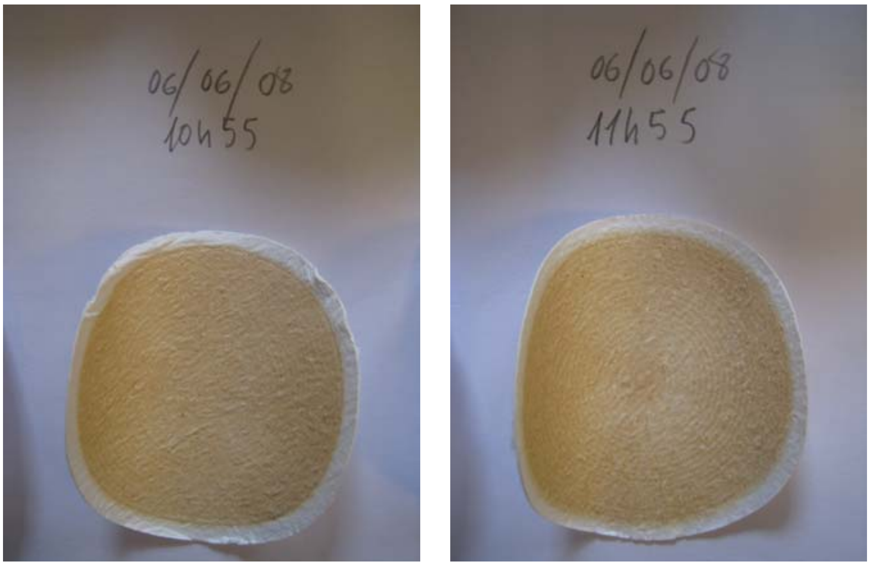


Figure 7. FSS and VSS concentration (%) obtained the 6th of June 2008 with the MuPFiS.

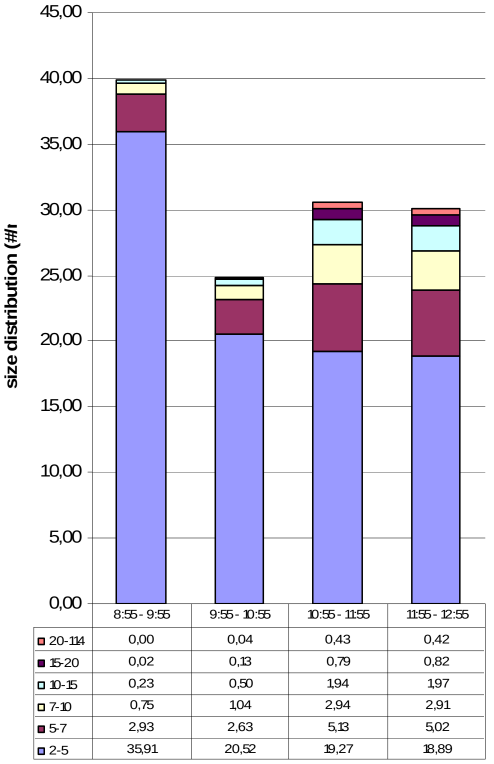


Run 1, 8:55 to 9:55 Run 2, 9:55 to 10:55



Run 3, 10:55 to 11:55 Run 4, 11:55 to 12:55

Figure 8. Photographs taken from the filters after the tests done with MuPFiS the 6th of June 2008.



	8:55 - 9:55	9:55 - 10:55	10:55 - 11:55	11:55 - 12:55
20-114	0,00	0,04	0,43	0,42
15-20	0,02	0,13	0,79	0,82
10-15	0,23	0,50	1,94	1,97
7-10	0,75	1,04	2,94	2,91
5-7	2,93	2,63	5,13	5,02
2-5	35,91	20,52	19,27	18,89

Figure 9. Particle size distribution (#/ml) per ranges, particle counter test of 6th of June 2008.

