Standardisation of the Use of Particle Counting for Potable Water Treatment in South Africa

AD Ceronio, J Haarhoff & M Pryor

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Water Research
Commission

Standardisation of the Use of Particle Counting for Potable Water Treatment in South Africa

by

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Umgeni Water

Report to the Water Research Commission on the Projects :
WRC Project K5/1024 : Evaluation of Phase Removal Processes at Full-Scale South African Water Treatment Plants in terms of Particle Size and Number
and
WRC Project K5/1025 : Investigation into the Use of Particle Size Analysis for Monitoring and Optimising Plant Performance for the Production of Potable Water

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EXECUTIVE SUMMARY

This document is the final report to the Water Research Commission for the following projects and is a collaboration between the Water Research Group of the Rand Afrikaans University and Umgeni Water:

- K5/1024 : Evaluation of Phase Removal Processes at Full-Scale South African Water Treatment Plants in terms of Particle Size and Number
- K5/1025 : Investigation into the Use of Particle Size Analysis for Monitoring and Optimising Plant Performance for the Production of Potable Water

Significant progress has already been made in introducing particle counting to potable water treatment in other countries, specifically the USA, and a significant body of work has already been published on this subject. It is not the intention of this report to repeat this work, but rather to standardise the use of the technology in this country. In this way, several problems that have been experienced elsewhere can be avoided.

This document aims to assist users and prospective users of particle counting technology and procedures in the water treatment field in the understanding of the basic principles of this technology. To this end the authors :

- explain the basic concepts and terminology that prospective users will come across,
- highlight some of the standards that are applied in the particle counting industry,
- include some guidelines on the use of particle counters in general,
- offer some guidelines on the handling of the data generated by the particle counters, and
- present results from case studies in order to illustrate the application of particle counting in potable water treatment.

The report is presented in four parts, each dealing with a different facet of particle counting. Each of the sections are summarised below.

Hardware and Methodology

Particle counting brings a new technology that is finding increasing acceptance in the potable water treatment field. With it comes several new concepts and a collection of terminology and jargon that is not familiar to practitioners in the water field. These concepts are explained in order to familiarise the water treatment practitioners with this technology. In addition, various types of particle counters are available and it is important to note that different particle counters will characterise the same sample differently due to fundamental reasons. ESZ (Electron Sensing Zone) sensors define particles by their

conductivity, light scattering sensors by their refractive index, and light blockage sensors by their degree of translucence and refractive index. The orientation of the particle passing through the sensor also plays a significant role during measurement. It must therefore be accepted that some variation will be evident when the same sample is re-measured on the same instrument and more so when different technologies are being compared. These variances are increased when different measurement techniques and procedures are followed - therefore the need for standardisation in the use of particle counters.

Some standards have been published to ensure that particle counters achieve some level of standardisation in its manufacture and calibration. These standards have their origins in other industries and, to date, no such standard has been published for the potable water industry. This problem is discussed. The discussion concludes with the following recommendations:

- particle counters should only be acquired if they conform to JIS B 9921:1997 in terms of its manufacture,
- particle counters that are delivered by suppliers should conform to the requirements of ISO 4402 in terms of its calibration and general performance, and that
- the calibration of the counters be checked on a routine basis.

Important issues regarding particle counting methodologies are highlighted. In recent years a number of excellent publications dealing specifically with these issues have seen the light. It is not the intention of this report to discuss the methodology issues in detail, but rather to point the operator in the right direction. Other issues that are discussed include dealing with on-line and batch particle counting as well as issues relating to accurate particle counting.

Data and Data Handling

Guidelines for the setting up and use of particle counters and also for the reporting of the data from particle counters are suggested. This is required to allow meaningful comparison of the data generated by different counters and operators. This section also shows how the large number of data points generated by particle counters can be reduced to fewer indices so that the results can be employed in a meaningful way by treatment plant operators.

Furthermore, a comprehensive database is required if data is to be sourced from several points. Such a database was developed and employed successfully during the course of this project.

Particle Counting Studies in Potable Water Treatment

Six particle counting studies were undertaken in the course of this study to illustrate the use and benefit of particle counting.

Particle counting offers a mechanism through which raw waters that have to date been classified along conceptual lines, can be sorted into distinctive groups according to the particulate matter suspended in it. Through this study a novel approach in the classification of water has been introduced. This is particularly true for raw waters.

The relationship between particle size and *Cryptosporidium* oocysts and *Giardia* cysts in filtered water was investigated. The study confirmed that *Cryptosporidium* could be detected at 2-5µm and *Giardia* at 3-10 µm using particle counters. It does not distinguish between *Cryptosporidium* and *Giardia* and other particles of the same size. Moreover, the numbers of cyst and oocyst particles are completely overshadowed by the other particles in these size ranges.

A study of the affect of pre-treatment using ozone on the clarity of filtered water was undertaken. In this study it was that particle counting of low turbidity filtered waters was a more sensitive monitoring method than “low-range” turbidity measurement.

The use of particle counting as a feed-back control mechanism in treatment plants was investigated. The particle counter is compared with zeta potential and streaming current detectors. It was found that the filter particle counts did not change much under different coagulant dosages. Some difference was noted on settled water. Due to the delay in response time, particle counting is not considered to be ideal for this purpose.

Flocculation theory differentiates between sweep floc and charge neutralisation and the second is considered to be more effective than the first. The two regimes are controlled by the pH at which the flocculation takes place. South African plants are forced to operate at higher pHs because of the well-buffered nature of the raw waters. In this study the suitability of particle counting is assessed as a means to measure flocculation kinetics. If this were successful, this would allow the use of the Argaman-Kaufman flocculation model in the analysis study of this complex field. Despite repeated attempts and the introduction of numerous precautions and improvements to the experimental methods, this study did not produce the precision and repeatability required by the researchers. It has been concluded that light blockage particle counting is not suitable to the monitoring of transient phenomena such as floc growth and break-up.

Control of filter start-up hydraulics appear to be the only viable alternative for many plants to reduce the impact of the filter ripening curve on final water quality. In this study particle counting is used to monitor ripening curves for various start-up procedures. Particle counting is again shown to be the more sensitive measurement technique when compared with turbidity. It has also emphasised the sensitive nature of filter performance, particularly during the ripening stages of filtration.

Recommendations for further work are included in the conclusion of each of the chapters dealing with the individual studies.

The report concludes with an evaluation of the work and the extent to which the contract objectives were reached. The project team is of the opinion that the benefit of particle counting have been illustrated in this report. Standards for the use of particle counters and particle counting has been suggested. Similar standards and guidelines for the potable water industry have not been published locally before. As far as the minor objectives relating to the application of particle counting in various different studies are concerned, a better understanding of the strengths and limitations of particle counters has been developed.

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- Dr. IM Msibi (Chairman) - Water Research Commission
- Mrs. ND Basson - Sedibeng Water
- Mr. J Geldenhuys - Rand Water
- Mr. JHB Joubert - East Rand Water Care Company
- Mrs. MJF Kruger - Midvaal Water Company
- Mr. F Mafete - North West Water Supply Authority
- Mr. T Nel - Magalies Water
- Mr. D Nozaic - Umgeni Water
- Mr. S Pieterse - Cape Metropolitan Council
- Mr. S Smith - CSIR
- Mr. M Tsotetsi - East Rand Water Care Company
- Dr. LJ du Preez - Water Research Commission (Secretary)

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STANDARDISATION OF THE USE OF PARTICLE COUNTING FOR POTABLE WATER TREATMENT IN SOUTH AFRICA

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LIST OF SYMBOLS

A	Antilog of the power law intercept coefficient α (.)
d_1	Arbitrary diameter of a particle (μm)
d_2	Arbitrary diameter of a particle (μm)
d^-	Lower threshold of a bin (μm)
d^+	Upper threshold of a bin (μm)
d_g	Geometric mean particle size (μm)
G	Root mean square velocity gradient (1/sec)
K_a	Aggregation constant (.)
K_b	Breakup constant (.)
N	Number of particles (.)
N_{i-j}	Incremental count between *i* μm and *j* μm (Counts/mℓ)
Nn	Normalised count (Counts/mℓ)
Nn_{i-j}	Normalised count between *i* μm and *j* μm (Counts/mℓ)
n_0	Number concentration of particles before flocculation period (.)
n_1	Number concentration of particles remaining after flocculation period (.)
r	Correlation coefficient (.)
S_{i-j}	Particle surface area concentration (mm^2/ℓ)
T	Flocculation period (sec)
V_{i-j}	Particle volume concentration ($m\ell/\ell$)
α	Intercept coefficient of the power law (.)
β	Slope coefficient of the power law (.)
Σn_i	Sum of Counts larger than *i* μm (counts/mℓ)

Chapter 1

INTRODUCTION

1.1 Project History

The Rand Afrikaans University (RAU) and Umgeni Water submitted two independent research proposals to the Water Research Commission (WRC) for funding in the period 1999-2000. On closer inspection, the WRC found that although the two proposals were both on particle counting in potable water treatment, they were complementary. On the recommendation of the WRC, the RAU and Umgeni Water had discussions and it was concluded that a combined project would be more meaningful in its content and practical in its execution. The conclusions from these discussions were conveyed to the WRC and a combined Steering Committee was appointed for WRC Project K5/1024 and WRC Project K5/1025. To facilitate further the cooperation, combined Steering Committee Meetings were held.

1.2 Objectives

The objectives of the individual proposals were taken as the initial objectives of the combined projects.

1.2.1 The RAU Objectives (Project K5/1024)

The RAU proposal was initially entitled “Evaluation of Phase Removal Processes at Full-Scale South African Water Treatment Plants in Terms of Particle Size and Number” and was motivated with the following aims :

- *Evaluation of available particle counters, configuring of the counters and standardising reporting procedures.*

  Research involving particle counters had been taking place internationally for a number of years and a mass of information had been gathered. The AWWARF (American Water Works Association Research Foundation) Symposium held in March 1999 attested to this. Despite the international interest not much had been done locally. The project created an ideal opportunity to standardise local testing and reporting procedures from the initial stages of research. This would assist in avoiding future situations where results from separate studies are not comparable due to fundamental differences in testing and reporting procedures. Furthermore, several suppliers are targeting the local market. The equipment that is being offered and the technology being employed varies significantly from supplier to supplier. A study of the equipment and a discussion on the technology would go a long way in assisting future users in selecting the equipment best suited to their application.

- *Establishing baselines in terms of general process performance using particle counters.*

  At the time of the proposal, no commonly accepted baseline for process performance in terms of particle counts and sizes were available. This could be used as a benchmark to evaluate own performance.

- *Study of the effect of flocculation regime (charge neutralisation vs. sweep floc) on final water quality.*

  Despite the more conservative approach of local designers towards loading rates on filters and flotation units, amongst other unit processes, overseas plants appear to be performing better in terms of the treatment objectives applied. The hypothesis was that it could be due to the fact that local plants are forced to operate at one to two pH units higher than normally encountered overseas. At the higher pH levels the flocculation regime changes from charge neutralisation to the sweep floc domain where particle contact is less optimal. Particle counting under local conditions would enable a quantative assessment to be carried out on the effects of different flocculation regimes and provide confirmation of overseas guidelines on flocculation and filter operation, amongst others, or provide alternative guidelines that are more appropriate to local conditions.

- *Evaluating filter performance under various hydraulic control regimes.*

  Particle breakthrough in filter operation is most likely in the first hour of operation. The work intended should lead to guidelines on the operation of filters to minimise the breakthrough of particle sizes in the range where protozoan cysts are expected to occur.

1.2.2 The Umgeni Water Objectives (Project K5/1025)

The Umgeni Water proposal was entitled “Investigation into the Use of Particle Size Analysis for Monitoring and Optimising Plant Performance for the Production of Potable Water” and was motivated with the following aims :

- *To investigate the use of particle size analysis as a control parameter for the optimisation of water quality, and to compare this to the control using zeta potential or streaming current.*

  Streaming current and zeta potential are often used as measures of the de-stabilisation of particles during coagulation in water treatment. These are also used as control parameters to set the chemical dose. The primary objective of water treatment is the removal of particles, and these measurements may be more suitable for use where certain particle size ranges are present. Filtration using sand filters is the final particle removal unit process and should be monitored for efficiency, especially as the filters are backwashed regularly.

- *To investigate the relationship between particle size and Cryptosporidium oocysts and Giardia cysts in filtered waters from water treatment plants.*

  Cryptosporidium has been responsible for a number of outbreaks of disease and water treatment companies are becoming more concerned with the performance of plants for its effective removal from potable water. *Cryptosporidium* oocysts are typically in the 6 - 8 μm size range and should be removed effectively by sand filtration. In order to effectively monitor *Cryptosporidium* in filtered water, particle counts in the relevant size range are necessary.

- *To study the effects of pre-treatment processes such as ozonation on the clarity of filtered water by measuring the particle size after filtration.*

  Pre-treatment using ozonation and other oxidising agent can affect the efficiency of coagulation and, in some cases, reduce the optimum coagulant dose. This may also improve the coagulation and removal of colloidal particles in the size range 1 to 10 µm. Other pre-treatment or post treatment processes such as membrane processes or GAC may improve the quality of the final filtered water.

- *To consider the use of particle size analysis together with Computational Fluid Dynamics for the optimisation of water treatment equipment.*

  This aim arose from collaborative work between Umgeni Water and the Pollution Research Group (University of Natal, Durban) during a project investigating the use of CFD for water treatment plant design. One of Umgeni Water's plants was used as a case study as it was anticipated that a higher demand for water would be required and the capacity of the plant may need to be increased during the December period. This did not materialise, but should the plant require upgrading in the future, the recommended modifications could be made and the use of turbidity and particle counters can be used to verify the modifications proposed by the CFD study.

1.3 Organisation of the Report

Due to the complementary nature of the two projects, it was decided by the Steering Committee that a combined final report would be submitted. This allowed for a re-arrangement of the initial objectives into a more complete document on particle counting. As the objectives also touched on a wide ranging number of issues relating to particle counting, a further comprehensive "user's guide" could be compiled. The document is finally presented in four parts.

1.3.1 Hardware and Methodology

This section contains chapters 2 to 4 and concentrates on the introduction to, as well as the practical issues pertaining to particle counters and particle counting.

1.3.2 Data and Data Handling

Chapters 5 and 6 are included in this section. During the course of the project it was found that particle counting data management required specific attention. This section highlights the important issues and makes suggestions about data management.

1.3.3 Particle Counting Studies in Potable Water Treatment

This section contains the bulk of the original objectives. These objectives are presented as case studies that illustrate how particle counting can be utilised in potable water treatment. This section is made up of chapters 7 to 12.

1.3.4 Conclusions and Project Appraisal

The report concludes with chapter 13 which offers conclusions to the project in general as well as a self appraisal of the objectives achieved.

STANDARDISATION OF THE USE OF PARTICLE COUNTING FOR POTABLE WATER TREATMENT IN SOUTH AFRICA

PART A : HARDWARE AND METHODOLOGY

Chapter 2

INTRODUCTION TO PARTICLE COUNTERS

Particle counting is a technology that is finding increasing application in the potable water treatment field. With it comes several new concepts and a collection of terminology and jargon that are not necessarily familiar to practitioners in the water field. The aim of this chapter is to explain these concepts and to familiarise the practitioners with the rudiments of this technology.

2.1 What is Particle Counting?

A particle counter is an instrument which is permanently installed or portable, and is used to monitor the clarity of fluids. Particle counters have been used successfully in the pharmaceutical, chemical and petroleum industries for a number of years and are now finding application in the drinking water industry.

In the potable water industry, particle counters offer a more detailed insight into the clarity of water samples than turbidity measurements. The counter measures actual particles suspended in the sample and reports the measurements in terms of *number of particles* as well as *size of particles* per measured volume of water. With this information, the operator is better able to assess the nature of the suspended matter and is better equipped to determine process alterations in order to produce a better water quality.

The introduction of the particle counter into the drinking water industry has been accelerated by concerns regarding the presence of protozoa (*Cryptosporidium* oocysts and *Giardia* cysts) in drinking water supplies. Because of the particle counter's ability to distinguish between various size ranges, the general expectation is that it can be used to monitor these pathogens. The counter can also be used to monitor water clarity in general and is also finding application in many other areas of water treatment plant operation and potable water research where turbidity had been used in the past.

To date particle counting has mainly been used to evaluate the performance of filtration processes, and in some full-scale applications, the source water quality is also monitored. In this way a log reduction of particles can be calculated.

The term “particle” is used frequently in this document, and in particle counting practice in general, but is poorly defined. Particles are generally solids in the water which can be removed during water treatment but can also be other fluid drops or gas bubbles that are dispersed in the sample. The solids may be mineral in nature (comprising sand, silt or clay suspensions), organic or humus particles (often resulting from the decomposition of plant tissue), microorganisms (bacteria or protozoan cysts and oocysts -*Giardia* or *Cryptosporidium*) or flocs that result from flocculation which may consist of a combination of the above and precipitated chemicals. By implication, anything that registers a response from a particle counter is considered to be a particle. It is also assumed that any single response from a particle counter, that is being operated within its concentration limit, is caused by a single particle. Particle counters do not discern between the individual particles that make up a floc particle. Particle counters also assume that particles are perfect spheres. The definition for a particle is fundamentally flawed and is easily discredited. This definition is limited by current technology and is considered to be sufficient for the scope of this report.

"Particle sizing", "particle size analysis","particle monitoring" and "particle counting" are terms that are used loosely in industry without much consensus and sometimes to the frustration of the uninitiated. "Particle counting" is the topic of this study and refers to a process where both particle size and number are noted. This exercise is sometimes also referred to as "particle size analysis". "Particle sizing", however, generally refers to an exercise where size distribution is determined and actual counts are not reported. For example, after an analysis with a particle *counter* a result will be returned in the form "120 particles were measured between 5 µm and 10 µm per 1 mℓ" whereas a particle *sizer* would return "30% of particles measured per 1 mℓ were between 5 µm and 10 µm". No indication of actual particle number is given by the particle sizer. The particle counter can be used to indicate water clarity while the particle sizer cannot. "Particle sizing" is sometimes called "particle monitoring". In general, particle counters use laser diodes whereas particle monitors use infrared.