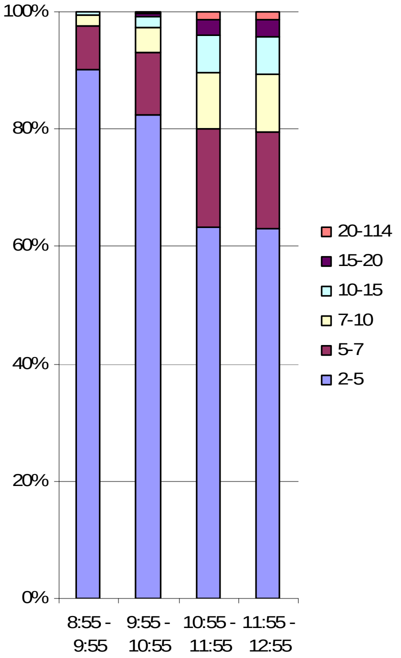
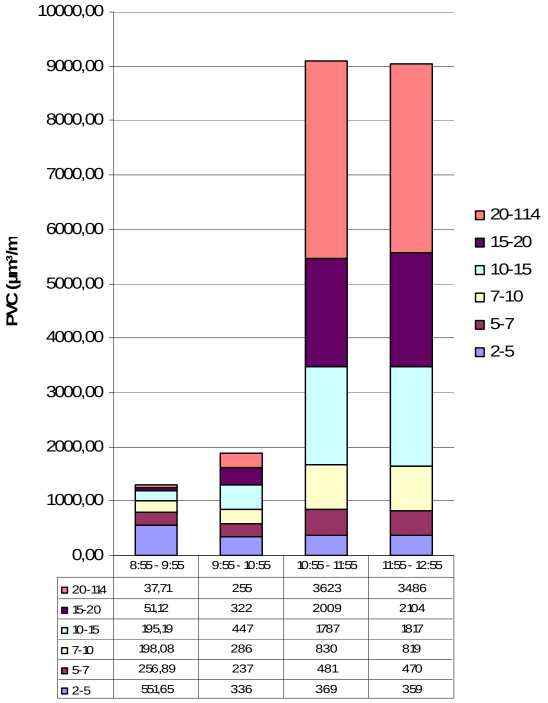


Figure 10. Particle size distribution (%) per ranges, particle counter test of 4th of June 2008.



	8:55 - 9:55	9:55 - 10:55	10:55 - 11:55	11:55 - 12:55
20-114	37,71	255	3623	3486
15-20	51,12	322	2009	2104
10-15	195,19	447	1787	1817
7-10	198,08	286	830	819
5-7	256,89	237	481	470
2-5	551,65	336	369	359

Figure 11. PVC (µm³/ml) per ranges, particle counter test of 6th of June 2008.

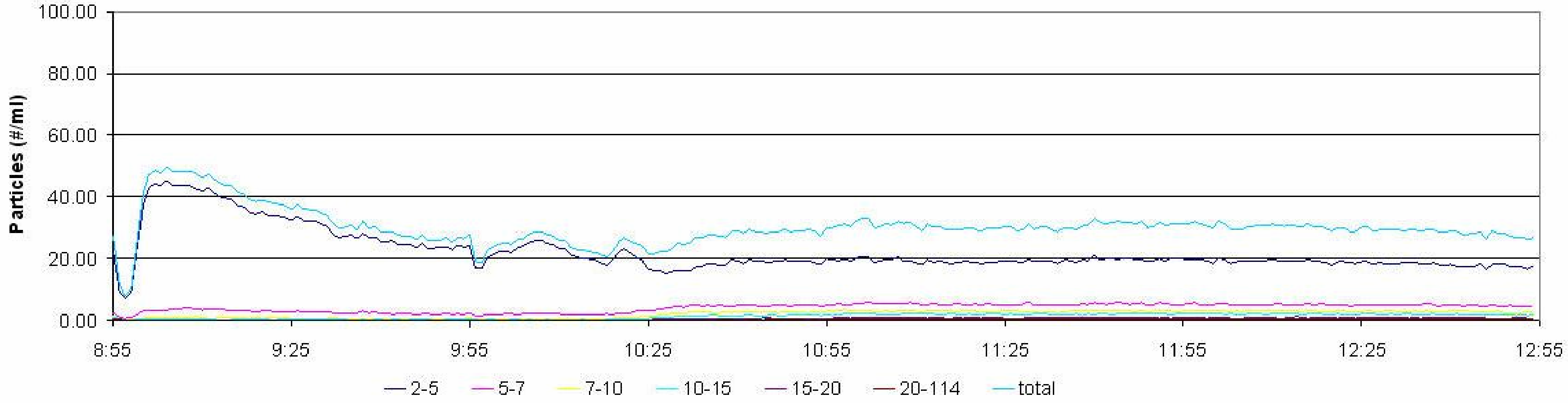


Figure 12. Size distribution (#/ml) per ranges, particle counter test of 6th of June 2008.