2.2 How Does a Particle Counter Work?

Not all partical counters operate in the same manner. The two most common types are the Electrical Sensing Zone counters (ESZ) and the Optical Particle Counters. These counters generate a signal in different ways when a particle passes its sensing mechanism. The counters are normally calibrated to relate the magnitude of a signal to a specific particle size.

After the analysis of the signals generated by the counter has been completed, the data gathered during the measurement is downloaded to an external computer. The external computer, with the assistance of the appropriate software, then processes the gathered information and reports all the necessary and meaningful results. These results can then be viewed on the computer screen or printed to a report.

For the portable particle counter there is a choice between printing the results immediately (on-site) or to connect the particle counter to a computer at a later time. Results can then be retrieved in the same way as for the permanently installed particle counter.

Suppliers of particle counters also supply specialised software for the manipulation and interpretation of the data generated by the counters as an add-on to the counter. This is generally a significant additional cost. Several sets of software are available for any given counter. The most basic will allow the operator to download the data from the counter in a text or spreadsheet format while the others include graphics capabilities for analysis of the data directly from the counters. This is discussed further in Chapter 6.

2.2.1 The Electrical Sensing Zone (ESZ) Method

Van Gelder et al.(1999) describe this as a method based on current flowing from one electrode to another through a conductive solution (electrolyte) that contains the particles to be measured. One electrode is positioned inside a glass tube that contains a small aperture, and the other is outside that tube in the suspension. The sample is drawn through the aperture, which forms the sensing zone. In an ESZ instrument, a constant current passes between the electrodes. As a particle passes through the sensing zone, the volume of the conductive fluid decreases, causing an increase in the resistance and a voltage pulse is generated. The size of the pulse is proportional to the volume of the particle.

These counters are generally used in research and are not generally employed for particle counting on water treatment plants. The remainder of the report will be devoted to optical particle counters.

2.2.2 The Optical Particle Counters

Two optical techniques, light extinction and light scattering techniques, are used to detect and size particles in water. Both methods employ a laser light source to illuminate single particles that flow in a flow cell which narrows, and an electronic photo detector to measure the interference of the particle on the light path (Sommer, not dated). The photo detector converts the light energy into electric signals that are processed by the counter electronics.

A frequently used term in optical particle counting is "sensor". A *sensor* consists of the laser and a photo detector while a *counter* consists of the sensor as well as the electronics that convert the photo detector measurements to particle size measurements.

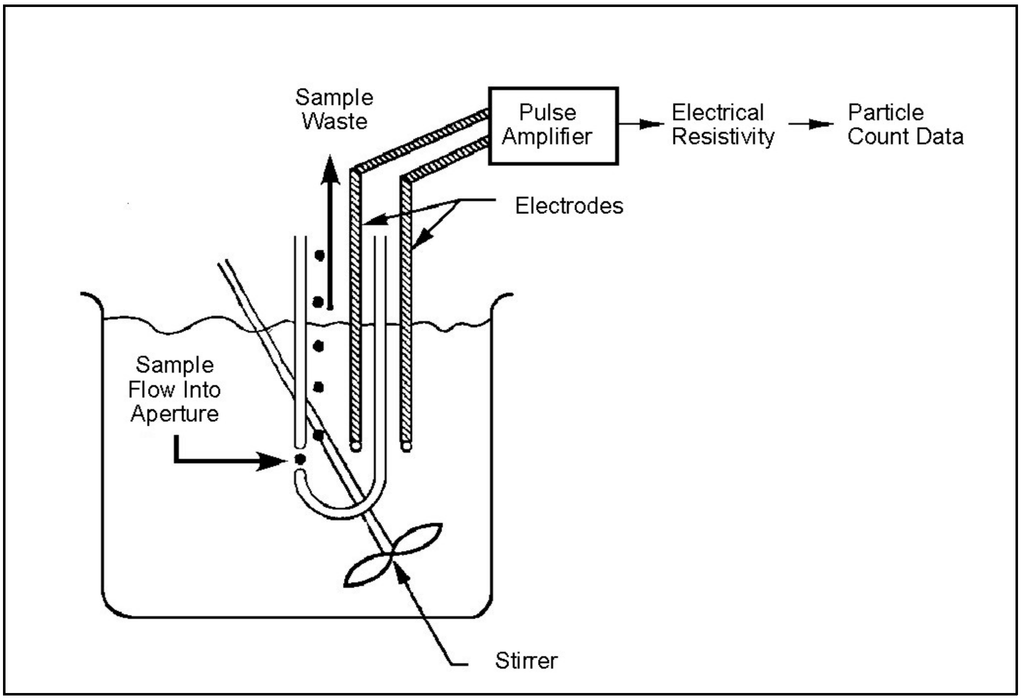

Figure 2.2.1 : Schematic representation of an Electrical Resistivity Particle Count Sensor. Taken from Hargesheimer et al. (1992)

2.2.2.1 Light Scattering Sensors

The light scattering sensors relate the light scattered by particles to the size of those particles. Particle detection based on the light scattering principle is more sensitive than detection based on the light extinction principle even though the amount of light energy collected by the receiving optics of a scattering sensor is normally only a fraction of the total light scattered by a particle. Although the extinction component may be greater than the detected component of the scattered light, particles smaller than 1-2 µm can be detected and sized by light-scattering techniques. Here the photo detector is placed at an angle to the light beam and monitors the light intensity variations in an area that is normally dark. When a particle passes through and light is diverted (scattered) onto to the detector, a signal is registered (Sommer, not dated).

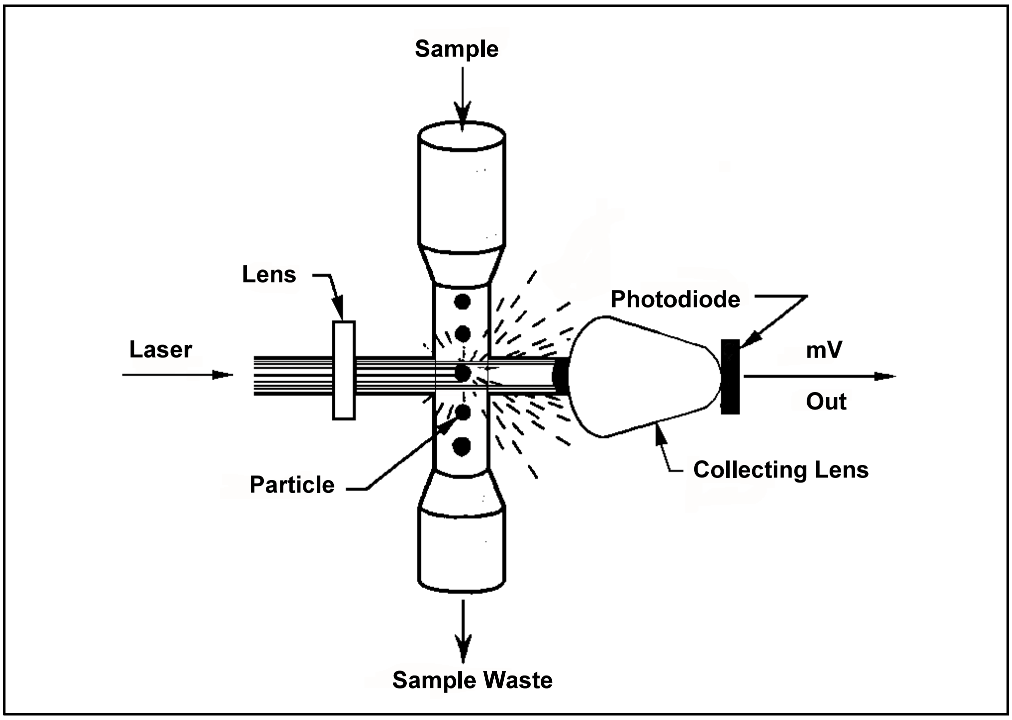

Figure 2.2.2.1 : Schematic representation of a Flow Based Light Scattering Particle Count Sensor. Taken from Hargesheimer *et al.* (1992)

2.2.2.2 Light Extinction Sensors

These sensors measure the loss of light when single particles pass through the gap between a light source and a photo detector. A shadow is cast by the particle and this reduces the voltage output from the photo detector. The size of the shadow and consequently, the size in voltage output drop is directly proportional to the size of the particle passing by.

Light extinction sensors should have a stronger measurement effect than scattering sensors because the particle signal is produced by monitoring all the energy removed from the beam and not just from collecting a small portion by receiving optics with a limited numerical aperture. However, a major portion of the light scattered by a particle is scattered in a forward direction. In the case of an extinction sensor, this is the position of the photo detector. The resulting signal is reduced by the portion of light scattered in the direction of the detector. The signal itself, for the same particle size, is stronger for extinction than for scattering (Sommer, not dated).

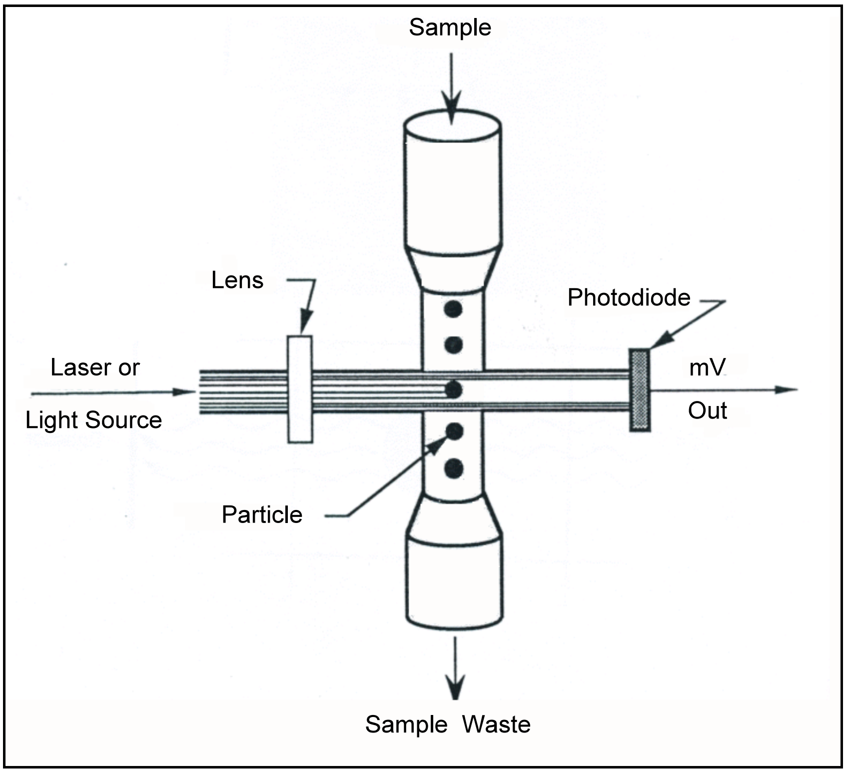

Figure 2.2.2.2 : Schematic representation of a Light Obscuration Particle Count Sensor. Taken from Hargesheimer *et al.* (1992)

From this discussion it should be clear that different particle counters will characterise the same sample differently due to fundamental reasons. ESZ sensors define particles by their effect on conductivity, light scattering sensors by their refractive index and light blockage sensors by their degree of translucence and refractive index. Furthermore, the orientation of the particle passing through the sensor also plays a significant role during measurement. It must therefore be accepted that some variation will be evident when even the same sample is re-measured on the same instrument and more so when different technologies are being compared. These variances are increased when different measurement techniques and procedures are followed; therefore the need for standardisation in the use of particle counters.

2.3 Glossary of Particle Counting Terminology

Not all particle counters are constructed to the same specifications and with the same task in mind. Suppliers and operators use terminology and specifications whereby these features are distinguished. An attempt has been made to list these terms and to describe their meaning. The list also includes other terminology that is used regularly in particle counting practice.

Accuracy:	Due to internal variations, a particle counter does not always measure particle sizes accurately. When a mono-disperse suspension (a suspension in which all the particles are the same size) is analysed, the counter will return a normal distribution in terms of particle sizes. Accuracy refers to the relationship between the actual particle size and the average particle size returned by the counter. Refer to the discussion on calibration paragraph 3.3. Also refer to the discussion on resolution later in this paragraph.
Batch vs. on-line counters:	All particle counters are batch counters in the traditional sense as they all analyse water in discrete batches. Refer to the later discussion on “sample time”. The distinction between the two types is based on the way in which water is delivered to the sensor. On-line counters are suited mainly for analysing water from a permanently installed position. These counters are supplied with water at a fixed rate, achieved by using pumps or constant head devices. The on-line counters are normally installed in a permanent position and require comparatively large water volumes to effect measurements as water is passed through the sensor on a continuous basis. Measurements are taken at pre-determined time intervals. Batch counters are particle counters suited mainly for site work and laboratory work and effect measurement on samples delivered to it on a discrete basis. These counters are usually supplied with vacuum samplers, pressure samplers or syringes. Some portable counters defy classification under this heading as they can be operated in both modes.
Battery power:	Particle counters require electrical energy to operate. Most portable particle counters are marketed with options for battery power, whereas permanently installed counters are generally not. The batteries supplied with the counters will provide sufficient power for 2 to 8 hours of operation.
Channels:	Sometimes referred to as “size bins”. Counters do not report individual measurements, but rather group the counts into convenient groups, ie. 2-3 µm, 3-4 µm, etc. These “groups” are referred to as channels or bins. The more channels a particle counter is equipped with, the more size ranges can then be specified. Some particle

	counters' channels are preset whereas other particle counters' channels can be adjusted according to personal preference. Typically counters have 6, 8,16 or more channels.
Coincidence loss:	This is also sometimes called “count loss”. Coincidence occurs when two or more particles in the flow cell/orifice enter the laser beam simultaneously. The coincidence of particles causes the particle counter to count one particle when two are present, or to count a larger particle when combining the two particles as they overlap. The loss of the other counts is termed “coincidence loss”. Loss values will differ from sensor to sensor, but typical loss values are 10% at 10 000 particles per mℓ. Refer to the next discussion on “concentration limits”. The international standards vary in their requirements on this issue. The US Pharmacopoeia requires 5% while JIS B 9925:1997 and ISO 4402 require 10%.
Concentration limits :	A sensor’s concentration limit is the maximum concentration of particles that a counter can handle before the coincidence loss becomes unacceptable. For a given measurement flow rate, the value of the sensor’s concentration limit is indirectly proportional to the size of the measurement area within the flow cell. The larger the orifice, the more likely that two or more particles will enter the flow cell at the same time. A sensor with a large measurement area will therefore require lower particle concentrations to operate acceptably than sensors with smaller measurement areas. Suppliers quote typical values of approximately 12000 counts per mℓ for flow cell areas of 1 mm^2 and 25000 counts per mℓ for 0,25 mm^2. It is generally accepted that this limit should be halved for best results during normal operation (Van Gelder et al.,1999).
Count matching:	Once a particle counter is constructed and calibrated according to the relevant specification, there is no guarantee that the counter will measure the same as the counter completed immediately prior, or directly after it. The error is greater when comparing instruments from different manufacturers. This is due to minute differences in the manufacture and set-up of the counter. In order to return the same values, mostly due to the insistence of the client, the manufacturer is

	forced to tweak the results of a counter to match that of another counter. This, of course, adjusts the original calibration.
Counting efficiency:	Counting accuracy or counting efficiency refers to the level of accuracy of actual particle counts. The US Pharmacopoeia requires the count to be within 20% of the actual number of particles tested. The JIS B 9925:1997 allows a 30% variation on the count. It requires that the counter measure between 70% and 130% of any standard sample and between 20% and 80% on the standard that is equal to the smallest size that the counter can measure. Refer to the discussion on “half count” later in this section for more clarity on the last statement. The requirements stated by ISO4402:1991 are slightly more complicated as they are based on a sliding scale based on the number of size ranges measured.
Data communication:	Data can be transferred to external computers via a data communication cable. Particle counters use either a RS-485 or RS-232-C serial communication cables and interfaces. These are standard communication technologies and are available freely.
Display:	Most particle counters are equipped with either a LED or a LCD display. This enables the user of the particle counter to view limited results. Some particle counters are equipped with an alarm function that is also displayed via the LED or LCD display.
Environment:	Particle counters are sensitive instruments and can not be operated under extreme environmental conditions. Two general guidelines for particle counters are highlighted: - Ambient temperature should be between 4°C and 40°C - Humidity to be non-condensing. JIS B 9925:1997 requires relative humidity of 20% to 80%
External surface material:	This is normally specified by the supplier but affords no real distinction between counters. All counters are generally sturdy and offer a sufficient level of protection against splashing to the electronic equipment.

False count:	Refer to the discussion on "zero count" later on in this section.
Flow cell:	A flow cell or a flow orifice is a transparent flow passage situated between the laser diode and the photo detector. The sample of water to be analysed by the particle counter is transported in the flow cell. A flow cell can vary in size, but it is usually not larger than 1,2 mm by 1,2 mm, or smaller than 0,25 mm by 0,25 mm. Refer to the later discussion on "Wetted surface materials".
Flow control device:	It is important that water be passed through the particle counter at a pre-determined, accurate flow rate. See the discussion on "Flow rate". The flow control device can be a peristaltic pump or a weir device that ensures that the flow is constant. The operator is still responsible to ensure that the weir is set up so that the flow is correct.
Flow rate:	A particle counter does not measure sample volume directly but calculates a volume based on the duration of a measurement. It is therefore crucial that the rate of flow through the counter is accurate and constant. Portable particle counters are equipped with a pump that either sucks or pumps the water sample through the particle counter at the required rate. Particle counters that do not have this feature are usually provided with constant head devices that reduce or eliminate bubbles and ensure a constant flow rate into the sensors. Flow rates through the flow cell are in the order of 25 to 100 mℓ/min. ISO 4402 allows some variation of the flow rate, but limits this variation to that which will not cause count variations of more than 5% above or below the count associated with the accurate base flow.
Half count:	When a sample containing monodisperse particles is measured, it will measure half of the particles above a certain size and the other half below that size and the distribution of counts will take the shape of a normal distribution. This is due to small variations in the monodisperse sample and in the counter itself. When the average size of the monodisperse sample measured lies exactly on the lowest size threshold setting of the sensor, the counter will discard the counts lower than the minimum setting and should only report half of the count. This is referred to as the half count principle.