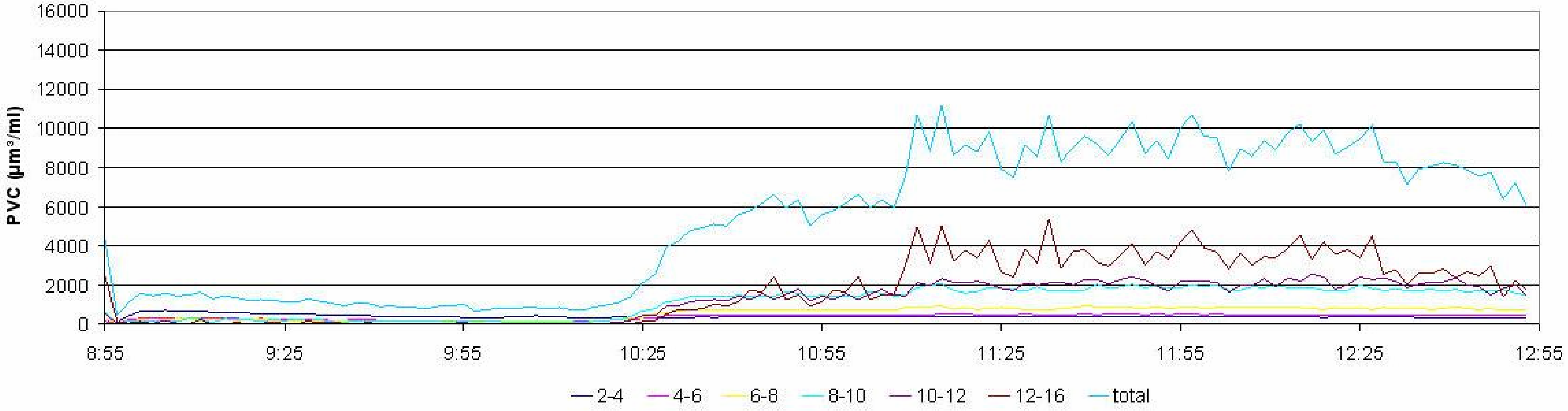


Figure 13. PVC (µm³/ml) per ranges, particle counter test of 6th of June 2008.

16th of June 2008 test

Test done in the TUDelft laboratory the 16th of June 2008 with the MuPFiS and particle counter working in parallel.

Table 3. MuPFiS' results from the test done the 16th of June 2008.

	run 1	run 2	run 3	run 4	run 5
Duration test	8:45 - 9:35	9:35 - 10:15	10:15 - 11:15	11:15 - 12:15	12:15 - 13:15
Filtered volume (l)	65,3	54	73,5	87,2	92,5
TSS concentration (µg/l)	51	59	44	46	41
FSS concentration (µg/l)	25	23	15	14	15
VSS concentration (µg/l)	26	37	29	32	26

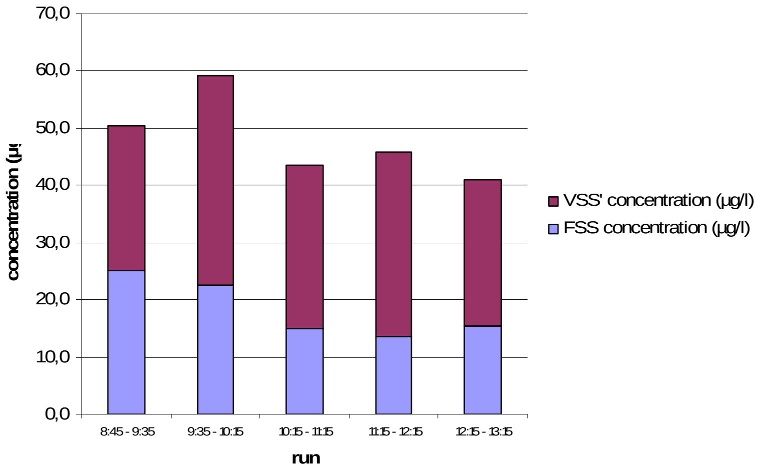


Figure 12. FSS and VSS concentration (µg/l) obtained the 16th of June 2008 with the MuPFiS.