Light source:	A laser diode is used by most particle counters as a light source. In the past infrared has been used, but this is being superceded.
Particle size range:	Every particle counter has limitations on the size of particles it can handle and analyse. This is due to background noise, coincidence loss, flow cell size, etc. Therefore, a particle size range is prescribed for every particle counter. This prevents an instrument from being damaged and ensures accurate results. A typical size range in the water industry is 2 µm to 400 µm. Particle size measurements in treated water rarely extend beyond 100 µm.
Resolution:	Refer to the discussion on "Accuracy" earlier in this section. Resolution refers to the extent to which measurements taken on a mono-disperse suspension differ from the average of the measurement. Both the US Pharmacopoeia and ISO 4402 allow a resolution of 10%, i.e. the standard deviation of the measurement should fall within 10% of the average size of the measurement.
Sample temperature:	The materials of construction limit the temperatures that a counter can handle. Also a minimum temperature of 4 °C is normally quoted to prevent ice crystals from forming in the counter.
Sample time:	This indicates the time over which a counter can analyse samples and can normally be set to any value. After this time the counter stops, processes the data and reports a count before it starts analysing the next sample. This exercise effectively averages the counts over the sample time. If a particularly long sample time is selected and the water quality changes during that interval, the particle counter will return the average count over that period.
Sampling mode:	This is normally associated with portable counters and refers to the way in which the sample is supplied to the sensor. There are two options available, namely vacuum sampling (a syringe actuated by a precision stepper motor draws a sample) and pressure sampling (with a self-contained pressure chamber, a sample is delivered to the sensor). Depending on the tests that will be done, these methods

	might have advantages and disadvantages. One method is not preferred over the other.
Sensing method:	Two methods for sensing are available for optical particle counters, light extinction and light scattering. For detail on these methods, refer to paragraph 2.2: "How Does A Particle Counter Work? ".
Signal-to-noise-ratio:	This gives an indication of the amount of background noise and how it relates to the signal generated by the smallest particle measurable by the counter. Typical values for this characteristic are in the order of 3:1 at 2 µm, meaning that the signal generated by a 2 µm particle will be three times the size of a particle generated by background noise. Specifications relating to the manufacture and calibration of particle counters require a ratio of at least 1.5:1 at the lowest measurable size (ISO/CD 4402).
Software:	Most particle counters have their own unique software packages used to: - control the particle counter, - store and manipulate data, and - present the data.
Threshold settings :	These are the predetermined voltage settings by which a particle counter determines to which channel or bin a specific signal or count is to be placed.
Weight:	The mass of a particle counter varies between 2 kg and 45 kg. For a permanently installed particle counter, mass should not be a critical characteristic, but for a portable particle counter, mass might become a consideration.
Wetted surface materials:	Particle counters are equipped with a wide range of wetted surface materials. This refers to the material that comes into contact with the fluid being analysed. The most important of these materials is the material making up the “window” through which the laser passes to measure particles. Several alternative “window”-types are available to users of particle counters in general, but only two are used in the

	drinking water industry. The first is quartz and this is used in almost all of the counters installed in water treatment plants. Sapphire is sometimes offered at additional cost by suppliers, as it is reputed to be more scratch resistant.
Zero count:	A particle counter does not normally return a zero value when it does not detect any particles. This is due to noise in the counter electronics. The JIS standard requires that particle counters are tested for this by doing a count when no fluid is passed through the counter or when a sample completely free of particles is used. The zero count is taken as the measurement returned by the counter under these conditions. As it is difficult to obtain water perfectly free of particles, the first method is normally employed. This is risky and should not be done over extended periods as some counters rely on the flow of water to cool pumps. ISO 4402 and JIS require that zero counts are lower than 1 per minute.

2.4 Particle Counter Availability Locally and Internationally

Particle counters have only recently been accepted as valuable tools in the potable water industry, but suppliers have already earmarked this industry as one that offers significant growth prospects. In South Africa, this has also been the case and several of the major suppliers are represented. The majority of these suppliers have been active in the local pharmaceutical and petroleum industries and found it fairly easy to include particle counters for the water industry in their existing product range. Great strides are still being made in the technology and the suppliers are constantly in a state of flux. The industry is a competitive one and mergers and take-overs amongst competitors, the emergence of new suppliers, and changes in representation are frequent occurrences.

A list of suppliers and agents as at the end of 2000 is presented in Table 2.4.

The compliance of the particular products presented below, to international standards and norms, must be checked with the individual suppliers at the time of purchase.

Table 2.4: Suppliers of Particle Counters at the End of 2000

Manufacturer	Local Agent	Product Range
Company: HACH Company Address: PO Box 389 Loveland Colorado 80539-0389 USA Tel: 800-227-4224 Fax: 970-669-2932 E-mail: orders@hach.com Website: http://www.hach.com	Local agent: Pelichem (Pty) Ltd Address: Stand 101 Schooner Street Lazer Park Honeydew Tel (011) 794-5902 Fax: (011) 795-1220 E-mail: joostej@colloids.co.za	Hach WGS 267 Hach Model PCX Hach 2200 Series Sensors
Company: Pacific Scientific Co. HIAC/ROYCO Division Address: 11801 Tech Road Silver Spring MD 20904-1909 USA Tel: (301) 680-7000 (800) 638-2790 Fax: (301) 622-0714	Local agent: Premier Technologies Address: Unit 10 & 11 79-84 Kyalami Crescent Kyalami Boulevard Kyalami Business Park Midrand 1685 Tel (011) 466-1410 Fax: (011) 466-1315 E-mail: manub@setpoint-premier.co.za	HIAC/ROYCO System 8011 HIAC/ROYCO System 9103 Note: This product is popular in the pharmaceutical and petroleum industries
Company: Particle Measuring Systems, Inc. Address: 5475 Airport Blvd. Boulder CO 80301 Tel: (303) 443-7100 Fax: (303) 449-6870 Website: www.pmeasuring.com	Local agent: Micron Scientific Address: 175 Princes Avenue Benoni Tel: (011) 422-4303 Fax: (011) 845-2923 E-mail: micscien@global.co.za	Model APSS-200 Note : This product is used more in the pharmaceutical industry
Company: CHEMTRAC Systems, Inc. Address: 6991 Peachtree Industrial Blvd., Bldg. 600, Norcross GA 30092 Tel: 770-449-6233 Fax: 770-447-0889 E-mail: chemtrac@mindspring.com Website: www.chemtrac.com	Local agent: BHT Water Treatment Address: Triton House Brian Ave Chloorkop Midrand Tel: 011 929 5800 Fax: 011 976 3170 E-mail: Pleopold@sued-chemie.co.za	Model PC 2400 D (Particle Counting System) Model PC 2400 PS (Particle Counter, Portable Sampler)

Table 2.4: Continued

Company: RION Company, LTD. Address: 20-41 Higashimotomachi 3-chome, Kokubunji Tokyo 185-8533 Japan Tel: +81-42-359-7878 Fax: +81-42-359-7441 Website: http://www.rion.co.jp/	Local agent: Optima (Natal) Pty Ltd. Address: Unit 3, 4 Le Mans Road Westmead Natal 3608 Tel: (031) 700-6822 Fax: (031) 700-6823 E-mail: optimahydraulics@new.co. za	KL-20A (Particle Counter) KL-22 (Particle Counter) KL-26 (Particle Counter) KL-27 (Particle Counter) KL-28B (In-line Particle Sensor) KL-28BF (In-line Particle Sensor)
Company: PAMAS Address: Dieselstrasse 10 D-71277 Rutesheim Tel: +49 7152 9963-0 Fax: +49 7152 548 62 E-mail: info@pamas.de	Local agent: Micron Scientific Address: 175 Princes Avenue Benoni Tel: (011) 422-4303 Fax: (011) 845-2923 E-mail: micscien@global.co.za	3108 FM (Mobile System for Particle Counting Filter test and Cleaning Control) 3316 FM (Water System for On-Line and Laboratory Applications)
Company: Fluid Systems Partners GmbH (ARTI) Address: Postfach 1160 D-76699 Kraichtal Germany Tel.: +49-7250-76-520 Fax: +49-7250-76-575	Local agent: Optima (Natal) Pty Ltd. Address: Unit 3, 4 Le Mans Road Westmead Natal 3608 Tel: (031) 700-6822 Fax: (031) 700-6823 E-mail:optimahydraulics@new.co.za	WPC (Water Particle Counter)

Chapter 3

STANDARDISATION OF PARTICLE COUNTER MANUFACTURE AND CALIBRATION

The previous chapter briefly mentioned some of the standards that have been published to ensure that particle counters are manufactured and calibrated according to specified procedures. These standards relate to other industries as no standards have been published for the potable water industry. This chapter briefly discusses the published standards and concludes with recommendations for the application of standards in the South African context.

3.1 Introduction

Not all particle counters measure correctly and accurately. Several studies show that tests repeated with different counters yield varying results (Van Gelder et al., 1999). This is due to variations in the way that counters are constructed and also in the way that they are calibrated. In order to address these issues, several standards have been published. These standards relate to the manufacturing of the particle counters, their calibration as well the settings to be used during operation.

Although the standards that are currently available were drawn up for other industries, they do contain information that might be applicable to the potable water industry. To date, a standard has not been accepted as the universal norm in the potable water industry. This statement is valid for the manufacture and calibration of, as well as measurement with particle counters. The direct result of this is that data generated by different counters cannot be compared with confidence due to variations in the counters themselves, the set-up of the counters and also the experimental procedure employed by the operator. This chapter discusses these standards and evaluates their applicability to the potable water industry.

3.2 Manufacturing Standards

Two standards are currently referred to in this regard. The first is the Japanese Standard “JIS B 9921:1997 - Light Scattering Automatic Particle Counter[s]” and the second is a German standard. These standards have been published in an attempt to standardise the manufacture and performance of particle counters for use in the petroleum industry and general air particle counting. It can, however, be assumed that the issues that are concerning these industries will also be of concern to the potable water industry. Typical topics that are addressed and norms that are set in the standards are:

- Accuracy
  Refer to the discussion on this topic in paragraph 2.3. There is no variation allowed in the standards for this item as the standards accept that the counters will be re-adjusted if they are not accurate and that the re-adjustment will eliminate all variation. This presents a problem as no guidelines are given that indicate which variations are excessive. Refer to the discussion on calibration in paragraph 3.3.
- Counting efficiency/Counting accuracy
  Refer to the earlier discussion on counting efficiency in paragraph 2.3.
- Resolution
  Refer to the earlier discussion on resolution in paragraph 2.3.
- Signal-to-noise ratios
  Refer to the earlier discussion on signal-to-noise ratios in paragraph 2.3.

In the absence of standards relating specifically to the potable water industry, it is recommended that particle counters purchased for the water industry comply with at least one of the above-mentioned standards. This will offer the purchaser some assurance of quality.

3.3 Calibration of Particle Counters

Several standards were published that specify the way in which particle counters should be calibrated. Again, these standards have their origin mainly in the petroleum industries. These standards include :

- ANSI/(NFPA)/T2.96R1-1990 Hydraulic Fluid Power - Liquid Automatic Particle Counters - Method for Calibration.
- ASTM F658-87 Standard Practice for Defining Size Calibration, Resolution, and Counting Accuracy of Liquid-borne Particle Counter, Using Near-Monodisperse Spherical Particulate Material.
- BS3406:Part 7:1988 British Standard Methods for Determination of Particle Size Distribution. Part 7. Recommendations for Single Particle Light Interaction Methods.
- ISO 4402:1991 Hydraulic Fluid Power - Calibration of Automatic Count Instruments or Particles suspended in Liquids - Method using Classified AC Fine Test Dust Contaminant.
- JIS B 9925-1997 Light Scattering Automatic Particle Counter.

The standards are continuously under review and amendments are published regularly. ISO 4402 is currently also circulating in two other incarnations, which are:

- ISO 4402 Draft International Standard ISO/DIS 4402 Hydraulic Fluid Power - Calibration of Liquid Automatic Particle Counters (Revision of Second Edition (ISO 4402:1991), and
- ISO/FDI 11171:1999(E) Hydraulic Fluid Power - Calibration of Automatic Particle Counters for Liquids.

3.3.1 Calibration Standards

One of the main differences amongst these standards, also the ISO 4402 range, is the material used in the calibration of the counters. There are two substances in general use. The first is Air Cleaner Fine Test Dust (ACFTD) and the second is a synthetic latex sphere. These methods have both advantages and disadvantages and neither one is considered to be ideal. The following table is taken from Van Gelder et al. (1999). The table lists the desired characteristics of a concentration standard and then compares the two available standards thereto.

Table 3.3.1: Desired Characteristics of Concentration Standards (Van Gelder et al., 1999)

Criterion	Test Dust	Latex Spheres
Know particle size and count distribution	Relatively	Yes, with some uncertainty
Stability	Possible	Yes
Reasonable size	Yes	Yes
Reasonable Concentration	Possible	Possible
Characteristics independently verifiable	No	Yes
Single characteristic dimension for each particle	No	Yes

Some of the more serious problems associated with the ACFTD is that the size distribution is only known on a relative basis. This, associated with the difficulty of making up the standard, makes the ACFTD less attractive as a concentration standard. Furthermore, the production of the ACFTD seems to have an uncertain future as reports of its discontinuation abound. The use of the latex spheres is also questionable mainly due to the fact that the spheres do not resemble the particles that are normally encountered in water. They differ in shape and also in terms of the diffraction properties. The counter's response to the latex spheres is therefore different to that of natural particles. A further point of concern relating to the use of latex spheres is the fact that there appears to be some variation in the standards received from the supplier of the standards (Van Gelder et al., 1999).

It would appear that the latex sphere standard should be accepted as the basis for a local standard as most specifications seem to be making the transition from ACFTD to latex spheres.

3.3.2 Calibration Procedures

The procedure whereby the counter is calibrated, is a fairly intricate, one although the principle is simple. A sample containing known particles is passed through the counter and the sensor's voltage response thereto is noted. After a number of particle sizes have been tested, a calibration curve can be drawn and is programmed into the counter. The voltage response generated by an unknown particle will then indicate the size of that particle. The procedure through which this is achieved, is more complex than the brief description above would indicate and it is best left to an expert. It is up to the client to indicate the standard to which the counter should be calibrated. It is suggested that ISO 4402 be used as the standard in this regard as it has been revised recently.

3.3.3 Other Procedures by the Supplier

ISO 4402 requires that, after a new water particle counter has been purchased and received, or repairs have been made to a particle counter, or when adjustments have been made to either the water particle counter or the sensor, the following procedures have to be conducted:

- Preliminary Instrument Check.
  This test is done to ensure that an instrument will deliver the same results repeatedly.
- Coincidence Error Procedure
  The coincidence error limit determined should be regarded as a benchmark indicator, which is useful for routine analyses.
- Flow Rate Limits Determination
  Most particle counters do not measure flow or volume directly, its calculation is based on the duration of flow. If the flow rate is incorrect, the flow rate or volume calculation as well as the reporting of the particle concentration will be incorrect. In extreme cases with high flow rates, contaminant particles may not be present in the sensing volume long enough for the electrical signal to fully develop. Also, the time interval separating successive particles may be so small that the electronics are unable to distinguish them as individual particles. It is important that a particle counter operates at the correct flow rate (ISO 4402:1991).

- Resolution Determination
  Since resolution varies as a function of particle size, every individual water particle counter's resolution should be determined.
- Sensor Performance Specification
  This procedure ensures that the sensor performs the same for three identical samples.
- Sizing Calibration Procedure
  The sizing calibration of water particle counters is very important. If water particle counters do not measure particles of the same size in the same way then no comparison can be made amongst different water particle counters.

3.3.4 Procedures by the Operator

Standards require that the operator of a counter check the instrument to verify if it is still within specification every three to six months. The accuracy, resolution and the concentration measurement should be checked. This is achieved by passing particle-free water spiked with a latex sphere standard through the counter and evaluating the result. If the operator finds that any of the parameters obtained from the test deviates from the norm by more than what the relevant specifications allow, then re-calibration is required. This exercise is a costly one as the latex standards are fairly expensive. The standards can be purchased in various unit sizes and concentrations. The cost varies between R 1 000 and R 2 800 depending on the concentration of the standard. Some standards are NIST (National Institute of Standards and Technology) traceable and are supplied with calibration certificates. These are the only standards that are allowable under ISO 4402 for calibrating counters. If non-traceable standards are used for calibration checks, the cost of the exercise can be reduced by a small margin. This is however not advisable. A standard of 100 mℓ at 2 000 particles per mℓ would be sufficient for four to five calibration checks. At least two, preferably three, size standards should be tested during a calibration check. The ANSI/(NFPA) T2.9.6 R1-1990 standard requires that at least four different sized latex batches be used. The standards are also subject to a limited shelf life and cannot be kept indefinitely.

Although the calibration checking procedures appears simple, it must be stressed that it can be very difficult to return an acceptable result. This will require several attempts to achieve accurate results for the first time. The procedure is very susceptible to contamination from a number of sources and experimental variability. The contamination can be from the water used in the test, the glassware and equipment used as well as from the particle standard itself if it is prepared incorrectly. At best, a fair indication of the accuracy and resolution of the counter can be obtained. It is almost impossible to return good results on concentration measurement with values ranging with ±20% of what was expected being considered very successful. The procedure is elaborated on in Chapter 4.

3.3.5 Count Matching of Particle Counters

It is often a requirement from the users of particle counters that when a sample is measured by two separate instruments the same result should be obtained. Although this should occur when two instruments have been manufactured and calibrated according to the specifications, it seldom occurs in practice. Some manufacturers claim that they can adjust the calibration of an instrument to ensure that it measures the same as another instrument to satisfy the customers' requirements.

This is not advisable as this deviates from the procedures of the specifications ISO 4402 and JIS B 9925.

3.4 RECOMMENDATIONS

In conclusion, it is recommended that:

- particle counters are only acquired if they conform to JIS B 9921:1997 in terms of its manufacture,
- particle counters that are delivered by suppliers conform to the requirements of ISO 4402 in terms of its calibration and general performance,
- the calibration of the counters be checked routinely by the operator, and that
- the practice of count matching be discouraged.

Chapter 4

GUIDELINES FOR PARTICLE COUNTING METHODOLOGY

This chapter summarises the most important issues relating to particle counting methodology. In recent years a number of excellent publications dealing specifically with these issues have been published. It is not the intention of this chapter to repeat everything in detail, but rather to highlight the important issues and to steer the operator in the right direction. This chapter briefly discusses some considerations when dealing with on-line and batch particle counting as well as some other matters relating to accurate particle counting.

4.1 Introduction

Effective and accurate particle counting requires meticulous procedure and attention to detail. Not only is it necessary to deal with the particle counter's own instability, but attention must also be given to outside interferences that might lead to contamination of the sample.

4.2 Batch vs. On-line Counting

Several factors should be taken into account when a decision between batch and on-line counting has to be made. Both modes of measurement have advantages and disadvantages. Tables 4.2(a), 4.2(b), 4.2(c) and 4.2(d) are taken from Hargesheimer et al. (1992). These tables describe in detail the issues that relate to this topic. In short, the batch counter is a more flexible tool, but it also requires significantly more operator input whereas the on-line particle counter requires less input, but is not very flexible in its application.

Some manufacturers are now supplying portable counters that can operate as both batch and on-line counters. These counters are normally provided with a pump or mechanism that controls the flow rate through the sensor which means that samples can be drawn from a number of points where normal on-line counters monitor only one sample point. Portable counters can also be provided with battery power so that they are truly portable. Data can be retrieved from these counters through printouts or through data downloads to computer.

It is therefore suggested that on-line counters be considered for permanent installation and portable or batch counters be considered for general analytical work and surveys or investigations.

Table 4.2(a) : Sampling Considerations for Discrete and On-line Particle Counting

(Hargesheimer et al., 1992)

Discrete (Batch) Particle Counting	On-line Particle Counting
S Actual samples are collected	S Samples are not collected
S Samples can be collected from virtually any location	S Sampling sites are limited to takeoffs with sufficient pressure. New locations require additional equipment or relocation of existing equipment
S Sampling frequency is limited by the larger sample volume requirements	S Intensive sampling frequency is possible
S Sample containers require labour-intensive preparation	S Sample containers are not required
S Sample contamination may originate from transmission-lines between process streams and the sampling port, and from the containers and other equipment coming in contact with the sample.	S Contamination may originate from transmission-lines between the process stream and the sensor.
S Distance of sampling site from source must be minimised.	S Distance of sampling site from source must be minimised.
S Devices upstream of sampling port must be avoided.	S Devices upstream of sampling port must be avoided.
S A batch sampler is required, a flow controller is not required.	S A flow controlling device is required, a batch sampler is not required.

Table 4.2(b) : Analytical Considerations for Discrete and On-line Particle Counting

(Hargesheimer et al., 1992)

Discrete (Batch) Particle Counting	On-line Particle Counting
S A variety of sensor types can be utilised.	S A variety of sensor types can be used.
S Sample analysis occurs some time after sample collection.	S Real-time monitoring is achieved.
S Sample storage may change particle size distribution.	S Sample storage is not possible.
S Samples may be manipulated prior to analysis (e.g., dilution) and during analysis (e.g., stirring)	S Manipulation of samples are impossible
S A batch sampler is used to deliver the sample.	S Analyses of samples occur in situ on a sample stream continuously flowing through the sensor.
S Sample volume must be selected suitable o the end use of the data.	S Sample interval and delay time must be selected suitable to end use of the data.
S Samples can be archived for later analysis.	S Sample archiving is not possible.
S A wide range of sample types can be analysed using the same sensor; high concentrations can be analysed following dilution.	S A particle count sensor appropriate for a specific application must be selected; high concentration samples will generate unusable data.
S Replicate analysis of samples can be performed.	S Replicate sample analysis is not possible.
S Each sample is processed by an analyst.	S Analysis is highly automated.
S Analysis at high sampling frequency is labour intensive and time consuming.	S High-sampling-frequency analysis can be done with little or no analyst time required.
S Flow rate fluctuations during analysis can invalidate results.	S Flow rate fluctuations during analysis can invalidate results.

Table 4.2(c) : Data Handling for Discrete and On-line Particle Counting

(Hargesheimer et al., 1992)

Discrete (Batch) Particle Counting	On-line Particle Counting
S Size channels must be selected based on the end use of the data. S Differential number of particles, cumulative number of particles, the particle size distribution presented as a percentage of the total cumulative particle counts, and the power law slope coefficient can be calculated. S There is no significant difference between results obtained by discrete and on-line monitoring when sampling frequencies are identical. S Computer assisted data collection is helpful.	S Size channels must be selected based on the end use of the data. S Differential number of particles, cumulative number of particles, the particle size distribution presented as a percentage of the total cumulative particle counts, and the power law slope coefficient can be calculated. S There is no significant difference between results obtained by discrete and on-line monitoring when sampling frequencies are identical. S Computer assisted data collection is recommended.

Table 4.2(d) : Quality Assurance Protocols for Discrete and On-line Particle Counting

(Hargesheimer et al., 1992)

Discrete (Batch) Particle Counting	On-line Particle Counting
Quality Assurance	
S Quality assurance is critical in the generation of valid data. S System configuration remains unchanged for quality control. S Inter-calibration of particle counters is possible. S Clean liquid and calibration spheres are required. S Sample flow rate must be checked on system start-up and on every analysis. S Routine checks for electrical noise must be performed.	S Quality assurance is critical in the generation of valid data. S System configuration must be altered to deliver quality control samples to the sensor. S Inter-calibration of particle counters is possible. S Clean liquid and calibration spheres are required. S Sample flow rate must be checked on system start-up and on every analysis. Sample flow must be controlled and an automated record of flow maintained. S Routine checks for electrical noise must be performed.
Maintenance	
S Biofilm and chemical film can build up on instrument parts. Chemical and/or physical cleaning are required.	S Biofilm and chemical film can build up on instrument parts. Chemical and/or physical cleaning are required.

4.3 On-line Counting

4.3.1 Setting Up of On-line Instruments

On-line instruments are supplied typically as wall mountable instruments in IP65 (or higher) splash-proof electrical enclosures. The light source and electronics for manipulating the signals received from the particle counting sensor are all housed in the enclosure. Connection is required to a computer system via a RS485 networking cable, and, in most cases, up to 16 particle counters can be connected in series to the same computer.

Provision is made for the instrument to receive inputs from other instrumentation either as 0-10V or 4-20mA signals. These are also communicated to the computer and displayed or stored in the database. Similarly provision is made to monitor digital alarm signals which could, for example, indicate low flow rate or other condition on the plant.

The calibration of the instruments is normally performed by the suppliers before shipment and cannot be changed on site. The electronic set-up of the instrument to monitor specific sizes of particles is performed from the computer. The correct network address is required where more than one instrument is connected to the same computer. The following aspects are addressed during installation :

- Sizes to monitor

  A range of particle sizes should be chosen for all the available channels and must include a >2 µm. Where 8 channels are used the following sizes are recommended : >2 µm; >3 µm; >5 µm; >7 µm; >10 µm; >15 µm; >25 µm and >50 µm. (as discussed in Chapter 3)

- Flow rate through the sensor

  The instruments commonly perform a volumetric determination and require an accurate and consistent flow rate through the sensor. When operating under low positive head conditions this is often difficult to obtain. The actual flow rate through the sensor should be measured and must be entered as a parameter in the software for accurate reporting of the data. Most instruments require a flow of exactly 100 mℓ/min, whereas with other instruments allowance is made for the measurement of the flow rate and entering this as a specific parameter.

- Counting of particles

  This is normally performed for a fixed duration. Thereafter the calculation of particles per mℓ is performed based on the actual flow rate through the sensor.

4.3.1.1 Connection of an On-line Particle Counter for Continuous Plant Monitoring

The instrument should be connected as described in the detailed operating manuals supplied with the instruments. This normally comprises a sampling pump or connection to a pressurised line. Particular attention should be paid to aspects of sampling described in Paragraph 4.5 with regard to sampling and de-gassing of samples.

Flow control devices are supplied by most companies which provide a constant head overflow weir where degassing can occur. The sample is then drawn from the flow control / constant head apparatus and allowed to gravitate through the sensor.

The height of the constant head can be adjusted to regulate the flow rate through the sensor. The flow is maintained without significant aeration or turbulence to provide the most accurate determination by the instrument.

4.3.1.2 Connection of an On-line Particle Counter for Batch Sampling

A batch sample can also be measured by the instrument by installing the instrument with other apparatus to be able to feed the sample to the instrument. Figure 4.3.1.2 shows two such configurations which can be used.

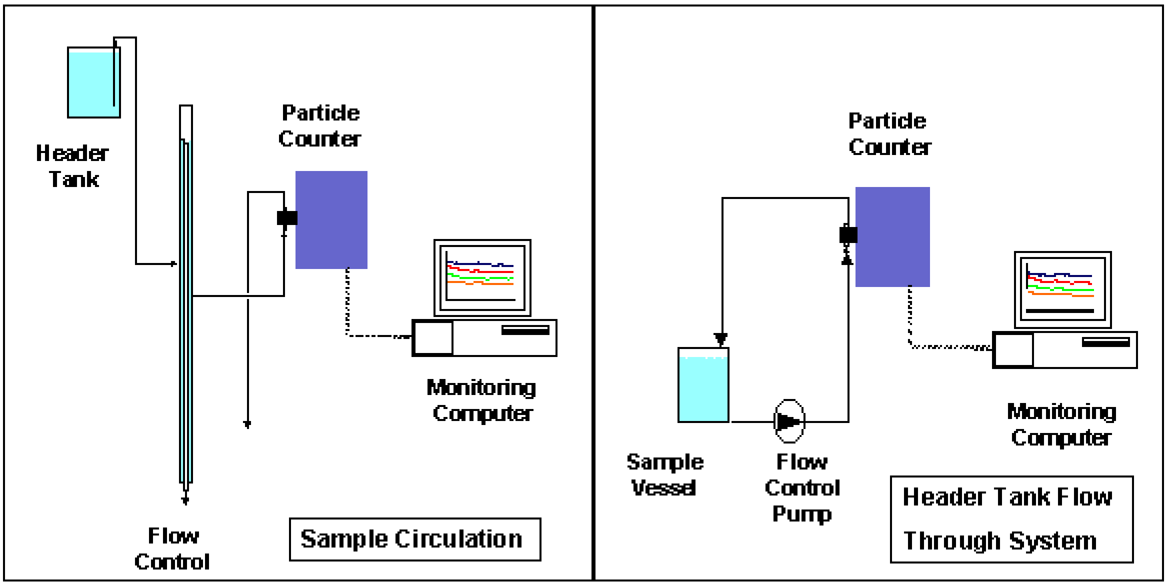

Figure 4.3.1.2 : Experimental Setup for Batch Sampling Using an On-line Particle Counter

- Sample Circulation

  A small sample volume can be circulated through the sensor at a specific rate and the particle count will be determined. This should be performed for a period long enough for the particle count to become stable. Contamination of samples from one to the next may become a problem and more than one sample may need to be measured before a totally representative measurement is made.

- Header tank flow through system

  A header tank can be installed whereby a large sample is allowed to flow through the particle counter as if the instrument is installed on-line. In this case the instrument is connected to the constant head flow regulator. Again the particle counts should be allowed to stabilise before the final acceptance of the measurement as representative of the particles in the sample. This technique can result however in a distorted measurement where particles settle in the header tank.

Both these techniques are extremely useful for measuring samples where the particle counts exceed the concentration limit of the instrument and manual dilution is required. The header tank system allows for larger volumes of samples and will reduce the effect of dust contamination and contamination from previous samples in the system. These techniques can also be used to check the calibration of the instrument using latex standards for example.

4.3.1.3 Maintenance of the On-line Instrument

The on-line particle counters are exposed to the same water sample for extended periods. It is normally advised that the instrument sensor should be cleaned at least once per month. Biofouling is common and deposits of iron and manganese can cause a reduction in the efficiency of the sensor by allowing less light penetration.

The instruments have a built-in system of measuring the light intensity and increasing the power to the light source, thereby compensating for a gradual deterioration of the cleanliness of the sensor. This is monitored and should the instrument detect a adverse condition it will normally stop functioning and indicate an alarm condition on the monitoring computer.

The cleaning of the sensor is performed using a detergent cleaning solution, and under severe fouling, a dilute acid may be required. A small wire brush is provided which can be inserted into the flow chamber after removing the flow tubing and a simple brushing will normally remove most accumulated solids and biofilm. In the operation of the instruments at Umgeni Water the cleaning of the sensors has been required occasionally, although on one occasion the sensor could not be properly cleaned. The instrument had to be returned to the manufacturer.

Once cleaning is complete, the sensor should be flushed to remove any particles which have been dislodged during the cleaning process. The design of the instrument requires that approximately 100 mℓ per minute is allowed to flow through the instrument to allow the particles to move past the sensor in laminar flow. It may be necessary to increase the flow through the sensor at least five fold to create turbulent conditions, thereby improving the flushing after cleaning. In order to clean the sensor properly it may become necessary to remove the instrument and install it in a "cleaning station" using the batch circulation set-up described in 4.3.1.2. In this way the sensor can be cleaned, ultra-pure water can be used to flush the sensor and the calibration of the instrument can be checked before putting it back into service.

4.3.2 Confirmation of Calibration of the On-line Instruments

NIST traceable particle size standards are manufactured by Duke Scientific and used for calibration of particle counters. These standards can also be purchased separately in 100 mℓ volumes of known particle size and number (2 000 per mℓ). Two methods can be used for checking calibration.

S Bench Test

Using a set-up similar to the sample circulation for batch sampling, a standard of known concentration can be circulated through the sensor and it will be measured directly.

- Standard Injection

This entails the injection of a known volume of standard into the water flowing through the particle counter and measuring the increase in particle numbers over a period of time. The particle count in the background water is then subtracted from the measured count in each of the size bins to determine the particle count of the standard that was injected.

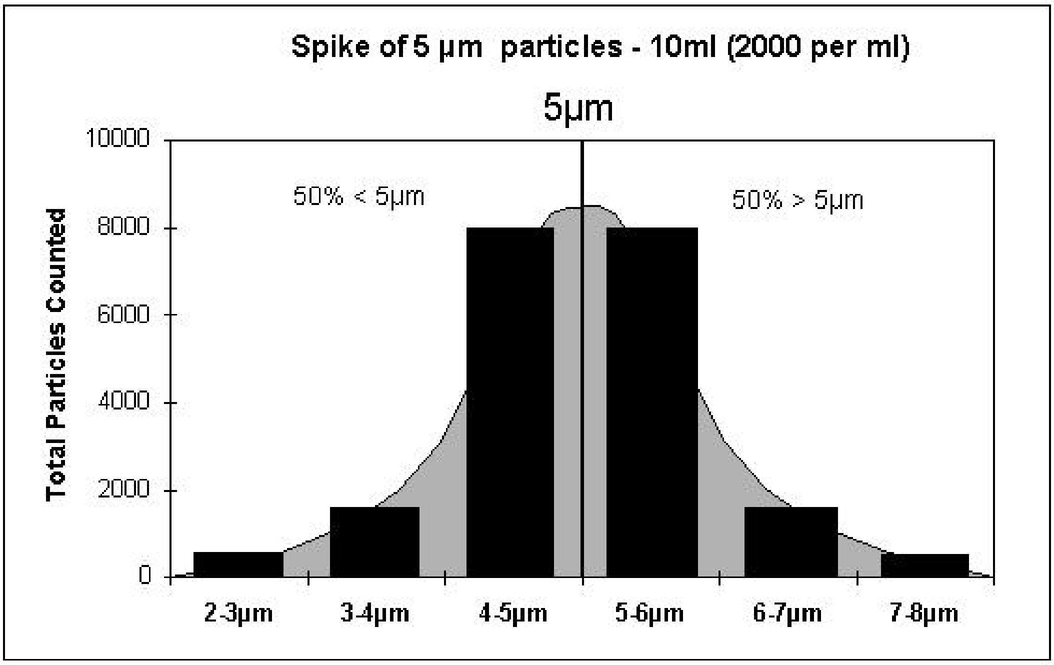

Figure 4.3.2 : Example of Particle Counts after Spiking with 10 mℓ of 5 µm Standards at a Concentration of 2 000 Particles per mℓ.

It is suggested that these tests should be performed with more than one particle size standard. The data obtained can be plotted in the form of a bar graph as in Figure 4.3.2 and should show an even distribution of particles on either side of the size of the standard used.