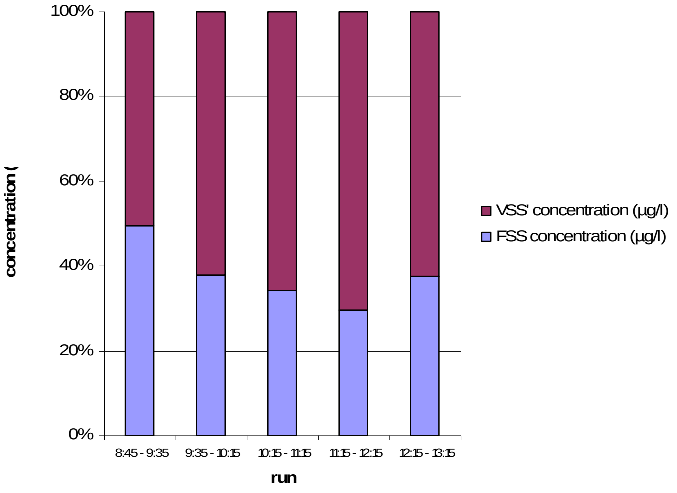
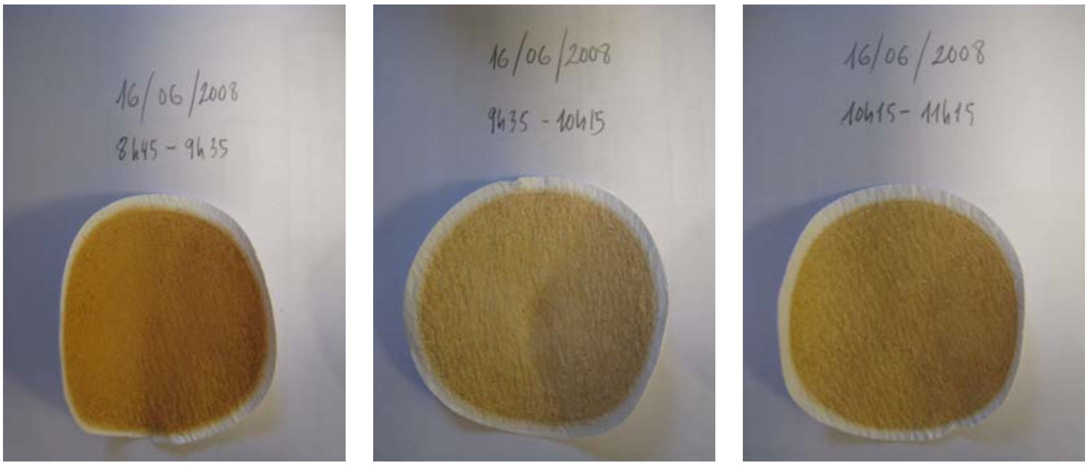
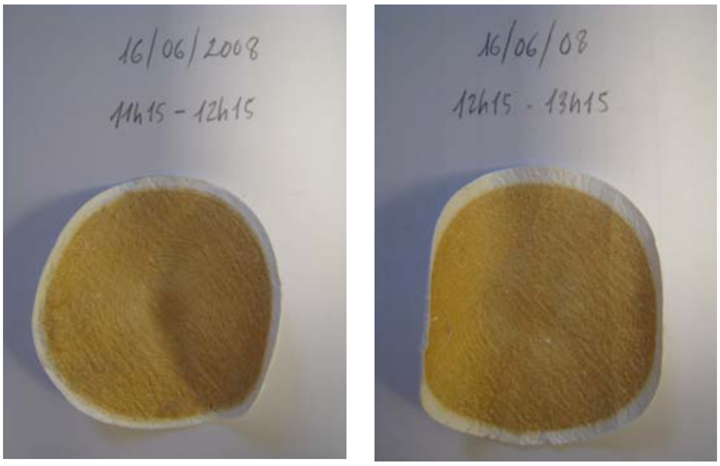


Figure 13. FSS and VSS concentration (%) obtained the 16th of June 2008 with the MuPFiS.