4.4 Batch and Portable Counting

4.4.1 Setting Up of Batch and Portable Instruments

In the general case, samples are brought to batch instruments. The instruments are therefor normally permanently set-up in a laboratory. The basic requirements for the laboratory include that:

- it is kept clean and free of dust that can contaminate samples and

S a source of particle-free water is available for dilutions (refer to paragraph 4.5.2).

Most batch particle counters have adjustable thresholds and these should be adjusted to reflect the particle ranges that are to be investigated. The flow through the sensor is normally automatically, controlled but this should be verified occasionally.

Because a variety of different samples are passed though batch and portable counters, it is essential that the operator ensures that the count is stable. This is achieved by testing volumes from the same sample until the same count is returned time after time. This can mean that 10 or more samples have to be tested before the count is stable and this is arbitrarily defined as a variation of less than 2% between 3 or 4 successive counts. This definition can be altered according to the inherent stability of the counter and the particle concentration of the sample being tested.

It is sometimes tempting to recycle samples to test again when a counter has not stabilised yet, especially when only a small sample volume is available. Previous samples pollute the sample during the initial stages of testing and floc could be broken up during the first measurement. This practice is not recommended. All samples that have passed through the particle counter must be discarded.

Batch and portable counters are normally used intermittently and hours, days and even weeks can pass between the measurement of samples. In order to avoid the encrustation of solids in the sensor and in the particle counter piping, it is recommended that the particle counter is rinsed for several minutes with particle-free water before and after its use. This will prevent fouling of the sensor as well as blockages in the piping and will ensure that when using the particle counter after a long period that the tubing and sensor are completely wet and that entrained air bubbles have been evacuated.

Suppliers will also provide guidelines for the cleaning and maintenance of the counters. These should be followed closely to ensure the continued reliability of the equipment.

4.4.2 Confirmation of Calibration of Batch and Portable Instruments

The measurement of particle size standards to confirm the calibration of batch and portable counters follows the same procedure as normal measurement. A particle size standard is dispersed into a particle-free water after a base count has been taken of the “particle-free” water. The particle size standard is then measured. The base count is subtracted from the size standard count in order to normalise for background counts that are not attributable to the particle size standard.

The particle counter's response to the size standard can then be analysed for compliance with the relevant specifications. As with the on-line counter, at least three particle size standards should be measured.

It is crucial that all forms of pollution are avoided in this test as they can skew results. The preparation of glassware (refer paragraph 4.5.3 in this regard) and the rinsing of the particle counter are the most crucial. Multiple transferring of the standard must also be avoided as this will increase the risk of sample pollution. It is also important to follow the instructions of the manufacturers of the particle standards closely as Van Gelder *et al.* (1999) have shown how differing results can be returned due to variations in this procedure.

It is recommended that the operator of a new particle counter requests the supplier to demonstrate this test to him/her for the first time.

4.5 General Guidelines for Counting

4.5.1 Warming up of the Particle Counter

Particle counters need to warm up for at least 30 minutes before they stabilise. Counts taken before this time has expired may not be repeatable. The warming-up period is ideal for rinsing the counter and checking its operational parameters in preparation for the work that is to commence (Hargesheimer and Lewis, 1995).

4.5.2 Particle-free Water

There is no such thing as particle-free water. The term is used loosely to describe low count water. Sources of particle-free water are (Standard Methods 2560):

- deionised water
- water passed through an ultra filter
- distilled, de-ionised water produced by ion exchange and cartridge filtration (preferably using continuous closed-loop membrane filtration).

Water passed through a 2 µm and then a 0,45 µm syringe filter can also be used. It is recommended that particle-free water from the same environment as the test sample is used for dilution water as it will ensure that the chemical environment remains unchanged.

4.5.3 Cleanliness of Apparatus

Glassware and apparatus must be kept clean at all times. Standard Methods 2560 requires that the particles contributing to a sample from the glassware used are not more that 5% of the total particles measured. Hargesheimer and Lewis (1995) suggest methods for the cleaning of glassware. This includes scrubbing, rinsing with a particle-free water and then sealing to prevent atmospheric contamination. It is also advisable to rinse the container properly with the sample before a final sample is taken for analysis.

This is also valid for the particle counter used. It is important to ensure that particle counter tubing is always clean and that it does not contain any microbial growth or inorganic deposit. This can be ensured by rinsing frequently with a weak acid or any other solution recommended by the supplier of the equipment.

As an indication of the care that must be taken when working with low count analyses, the following cleaning procedure for bottles intended for use with particle counting is copied from Chowdhury et al. (2000):

1. Rinse three times with de-ionised water.
2. Scrub (by bristle brush) with a detergent solution by diluting 1 mℓ Liquinox detergent (Alconox inc., New York, NY) to 1000 mℓ with de-ionised water. (A local alternative can be substituted)
3. Rinse three times with de-ionised water.
4. Rinse with 10% HCl acid, then fill with de-ionised water.
5. Sonicate for two minutes.
6. Empty acid/water solution and rinse three times with Milli-Q water.
7. Allow the bottles to dry, inverted, overnight.

4.5.4 Sampling

Standards Methods 2560 summarises the sampling method for batch counts. Of importance is the fact that the sample is taken at a representative time and place. The sample must be taken so that the particles are subjected to the minimum shear and that bubble entrainment is avoided. Standard Methods suggests that the sample is drawn from below the water level. Once the sample has been taken the sample should be protected against any pollution from the sampling equipment, containers or the atmosphere. The sample must be sealed. Hargesheimer *et al.* (1992) report that rigorously cleaned glass containers were superior to plastic or Teflon bottles. The glass is suitable for analyses of particles as low as 0,5 µm while the plastic and Teflon bottles were adequate for studies involving analyses of particles greater than 5 µm.

4.5.5 Dilution of samples

Occasionally it is necessary to dilute measurement samples due to exceeded concentration limits. This may be necessary for some raw waters and sometimes pre-settled water samples. Post-filtration samples normally do not require dilution.

Due to the nature of sampling, it is normally very difficult to dilute samples for on-line counters and this practice is not recommended. It is much easier to dilute for batch counters.

Samples are diluted to obtain measurable particle concentrations in the sample, but in doing so, the sample can be altered to a point where the sample is no longer representative of the water that is to be characterised. Standard Methods 2560 discusses this issue in some detail and some of the major recommendations include:

- The dilution water used must obviously have very low counts but must also preferably be from the same source as the sample that is to be diluted as this will assist in preventing any chemical changes in the sample during dilution.
- The sample to be diluted must be added to the dilution water carefully in order to prevent any breakup of floc or particles in the sample.
- Diluted samples should be measured immediately and should not be stored.

The German Standard DIN 65668 - Verdünnen von Ölen zum Zwecke der Partikelzählung, loosely translated to mean “Dilution of Oils for Particle Counting”, supplies some dilution ratios to assist in the standardisation of this procedure. These recommendations are copied in Tables 4.5.5(a) and 4.5.5(b).

4.5.6 Mixing of Samples

Particles that have settled must be re-suspended before the sample is analysed. Similarly, when a sample is diluted, it should be mixed thoroughly without changing the nature of the particles. Standard Methods 2560 suggests that both mechanical stirrers inside bottles and sonication be avoided. The sample should be mixed in a sealed round bottle by gently rolling and inverting the bottle. Chowdhury *et al.* (2000) suggest mechanical means for the rolling and inversion of the bottles, which would improve repeatability.

Table 4.5.5(a) : Volumetric Quantities and Dilution Ratios (D_V)

Nr	$D_V = 1 + \frac{V_F}{V_P}$	Sample volume V_P(mℓ)	Dilution liquid volume V_F(mℓ)	Total volume (mℓ)
1	2	50	50	100
2	5	20	80	100
3	10	10	90	100
4	20	5	95	100
5	50	2	98	100

Table 4.5.5(b) : Gravimetric Quantities and Dilution Ratios (D_M)

Nr	$D_M = 1 + \frac{M_F}{M_P}$	Sample mass M_P(g)	Dilution liquid mass M_F(g)	Total mass (g)
1	2	50	50	100
2	5	20	80	100
3	10	10	90	100
4	20	5	95	100
5	50	2	98	100
6	100	1	99	100
7	200	5	995	100
8	500	2	998	100
9	1 000	1	999	100

4.5.7 Degassing of Samples

Samples occasionally contain bubbles that may occur due to turbulence or processes, i.e., cascades or flotation. These bubbles might be very small and not visible to the naked eye. They are, however, measurable and might lie in the sub 10 µm range. The problem may be increased when the sample is cold. Hargesheimer and Lewis (1995) studied this phenomenon and found that the best way to degas a sample is to let it warm to room temperature. Other methods such as applying a vacuum and sonication proved to be less effective and even detrimental to the sample.

4.5.8 Response Time of Counters (Count Stabilisation)

Particle counters do not respond immediately to a change in water quality and the change from one set of stable results to a next set of stable results is a gradual one. The re-stabilisation can take up to several minutes for portable counters and significantly longer for on-line counters. This can pose a problem if rapidly changing water quality profiles are being studied. This will includ flocculation processes and filter ripening. This is mainly due to the mixing of "old" and "new" samples in the counter's inlet manifold as well as the pollution of the previous sample's particles that are trapped in the system and that need to be flushed out.

4.5.9 Counting of Large Particles and Flocs

The counting of large particles and flocs poses a significant problem. Firstly, the danger of blockages and serious damage to the counter has to be considered. In some cases filters have been installed in-line prior to the sensor orifice in order to protect it, but this is not a practice to be recommended as the filter will change the nature of the suspension being studied. Particles that can block the flow cell should be avoided and the operator should always be attentive to this issue.

Secondly, it is unlikely that flocs can be measured accurately by particle counters due to the nature of the flow in the particle counter. As a result of the significant shear forces exerted on the floc, they are likely to break-up or change size and shape before they are measured.

4.5.10 Storage of Samples

Hargesheimer and Lewis (1995) as well as Van Gelder et al. (1999a) have shown that samples for counting cannot be stored successfully. In all cases studied it was found that the sample containers altered the particle composition of the samples significantly. This means that samples have to be analysed shortly after they are taken. Storage times in excess of 30 minutes are not recommended. This is especially true for unsettled (pre-filtered) water.

4.5.11 Pumping of Samples

It is sometimes necessary to displace samples by using a pump. Some batch counters are fitted with permanent pumps to control the flow through the sensor. It should be noted that the pumps could have a significant effect on some more fragile particles and it is recommended that the pumps are installed after the sensor as opposed to before the sensor.

STANDARDISATION OF THE USE OF PARTICLE COUNTING FOR POTABLE WATER TREATMENT IN SOUTH AFRICA

PART B : DATA AND DATA HANDLING

Chapter 5

REPORTING STANDARDISATION

This chapter suggests guidelines for the setting up and use of particle counters and also for the reporting of the data from particle counters. This is required to allow meaningful comparison of the data generated by different counters and operators. This chapter also shows how the large number of data points generated by particle counters can be reduced to fewer indices so that the technology can be employed in a practical way by treatment plant operators.

5.1 Introduction

Inconsistencies in the reporting of data complicate comparison of data sets as much as inconsistencies in the particle counters themselves. This is due to the fact that a particle counter does not report individual counts and measurements, but rather groups them into categories or bin sizes. Individual counts cannot be retrieved from these bins. If the bins are therefore set-up “incorrectly” the data is difficult to compare with other data sets. This also relates to the conditions under which the data was generated. It is imperative that particle counting guidelines include suggestions toward the actual particle counting measurement and storage of data.

The recommendations that follow in this section are based on a successful system worked out by the Rand Afrikaans University and Umgeni Water to allow both parties meaningful access to all the data generated in the course of this project.

5.2 Measurement

Particle counters allow several degrees of freedom which must be curtailed if the data is to be meaningful to a broader audience. Also, particle counts are meaningless if the counts are not accompanied by information describing the conditions under which the data was generated. In order to enhance the exchangeability of data, it is suggested that the data gathering procedure should be standardised and that it should include a number of other parameters not directly related to the actual particle counting exercise.

Batch and laboratory counters have on-board storage capacity. The data stored will only include the date and time of measurement as well as the actual count. Any additional information will have to be recorded separately and combined with the particle count as soon as possible to avoid future confusion. It is also recommended that the memory of the particle counter is cleared as soon as the data has been retrieved successfully to avoid possible confusion with new data sets.

On-line particle counters are connected to a computer monitoring system and continually download measurements to a database. The particle counters often have facilities to monitor inputs from other instrumentation on the plant (e.g. turbidity, pH, conductivity, temperature) provided these are connected to the particle counter by either 4-20 mA or 0-10V signals. The particle count information and the other signals are then stored in a common database and displayed as a continuous trend on the computer screen for use by the plant personnel.

The standardised measurement guidelines are expanded on in the following paragraphs.

5.2.1 Bin Sizes

The Environmental Protection Agency of the USA provides no guidance on the use of standard size intervals through the Surface Water Treatment Rule (SWTR). Some American researchers (Lawler, 2000) have however standardised on the following six channels : 2-3 µm, 3-5 µm, 5-7 µm, 7-10 µm, 10-15 µm and >15 µm. In principle, these size ranges are acceptable and should be utilised, but it does present some complications as it is rather limited. While some particle counters have provision for 6 bin sizes, others can cope with up to 256 bins. Most counters have only 8 or 16 bins. Operators would justifiably want to work with as many bins as their equipment would allow as they provide more information. In order to allow this without compromising the standards, the unofficial American standard is expanded on as illustrated below. Also, to facilitate the conversion of any set of data to the unofficial standard, it was decided to report particle size in terms of cumulative counts. Most particle counters report their data in a cumulative format already. The suggested standard thresholds are reported in Table 5.2.1.

The expanded bin ranges all keep the unofficial standard as a basis and can be related to that standard. It is suggested that the number of bins be limited to 16 as more bins will add to the already significant effort required in handling the data.

Generally, samples contain particles in size ranges that extend below and above the capability of particle counters. It is important to note that not all particles in a sample are measured; only those within the counter's size range. Light obscuration particle counters are always limited on the small size of a range due to "signal-to-noise" limitations (normally 1 or 2 µm) and on the upper end due to physical orifice size limitations. In the standard thresholds suggested above, and also generally in literature the measurement limit is 2 µm. Should the limit be set at 1 µm, the counts will be significantly higher due to the very high concentration of particles that occur naturally between 1 and 2 µm. The suggested standard thresholds are not limited on the upper end of the scale. The counts within the larger fractions are normally very low and can be accommodated by the suggested thresholds without loss of resolution.

Refer to the discussions in paragraph 2.3.1 on “Particle size range” and “Signal-to-noise ratio”

Table 5.2.1 : Suggested Standard Thresholds

Number of Bins	Suggested Standard Thresholds
6 Bins	> 2 µm ; >3 µm ; >5 µm ; >7 µm ; >10 µm ; >15 µm
16 Bins	> 2 µm ; >3 µm ; >4 µm ; >5 µm ; >6 µm ; >7 µm ; >8 µm ; >10 µm ; >12 µm ; >15 µm ; >20 µm ; >25 µm ; >35 µm ; >50 µm ; >100 µm ; >200 µm
8 Bins	All six bins from the 6 Bin standard plus any two other bins from the 16 Bin standard
> 16 Bins	All sixteen bins from the 16 Bin standard plus any other bins up to the maximum number of bins required

5.2.2 *Other Water Quality Parameters*

Some general water quality parameters should be measured and stored along with the particle count. These parameters will assist in evaluating the data at a later stage. The parameters should ideally be easily measurable and must characterise the sample sufficiently in order to establish the water sample quality independently of the particle count. This will allow for a broader interpretation of the particle count. The suggested parameters are:

- Turbidity
  This parameter gives an indication of the clarity of the water. Although there is no fixed relationship between turbidity and particle counts, it is a handy verification of unexpected high or low particle counts

- Conductivity and pH
  This will assist in tracking water quality changes in a process stream and will assist in establishing reasons for sudden changes in particle counts

- Water temperature
- Suspended solids where necessary, as this will also assist in the interpretation of particle counts in some cases.

5.2.3 General Information Regarding Counts

Information required here includes:

- a general description of the test,
- which particle counter was used if more than one counter is available,
- when and where the test was conducted,
- the type of water analysed, and
- who the operator was.

It is of particular importance to note this information where a portable counter is used or where a counter is used in a number of tests. It is quite possible to lose track of which set of data relates to which test.

5.3 Storage of Data

Each particle count can include up to sixteen bins with data, and measurements can be repeated as frequently as one per minute over extended periods. Also, provision must be made for the other data requirements as explained in the previous section. It should be clear that particle counting can generate enormous quantities of data. A proper data management system should be in place. The data management system should:

- allow for the storage of all the data in an orderly manner,
- allow for the easy retrieval of data based on any number of parameters, and
- process the data to some level.

Many of the data sets will need to undergo some basic form of processing before interpretation and it is recommended that the data management system includes the worked data. The same raw data may be needed at a later stage to evaluate some other characteristic of the sample that was initially not anticipated. It is recommended that the data be stored in its most basic (“raw”) form as well as this will prevent the loss of any aspect of the data.

A database was developed to cope with the data generated by the WRC Projects K5/1024 and K5/1025. The database accepts data that are offered in any of the standard forms that have been suggested in this report. This database serves as a record of these projects and is able to deliver the results to detailed queries easily and effectively. This makes the data available to future researchers. It is also possible to incorporate data generated by other researchers should they be willing to make their data available.

Apart from storing the data, the database also processes the data to an advanced level. Included are conversions from cumulative to discrete counts, count normalisation calculations, data reduction calculations, particle volume calculations, average size calculations and the fitting of least square power law coefficients. The detail of each of these calculations is expanded on later in the chapter. A broader discussion on data management is presented in Chapter 6.

5.4 Reporting of Data

Depending on what is required from the data, the data can be reported in various ways. This section will distinguish briefly amongst various ways of data representation and will also illustrate some ways in which the data can be represented graphically.