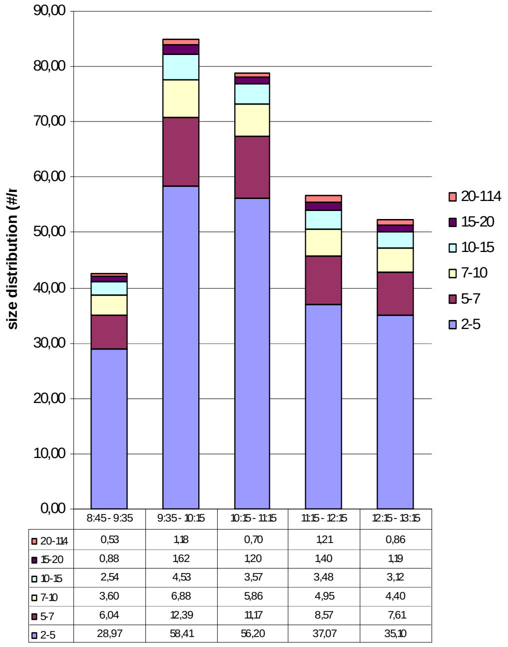


Run 1, 8:55 to 9:35 Run 2, 9:35 to 10:15 Run 3, 10:15 to 11:15



Run 4, 11:15 to 12:15 Run 5, 12:15 to 13:15

Figure 14. Photographs taken from the filters after the tests done with MuPFiS the 16th of June 2008.



	8:45 - 9:35	9:35 - 10:15	10:15 - 11:15	11:15 - 12:15	12:15 - 13:15
🟥 20-114	0,53	1,18	0,70	1,21	0,86
■ 15-20	0,88	1,62	1,20	1,40	1,19
□ 10-15	2,54	4,53	3,57	3,48	3,12
□ 7-10	3,60	6,88	5,86	4,95	4,40
■ 5-7	6,04	12,39	11,17	8,57	7,61
🟦 2-5	28,97	58,41	56,20	37,07	35,10

Figure 15. Particle size distribution (#/ml) per ranges, particle counter test of 16th of June 2008.

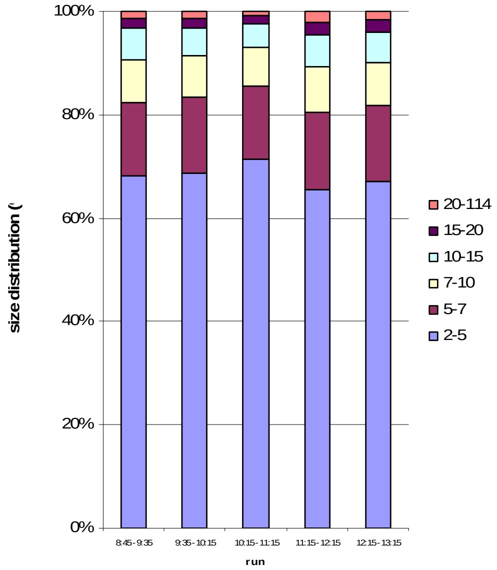
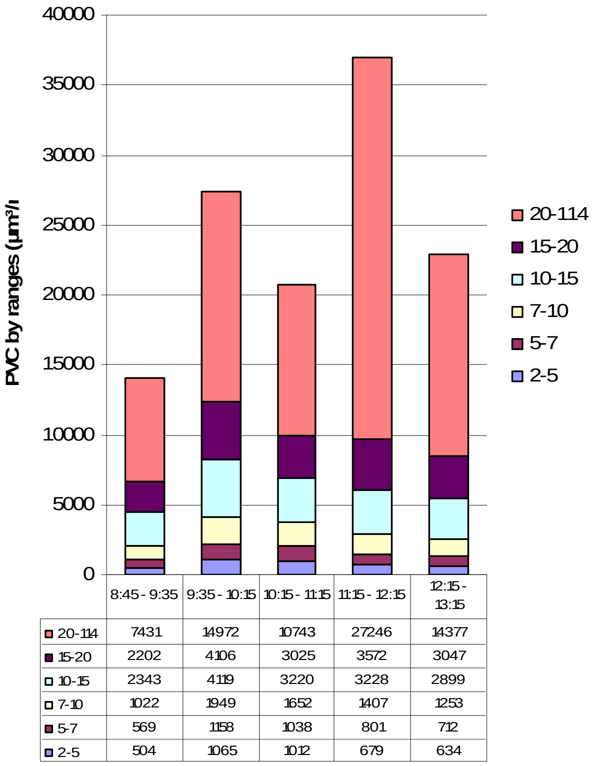


Figure 16. Particle size distribution (%) per ranges, particle counter test of 16th of June 2008.