5.4.1 Total Counts (ΣN_2)

The phrase “total counts” is commonly used to describe all the particles measured. In order to be meaningful it must be accompanied by two other essential pieces of information, ie. above which threshold the measurement was done and the volume of water that the count relates to. A proper format would be: ”Total counts >2 µm per mℓ” which is the most common way to report particle counting data. Typically, this will be used to track counts over a period of time or to compare different processes with each other or the response of a process to changing conditions. This is sometimes called a “Time trend particle count” and an example is shown in Figure 5.4.1.

These graphs are used to identify problems in plant performance as sudden changes in the values will be clearly visible on the “log axes”. Occasionally, other threshold limits are also included in the same graph although these are generally redundant as it is difficult to pick up relative changes in the counts on these graphs.

5.4.2 Cumulative Counts (ΣN_i)

Cumulative counts are similar to total counts but will be used to study the particle count of a given sample or group of samples in more detail. The count per mℓ larger than every threshold in the size range is reported. As with the total count it is also difficult to determine the relative changes in the counts from these counts and graphs.

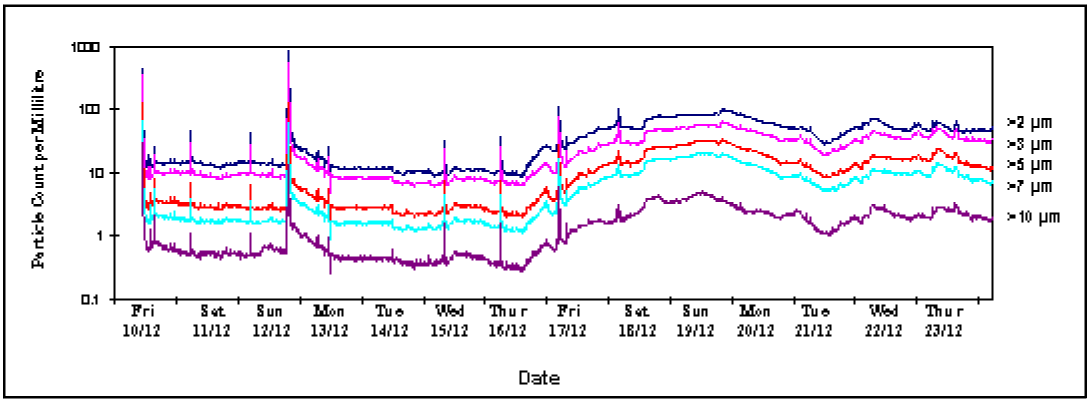

Figure 5.4.1 : Time Trend Particle Counts including Secondary Thresholds

These graphs can be plotted as bar charts or line graphs with the y-axis generally on a log scale. Figure 5.4.2 shows a typical cumulative plot of a raw water using bars.

5.4.3 Incremental or Discrete Counts (N_{i-j})

From the cumulative counts, data can be re-analysed in terms of the numbers of particles between two thresholds or per bin size using the equation :.

$$N_{i-j} = \sum N_i - \sum N_j \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots \quad 5.4.3$$

This is called the incremental or discrete count. This allows a detailed analysis of the relative importance of each of the bin sizes. Due to the sharply decreasing number of counts with increasing bin size it is generally still necessary to plot the counts on a log axis. The unit for the count for each bin remains “Counts per mℓ”.

5.4.4 Normalised Counts (Nn_{i-j})

Incremental counts can sometimes be misleading because it compares counts in increments (“bins”) that are not all the same size. Some bins are 1 µm “wide”, others are 2 µm “wide” while others are 100 µm “wide”. In order to determine the relative importance of a incremental count it has to be “normalised” to a standard bin size. The 1 µm bin size offers a handy standard and all incremental counts are divided by its own bin size to return a normalised count.

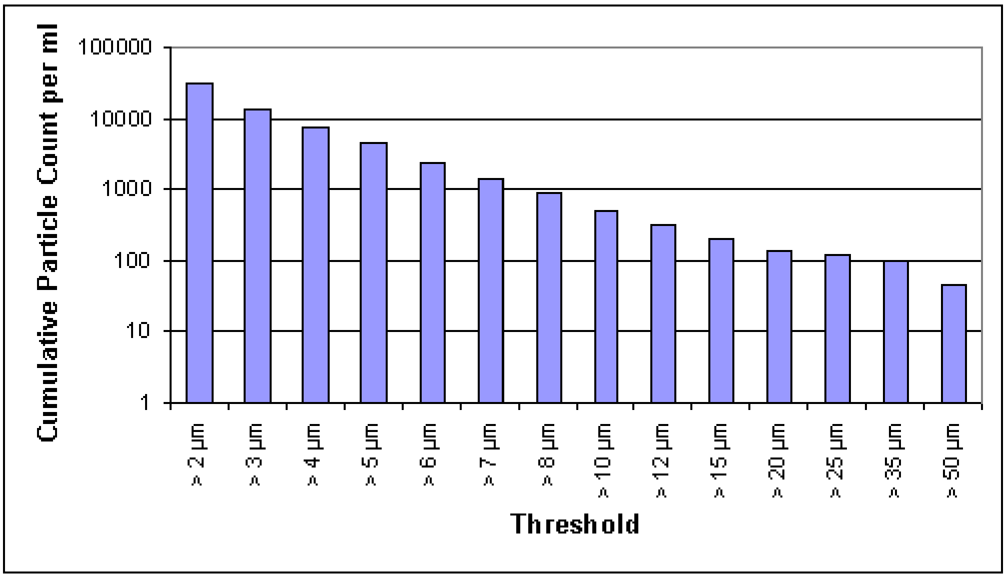

Figure 5.4.2 : Cumulative Plot of a Typical Raw Water Count

Mathematically it is expressed as follows:

$$Nn_{i\text{-}j} = \frac{\sum N_i - \sum N_j}{d_j - d_i} \qquad \text{5.4.4}$$

Due to the normalisation the unit for the count changes to “Counts per mℓ per µm width”. This explicit unit is not used frequently, and literature generally makes use of the abbreviated “Normalised counts per mℓ”.

Figure 5.4.4 compares the discrete count of the raw water illustrated in Fig. 5.4.2. Note how the normalised counts drop toward the larger size fractions when compared to the discrete counts. This particular sample illustrates a strange phenomenon as the normalised counts increase as well. This is not normally the case. The increase is not as much as the discrete count would suggest at first glance.

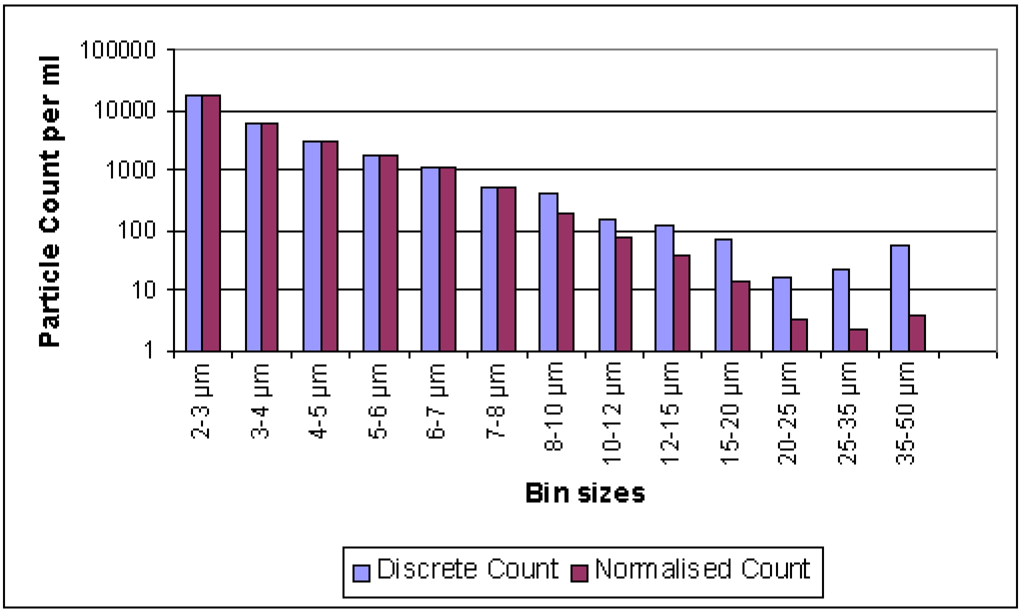

Figure 5.4.4 : Discrete and Normalised Plot of the Raw Water Shown in Figure 5.4.2.

5.4.5 *Geometric Mean Size (d_g)*

The use of the geometric mean has its origins in sieve analyses where the geometric mean size is used as an approximation of the average particle size between two sieves. In particle counting, the thresholds or "bin boundaries" act as the sieves. The geometric mean size of a bin is calculated as shown below and is used when plotting the incremental and normalised counts as these counts do not relate to a specific threshold, but rather to a count between thresholds.

$$d_g = \sqrt{d_i . d_j} \qquad \text{5.4.5}$$

Here d_i and d_j denote the upper and lower thresholds of a bin.

5.4.6 *Particle Volume (V_{i-j}) and Particle Surface Area (S_{i-j})*

Particle Volume and Particle Surface Area are parameters derived from the actual particle count in order to shed more light on the nature of the suspended material. Particle volume, and by implication particle mass, is useful when the behaviour of the suspension is determined by the larger particles in the suspension, i.e during orthokinetic flocculation and the consideration of issues relating to treatment residuals such as sludge. Surface area is important when the adsorption of contaminants onto particles are considered (Tobiason, 2000). As both surface and volume are measures of the particle *concentrations*, it is important that they be linked to volume of water measured. The calculation of particle surface and volume is based on the assumption that the particles are perfectly spherical and the following equations are used:

$$V_{i-j} = N_{i-j} \frac{\pi d_g^3}{6} \quad \ldots\ldots\ldots\ldots \quad \textit{5.4.6(a)}$$

$$S_{i-j} = N_{i-j} \pi d_g^2 \quad \ldots\ldots\ldots\ldots \quad \textit{5.4.6(b)}$$

The most convenient units to use for concentration parameters are mℓ/ℓ (or mm^3/ℓ) for V_{i-j} and mm^2/ℓ or S_{i-j}.

5.5 Data Reduction

Although it is desirable to retain as much of the data generated during measurements, it complicates matters when the data is required for the day to day operation of a plant or when only specific parameters are monitored. Several procedures exist to reduce the multiple data points to one or two meaningful indexes or figures. Three of these procedures are presented below.

5.5.1 Counts per mℓ > 2 µm

As discussed under the previous heading, the total number of particles present a easy way of reducing the particle count to a single index and this is handy to monitor a process from one day to the next. In this form, it can be used like turbidity which is also a single figure indicator of water clarity. However, one of the major advantages of particle counters is that they not only give an indication of the total water clarity, but also show the character of the particles that effect the clarity of the water. If only the total counts are reported, this advantage is lost. This method is not recommended.

5.5.2 Three-point Classification Rule

The most promising and most widely used method for particle count data reduction is standardised in the form of ISO 4406: “Hydraulic Fluid Power - Fluids - Method for coding level of contamination by solid particles”. This standard was developed for and is employed extensively by the petroleum industry.

For ISO 4406:1987, two critical particle counts were decided upon, ie. > 5 and >15 µm. These sizes were taken as indicators of the state of the machinery as sudden increases in the counts in any of the three ranges would indicate the onset of a particular problem. In the 1999 revision of ISO 4406 these critical sizes were revised to >6 µm and >14 µm and an additional size, >4 µm, was added to the list. ISO 4406 supplies a table whereby the counts for the critical sizes can be compared. An index is then taken from the table for each size range and the fluid can be classified according to three indices that reflect the critical parameters. This is known as the three-point classification rule and it allows the operators to identify significant changes in oil quality without much difficulty.

This principle of data reduction could be employed fruitfully in the potable water industry with some minor alterations that will leave the principle intact, but will make the system more suitable.

There are two main reasons for the introduction of particle counting to the potable water industry. The first is the rising concern surrounding the presence of *Cryptosporidium* and *Giardia* in treated water and the need to check for their presence. The second is the fact that traditional turbidity measurement is not considered adequate for the measurement of water clarity despite the introduction of highly sensitive new turbidimeters. The critical sizes recommended and selected from the standard particle counting range should reflect these two considerations. The following were selected:

>2 µm : This is also equivalent to the total particle count and will reflect the clarity of the water.

>5 µm : This size measurement along with 15 µm will reflect the size increment in which the *Giardia* and *Cryptosporidium* is expected to occur. A rise in this range will indicate an increased likelihood of the presence of these organisms in the treated water.

>15 µm : High counts in this range will indicate significant filter failures.

The ISO 4406 index table was tested for direct application in the potable water industry, but it was found that the index resolution was not small enough to reflect changes in water clarity that were thought to be significant. Therefore, the index table was modified. The original ISO 4406 table contained scale numbers from 0.9 to 24 and the counts linked to a given scale number was double that of the previous scale. For example, scale number 8 was applied to counts between 1,3 and 2,5 counts per mℓ and scale number 9 was applied to counts between 2,5 and 5,0 counts per mℓ. In the revised table the factor applied to the intervals is $\sqrt{2}$ which effectively doubles the number of scales and provides a resolution better suited to the potable water industry. It was also necessary to increase the scale to a scale number of 60 to deal with raw waters. The revised scale values are presented in Table 5.5.2.

Application of the rule is fairly simple. Assume the corresponding particle counting values for values for >2, >5 and >15 µm are 50 478, 11 189 and 199 counts per mℓ respectively, the classification would then be 45:41:29 based on the corresponding scale numbers from Table 5.2.2.

Table 5.5.2 : Scale numbers for the application of the Three Point Classification Rule in the Potable Water Treatment Industry

Number of Particles per mℓ		Scale Number
More Than	Up To And Including	
7 500 000	10 000 000	60
5 000 000	7 500 000	59
3 750 000	5 000 000	58
2 500 000	3 750 000	57
1 950 000	2 500 000	56
1 300 000	1 960 000	55
960 000	1 300 000	54
640 000	960 000	53
480 000	640 000	52
320 000	480 000	51
240 000	320 000	50
160 000	240 000	49
120 000	160 000	48
80 000	120 000	47
60 000	80 000	46
40 000	60 000	45
30 000	40 000	44
20 000	30 000	43
15 000	20 000	42
10 000	15 000	41
7 500	10 000	40
5 000	7 500	39
3 750	5 000	38
2 500	3 750	37
1 950	2 500	36
1 300	1 950	35
960	1 300	34
640	960	33
480	640	32
320	480	31
240	320	30
160	240	29
120	160	28
80	120	27
60	80	26
40	60	25

Table 5.5.2 (Continued)

30	40	24
20	30	23
15	20	22
10	15	21
8	10	20
5	8	19
3.75	5	18
2.5	3.75	17
1.95	2.5	16
1.3	1.95	15
0.96	1.3	14
0.64	0.96	13
0.48	0.64	12
0.32	0.48	11
0.24	0.32	10
0.16	0.24	9
0.12	0.16	8
0.08	0.12	7
0.06	0.08	6
0.04	0.06	5
0.03	0.04	4
0.02	0.03	3
0.015	0.02	2
0.01	0.015	1
0.0075	0.01	0

The classification rule has the advantage that it mitigates smaller variations in counts. Any variation in the classification of the water indicates fairly significant changes in water quality and should serve as an indication to the operator that some action is required.

5.5.3 Power Law Coefficients

When the normalised particle count (i.e., particle counts divided by the relevant bin size) is plotted on a log-log graph, it usually displays a near linear nature and it can be represented in the form of a power law function as given below:

$$Nn = A \cdot {d_g}^{\beta} \quad \ldots\ldots\ldots\ldots \quad 5.5.3(a)$$

Where Nn is the normalised count per size range per mℓ, d_g is the geometric mean size for the size range (µm) and A and β are the correlation coefficients . The equation can be rewritten in log form:

$$\log(Nn) = \log(A) + \beta \log(d_g)$$
$$\alpha = \log(A) \qquad \ldots\ldots\ldots\ldots \quad 5.5.3(b)$$
$$\log(Nn) = \alpha + \beta \log(d_g)$$

Here α and β are the power law coefficients that can be used to describe any particle count. The first variable, α, gives an indication of how many particles are present in the sample and it can be related to the exact count of particles at d_g=1 µm since then:

$$\log(Nn) = \alpha \qquad \ldots\ldots\ldots\ldots \quad 5.5.3(c)$$

The advantage of this system over the “total count > 2 µm” system becomes apparent when the second coefficient, β, is considered. In the log form of the power law β describes the slope of the line. In terms of the particle count, this indicates the spread of the particle sizes in a sample. An increasing β-value indicates that the spread of particle sizes is increasing, while a decrease in the β-value indicates that the spread in particle sizes is shrinking.

It is has been assumed that β remains constant throughout the size range. Fundamentally, this cannot be (Lawler, 1996). To stop the *number* concentration of particles to become infinite as particle size approaches zero, the value of β has to be smaller than 1. On the other hand, the value of β has to be greater than 4 to stop the particle *volume* from becoming infinite as particle sizes become larger. A single β can not be valid over a very broad size range. To circumvent this conceptual problem, the exponent β can be made to vary with particle size. This was not done for this project, instead the particle size range considered was limited to between 2 and 50 µm. In this size range the particle size distribution displays a highly linear nature on log-log.

The power law should be used with circumspection. Its fit has to be carefully checked with actual counting data. If it fits the data closely over the complete measured size range, it may be used purely as a convenient data reduction tool. Even then, it may be inappropriate to extend the distribution beyond the sizes from which it had been calibrated.

Provided that the power law does fit the measured data with satisfactory goodness-of-fit, integration of the power law may be used to extract some additional descriptive parameters. Integration of the power law between two arbitrary diameters will yield the total number of particles in that size range:

$$N_{i-j} = \frac{A}{\beta+1}\left(d_j^{\beta+1} - d_i^{\beta+1}\right) \quad \text{5.5.3(d)}$$

Here N_{i-j} is the number of particles between two arbitrary particle sizes d_i and d_j.