	8:45 - 9:35	9:35 - 10:15	10:15 - 11:15	11:15 - 12:15	12:15 - 13:15
🟥 20-114	7431	14972	10743	27246	14377
■ 15-20	2202	4106	3025	3572	3047
□ 10-15	2343	4119	3220	3228	2899
□ 7-10	1022	1949	1652	1407	1253
■ 5-7	569	1158	1038	801	712
🟦 2-5	504	1065	1012	679	634

Figure 17. PVC (µm³/ml) per ranges, particle counter test of 16th of June 2008.

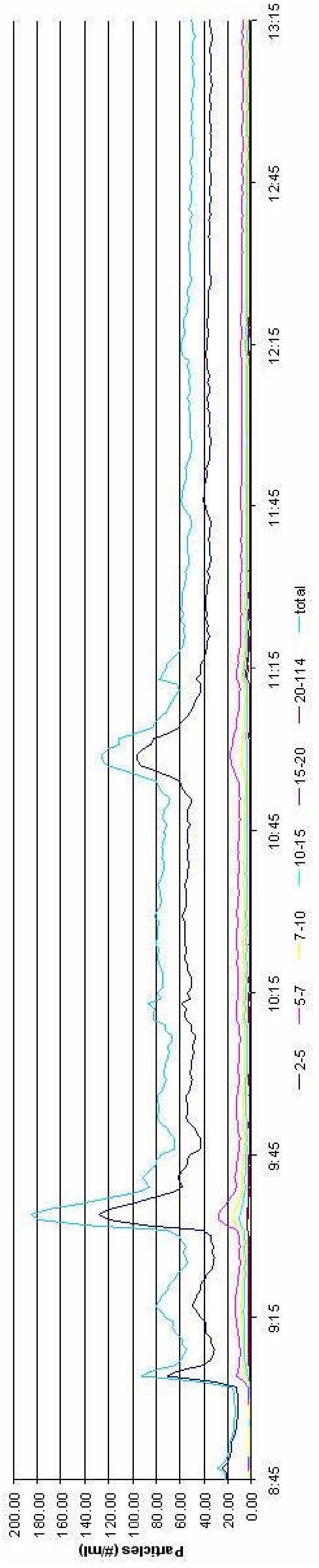


Figure 18. Size distribution (#/ml) per ranges, particle counter test of 16th of June 2008.

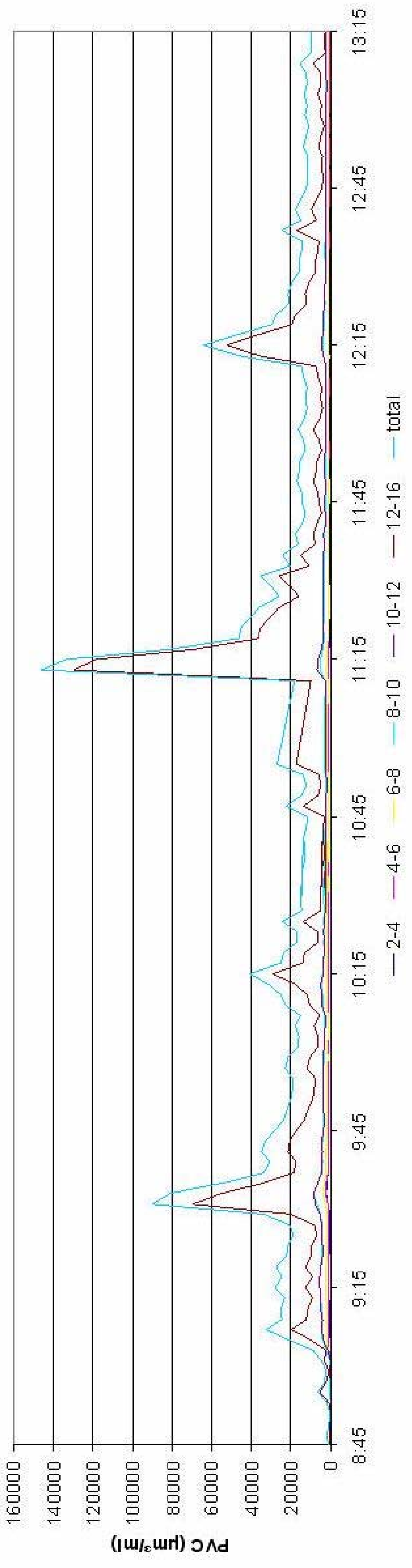


Figure 19. PVC (µm³/ml) per ranges, particle counter test of 16th of June 2008.