Likewise, the volume of particles between two arbitrary diameters can be derived from integration:

$$V = \frac{\pi A}{6(\beta+4)}\left(d_j^{\beta+4} - d_i^{\beta+4}\right) \quad \text{5.5.3(e)}$$

This latter equation, where V is the combined volume of the particles between the arbitrary particle sizes, is less subject to excessive scatter than volume determinations based on primary counts when the larger particles, of which very few are sometimes measured, are considered.

The power law coefficients can be calculated from measured data using the following statistical formulae:

$$S_{xx} = \sum x^2 - \frac{1}{n}\left(\sum x\right)^2 \quad \text{5.5.3(f)}$$

$$S_{yy} = \sum y^2 - \frac{1}{n}\left(\sum y\right)^2 \quad \text{5.5.3(g)}$$

$$S_{xx} = \sum xy - \frac{1}{n}\left(\sum xy\right)^2 \quad \text{5.5.3(h)}$$

$$\alpha = \frac{\sum y - \beta \sum x}{n} \quad \text{5.5.3(i)}$$

$$\beta = \frac{S_{xy}}{S_{xy}} \quad \text{5.5.3(j)}$$

$$r^2 = \frac{S_{xy}^2}{S_{xx} S_{yy}} \quad \text{5.5.3(k)}$$

In these equations x is d_g (µm), y is Nn (per mℓ) and r^2 is the correlation of the data to the function. The correlation to the function is very good and it can be expected that the values of stable water samples will lie above 0,95 if particles between 2 and 50 µm are considered.

Hargesheimer *et al.* (1992) suggest that the most reliable values for β are obtained if more than 10 particles per mℓ have been enumerated in all channels. It is important to obtain a particle count data set with high counts over a wide range of channels to obtain a good estimation of β.

Chapter 6

DATA MANAGEMENT

6.1 Introduction

Particle counters generate lots of data. In order to derive the most benefit from this tool it is necessary that the data be managed effectively. This relates to both the storage of data and also to the accessing of data for further analysis. This becomes particularly challenging when several on-line counters have to be managed or when a portable or batch counter is used in a number of different tests. If information other than time trend counts are required, further analysis of the data is required. This is tedious work and a level of automation assists in this effort.

6.2 Commercial Particle Counter Software

Manufacturers of particle counters will supply software to manage data for their specific products. Often, the supplier will have several packages available that will increase in levels of complexity and capability. This may include the facility to continually log the information from an on-line particle counter on a network and display this in a graphical format. These packages can be extremely useful where a plant does not have access to a SCADA system.

6.2.1 Download Software

This is the most basic form of software available. The software transfers the data from the particle counter to a computer and has no display or analysis function. The data is normally stored as a ".txt" or ".csv" file which can then be imported into a spreadsheet package for further analysis.

6.2.2 Information Display Software

These packages will download and display the information. The information is generally displayed in graphical format and several display alternatives are available. Other than this the software has limited analysis capabilities.

6.2.3 *Comprehensive Logging Systems*

It is normally possible to store information from the counter and information from other sources such as on-line turbidity meters or other on-line particle counters that can supply an electronic signal. The information can normally be viewed in graphical form. As with the information display software, this is normally limited to cumulative and discrete count time trend plots with other measurements superimposed on that. All the data from the particle counters (particle data as well as other inputs) will be stored in the database. Procedures are also normally provided to access the data and write it to a separate data file for further analysis. The form of the data can not be changed and this might be taken into consideration when setting up the on-line instrument as described in paragraph 4.3.

During the course of this project, two commercially available logging systems were used. Although both sets of software performed adequately for the purpose of daily monitoring of water quality and particle counts, both were found to be lacking when more extensive graphing and reporting functions were required. Some problems encountered while working with the software included:

Commercial software set 1.

- The software stored particle counting data and analogue data in a separate file for each day for each on-line instrument on the network.
  012504.dbf - (Sensor 1; 25 April)
  022803.dbf - (Sensor 2; 28 March)
  No reference is made to the year in the filename, therefore every year data will be overwritten.
- The software limits the number of days worth of historical data that can be incorporated into a report to 22 days. Historical data can not be extracted using the software.
- The graphing module allows visual presentation of only 8 data records. On a single graph the user is not able to view all 8 particle size bins as well as analogue inputs (i.e. turbidity, pH).
- If any of the settings for a particle counter are changed (i.e. particle size bins), the datafile is renamed to a backup file and the built-in database file only contains data of the new particle size channels. If the settings are changed more than once per day, data will be lost.

Commercial software set 2.

- One database file is maintained and becomes increasingly large and cumbersome, resulting in slow graphing and reporting.
- Although an automatic backup takes place regularly, the risk of losing data is increased when compared to storing data in separate files on a daily basis.
- The graphs that can be plotted only allow presentation of four data records, although up to 4 graphs can be displayed at a time.
- On three occasions the software would not initialise properly. This was overcome by reloading the software and ensuring that the settings were specified correctly.

The systems described above can generally not log all the information required as described under paragraph 5.2 (i.e. particle index or power law coefficients). A more comprehensive system is required for this.

If an on-line particle counter is being used as a batch counter and is installed in the configuration as described in paragraph 4.3.1, there is a period during each sample measurement when the counts have not stabilised. This data is irrelevant and should be discarded. It should not remain in the database and result in excessive memory being used by data of uncertain origin.

6.3 Comprehensive Data Management Tools

When a number of on-line counters have to be managed or when a portable or batch counter is used in a number of different tests, good data management tools become essential. The tool has to:

- provide a comprehensive record of the particle counts,
- contain supporting information regarding the origin of the counts as well as other water quality parameters that will assist in the interpretation of the counts,
- allow for the retrieval of the data based on a number of parameters
- process data to a level that will facilitate the interpretation of the results.

These tools are not available commercially yet. In order to allow for the efficient management of the data generated during the course of this investigation, a database was developed to function as such a comprehensive data management tool. The database was used extensively in the course of this project and provided a convenient format for the archiving of the data as required by the WRC. The structure of the database is expanded on in the following paragraph.

6.4 The RAU / Umgeni Water Particle Counting Database

The RAU/Umgeni Water database formed an integral part of the work undertaken in this project. It was used to store the data gathered during the course of the various investigations and also did a significant portion of the data processing. Large amounts of data were generated and therefore an intensive processing effort was required to convert the particle counter output into usable data. Since most of the processing could be standardised, the database was programmed to handle this. The data stored and the calculations performed included:

- The place, date and nature of water tested,
- the particle counter used,
- special notes regarding the count,
- the time of measurement,
- the cumulative count information
- the differential count information,
- other water quality parameters that were measured including temperature, conductivity, pH, suspended solids and turbidity,
- the normalised counts,
- the index classification of the sample,
- the power law coefficients and correlations,
- the incremental volume counts, and the
- volume average diameters.

Having these functions available in a data management tool proved to be invaluable in the successful completion of this project.

STANDARDISATION OF THE USE OF PARTICLE COUNTING FOR POTABLE WATER TREATMENT IN SOUTH AFRICA

PART C : PARTICLE COUNTING STUDIES IN POTABLE WATER TREATMENT

Chapter 7

CHARACTERISATION OF RAW WATERS AND FILTERED WATERS

7.1 Background

The objective of this case study is to determine whether particle counting can bring a meaningful improvement in the way that raw and final waters are characterised. Raw water classification is important for process designers, which have to anticipate the range of water quality expected during the lifetime of a treatment plant, and have to select processes accordingly. Raw water classification is equally important for treatment plant operators, who wish to draw on their experience in dealing with different water quality profiles at different times. Proper filtered water characterisation may provide an external benchmark for operators against which to evaluate their own plant performance.

7.2 Literature Study

7.2.1 Raw Water Characterisation

Raw water is commonly characterised by single water quality parameters. In South Africa, there are a few parameters which are used commonly:

- Turbidity is an important parameter where surface waters become extremely turbid during flood periods. This poses a problem over most parts of South Africa, but nowhere more so than in the Caledon River where steep river gradients are compounded by easily erodible Drakensberg basalt in their catchment areas.
- Chlorophyll is important where algal growth proliferates. This is a problem in eutrophic waters, which are encountered over all South Africa, but most pronounced in the surface waters downstream of larger metropolitan areas. This problem is likely to get worse as indirect reuse becomes more intense with increasing pressure on water resources.
- Colour is a problem in the coastal areas of the southern and western Cape. In fact, some of the highest colour values ever recorded, were measured in these regions.
- Alkalinity may drop very low in some parts (often coinciding with areas with high colour), which raises problems with stability. In other parts it may be quite high.

These concerns have led to a loose verbal classification used locally, namely *turbid flood* waters, *eutrophic inland* waters, and *soft, coloured coastal* waters.

Some attempts have been made towards more quantitative classification. Raw water turbidity limits have been used for evaluating the applicability of slow sand filtration or direct filtration. Raw water turbidity has been combined with raw water chlorophyll to come up with a two-way classification for evaluating the preferred domains for direct filtration, settling, dissolved air flotation, and where pilot testing may be required.

Particle counting is rarely used for raw water classification. A study was published (Wiesner et al; 1987) where the optimal selection of treatment processes on the basis of particle size and concentration was proposed.

The thrust of this case study will not be to verify the model of Wiesner, or to come up with new treatment selection criteria, but simply to relate a number of standard particle count parameters to the very commonly used turbidity measure, and to determine whether particle counts could possibly reveal more of the raw water nature than turbidity.

_____*7.2.2 Filtered Water Characterisation*

Particle counting should be a powerful indicator of filtered water clarity. Conventional turbidity sensitivity drops off at the very low turbidity of well filtered water, whereas particle counts produce much larger numbers which, theoretically, allow finer resolution. Particle counts are, in fact, used by many overseas utilities for this purpose, but the counts still lack the reproducibility and robustness to allow firm quantitative comparison. For this reason, water quality standards have not been phrased in terms of particle counts, but still rely on turbidity.

With the growing sensitivity to the possibility of protozoan oocysts breaking through conventional treatment, particle counts in the specific size range of the oocysts have received much attention. Once again, the technology does not meet all the expectations. Firstly, particle counting cannot distinguish between viable and non-viable cysts and oocysts. Secondly, there is no guarantee that cysts and oocysts will not be associated with larger flocs and thus break through in the larger size ranges. Another case study in this report will deal with this problem in much more detail.

The thrust of this case study will be to compare particle counts with turbidity, to see whether particle counting will bring additional illumination, and to provide a preliminary baseline for filtered water quality in South Africa to allow new users of particle counters some external comparison.

7.3 Materials and Methods

A total of nine treatment plants (labelled A to J, excluding I) were visited between June and August 2000 and again during November 2000 and January 2001. This allowed for the inclusion of both winter and summer data. During each visit, which lasted a few days, a number of samples were drawn from the raw water source, as well as the filtered water. The number of samples per plant drawn ranged from four to sixteen. This produced a total of 114 independent raw water samples, and 126 independent filtered water samples. (At one treatment plant, there were two filter blocks of different age and design, but which shared the same raw water source; therefore the larger number of filtered water samples than raw water samples.) All samples were also analysed for turbidity (and a few other parameters not reported on here). All samples were analysed with the same machine and the exact same procedures were followed in each case – only two particle counter operators were used for all tests.

For the particle counts, all counts between 2 µm and 200 µm were taken into account. For the calculation of particle volume, only particles between 2 µm and 50 µm were considered. The *volume-averaged particle diameter* was taken as the particle diameter at which 50% of the total particle volume was contained in particles smaller than that diameter and 50% of the volume was contained in particles larger than that diameter. Particles larger that 50 µm were not included in this calculation as it contained very limited counts that skewed the result.

7.4 Results and Discussion

7.4.1 Raw Water Characterisation

The raw water is initially characterised by the better known parameter of turbidity (Figure 7.4.1(a)) and pH (Figure 7.4.1(b)). Five sources are characterised by medium turbidity (between 5 NTU and 30 NTU), while four can be classified as low turbidity waters (median turbidity less than 5 NTU). Five sources would be classified as waters with medium-low pH (median values less than 7.5) with the others at higher pH, as is shown in Figure 7.4.1(b).

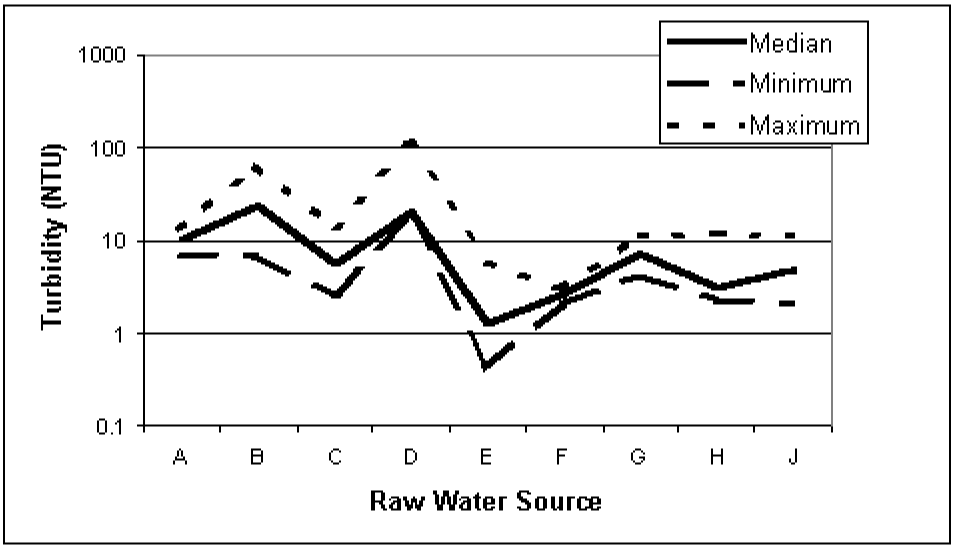

Figure 7.4.1(a) : Turbidity of the of Raw Water Sources

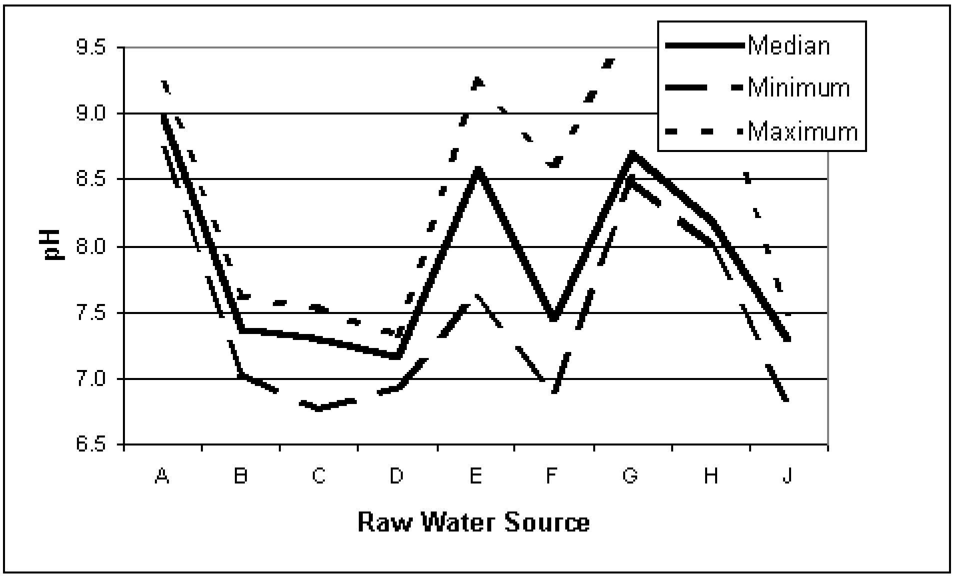

Figure 7.4.1(b) : pH of the Raw Water Sources

When one turns to particle counts, more dramatic differences between sources become apparent. The particle counts of all particles larger than 2 μm (Figure 7.4.1(c)) show that counts for individual samples range over three orders of magnitude. Counts taken from the same source, separated only by a few days, differ as much as one order of magnitude. When the counts are manipulated to give total particle volume (Figure 7.4.1(d)) and the volume-averaged diameter (Figure 7.4.1(e)), the results are equally striking. Sources A and B have a significantly higher particle volume concentration, with the other sources being fairly similar. The diameters, however, are clearly different. Only three sources have an average diameter of less than 10 µm, while some other sources have diameters as high as 30 µm. This can only point to a significantly different nature of the suspensions. The small diameters are probably due to clay colloids, as these sources are fairly turbid water taken directly from flowing rivers. The other sources are mostly eutrophic impoundments, where the suspensions are probably mainly algal in nature.

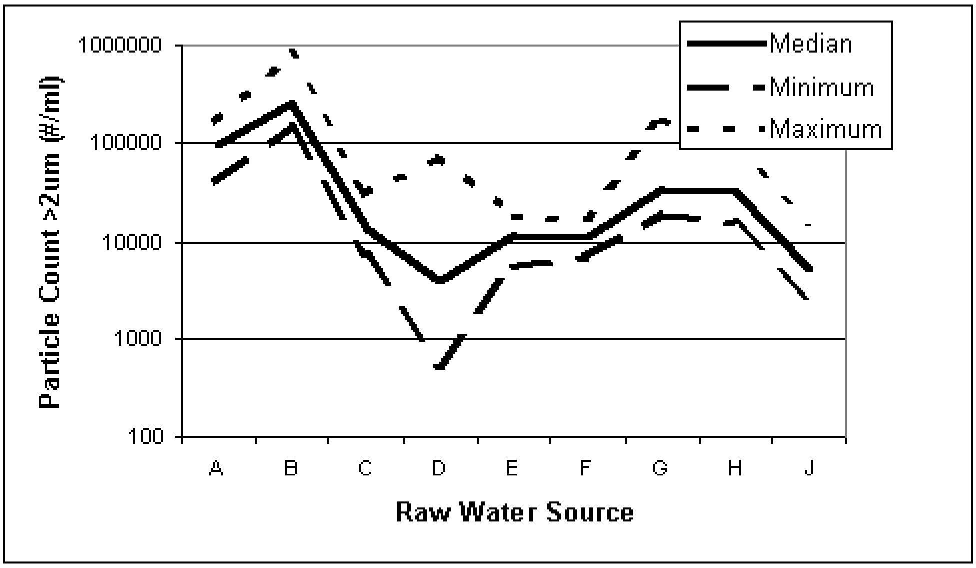

Figure 7.4.1(c) : Particle Counts of the Raw Water Sources

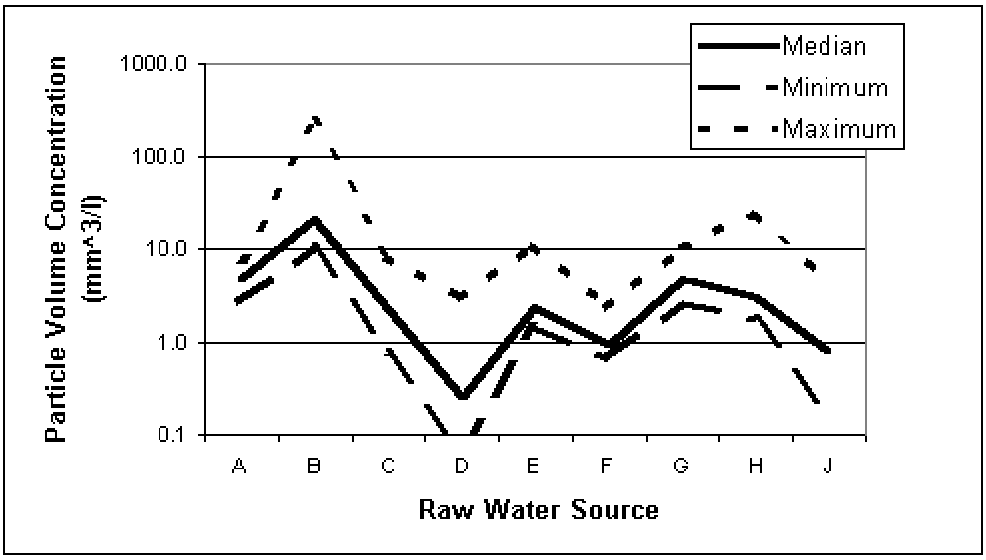

Figure 7.4.1(d) : Particle Volume Concentrations of the Raw Water Sources

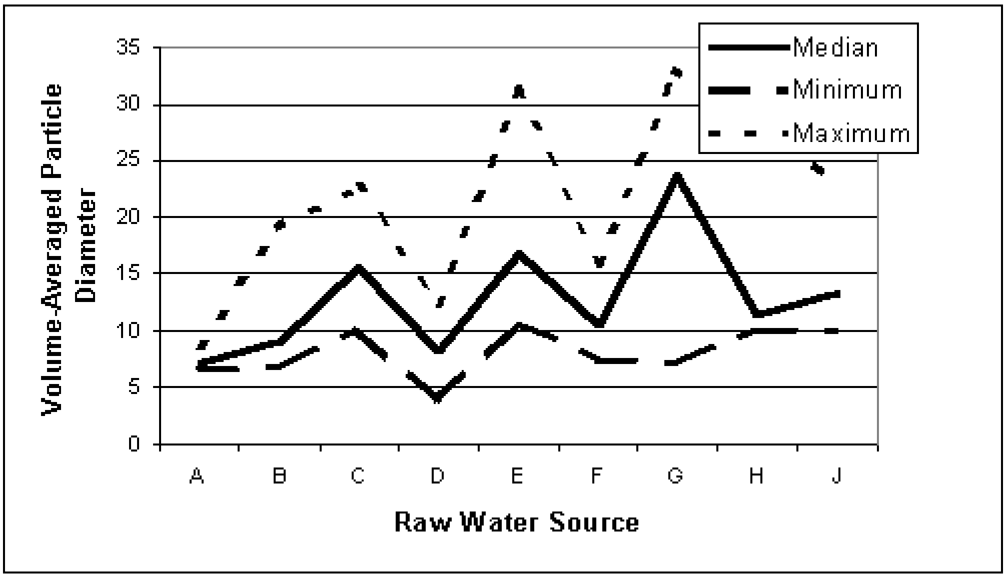

Figure 7.4.1(e) : Volume-Averaged Particle Diameters of the Raw Water Sources

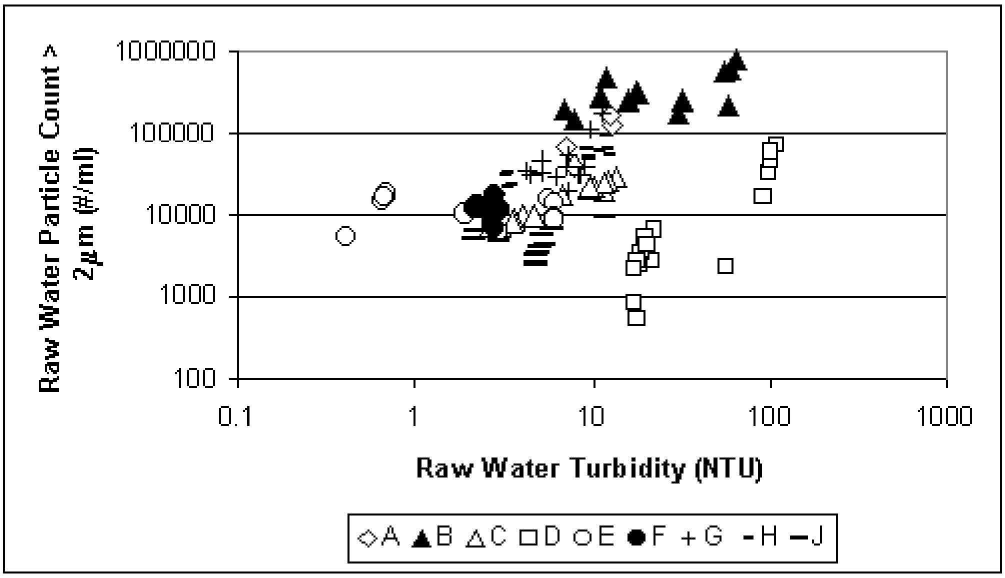

Figure 7.4.1(f) : Raw Water Turbidity vs. Raw Water Particle Counts

It is also instructive to look at correlations between some selected variables. The first obvious question is how the counts relate to the often-used turbidity. This two-way plot is shown in Figure 7.4.1(f). Here it is very obvious that water from different sources tend to group on different regions of the plot. Within the data points for each source, there is an obvious relationship between counts and turbidity. When all the data points are viewed together, there is significant scatter, as many other researchers have found. Sources D and E illustrate this well. Although, at times, source D is ten times more turbid than source E, it still has a lower particle concentration!

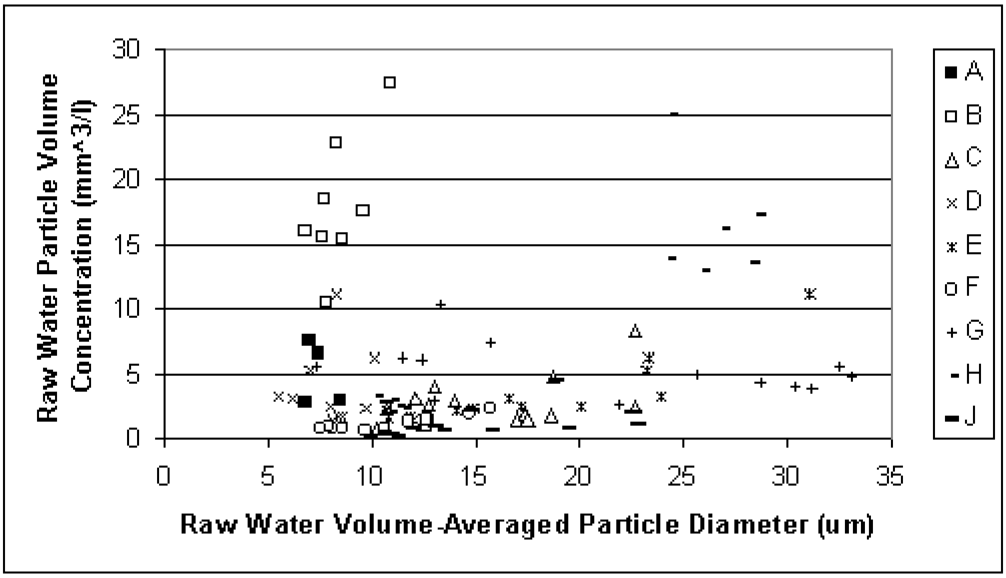

Figure 7.4.1(g) : Raw Water Volume-Averaged Particle Diameter vs. Particle Volume Concentrations

A most interesting correlation is between the volume-averaged particle diameter and the calculated particle volume, shown in Figure 7.4.1(g). The graph shows two distinctly different regions. The one region shows a band of small particles which can reach fairly high particle volume concentrations. The sources which fall in this band, are the sources which are derived from flowing, turbid rivers, while the other sources are mainly eutrophic with organic suspensions.

7.4.2 *Final Water Characterisation*

The turbidity of the filtered water from the 10 filter plants is shown in Figure 7.4.2(a). Except for a small number of outliers, the values are consistently good, with some values exceptionally low at or below 0,10 NTU.

The particle counts are shown in Figure 7.4.2(b) and the particle volumes in Figure 7.4.2(c). Once again, much more dramatic differences show up. The counts cover a range of almost three orders of magnitude, and the particle volume two orders of magnitude. High counts do not necessarily translate in high particle volumes, which indicate differences in the volume-averaged particle diameter, even after treatment.

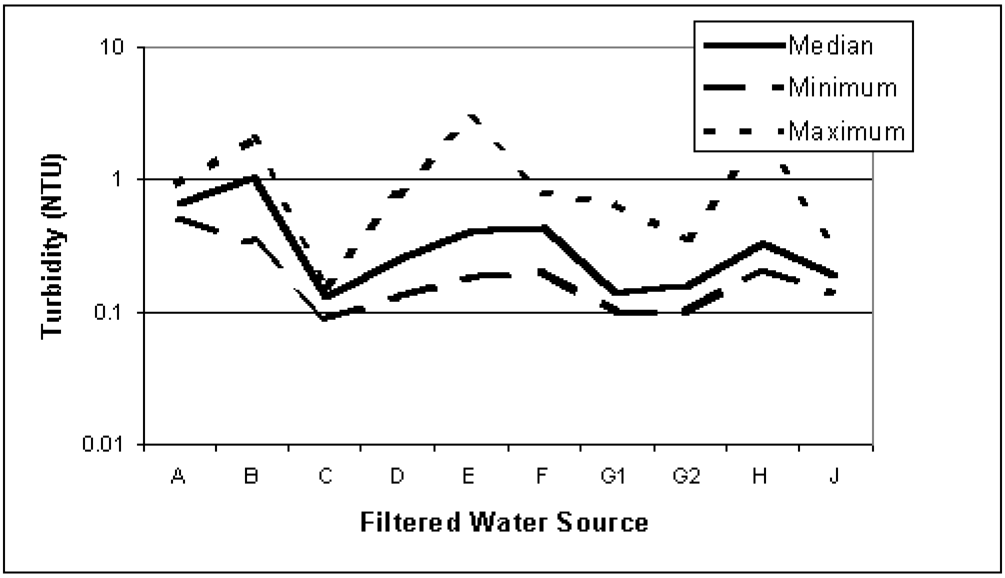

Figure 7.4.2(a) : Turbidity of Filtered Waters

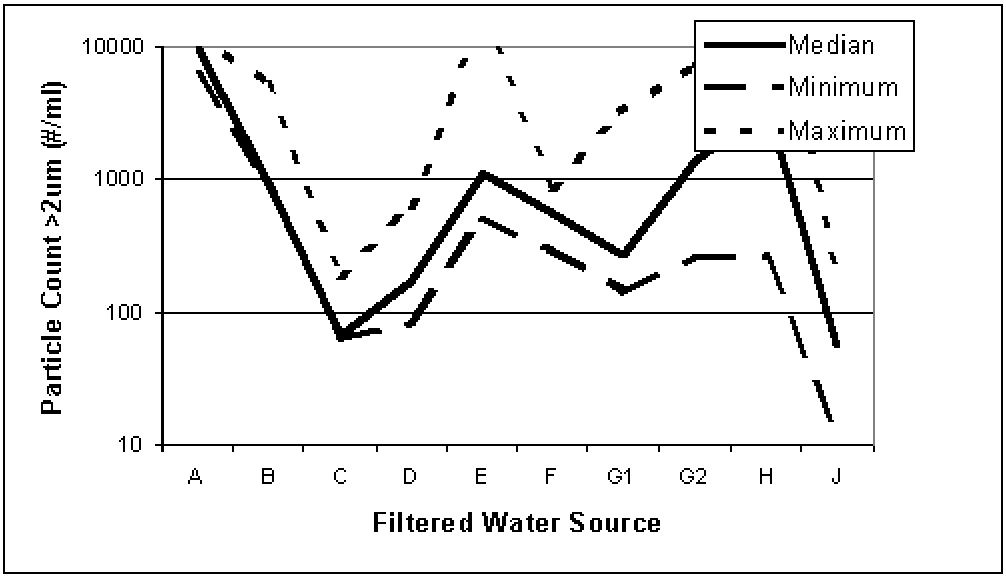

Figure 7.4.2(b) : Particle Counts of Filtered Waters

It was demonstrated in the previous section that large differences exist between raw water sources in terms of the volume-averaged particle diameter. One would expect, though, that these differences would be largely ironed out during the treatment processes. Figure 7.4.2(d) shows that this is not the case.

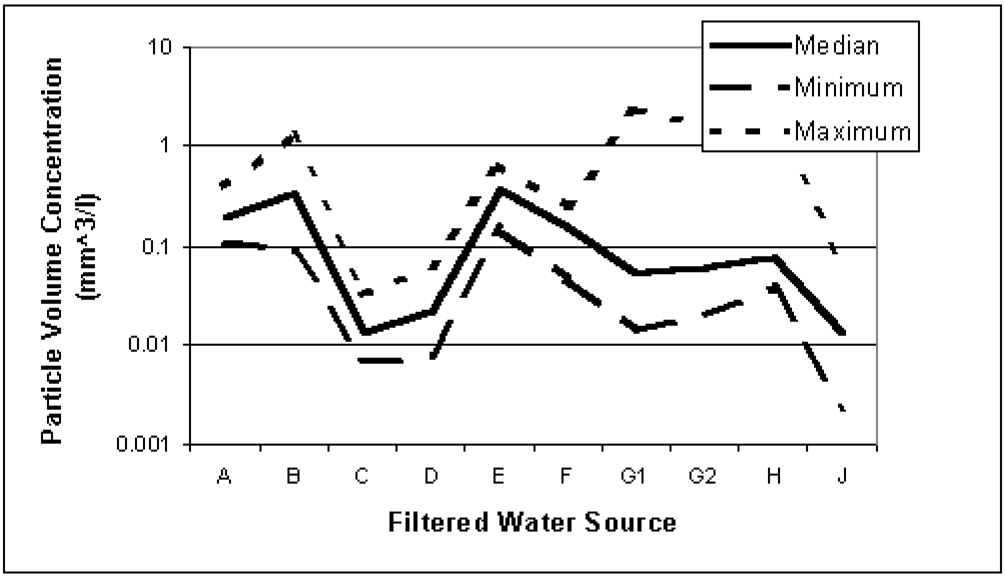

Figure 7.4.2(c) : Particle Volume Concentrations in the Filtered Water Source

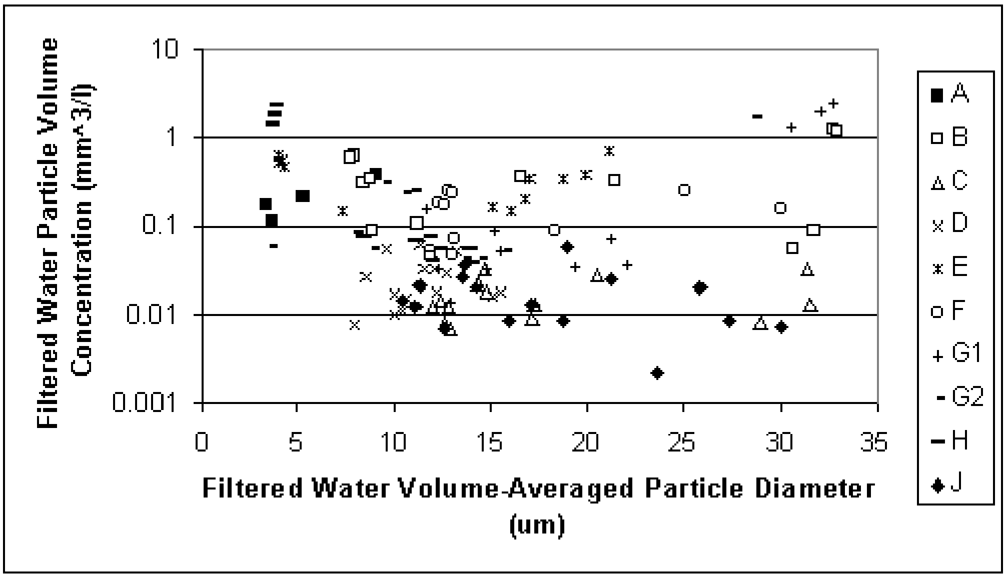

Figure 7.4.2(d) : Filtered Water Volume-Averaged Particle Diameters vs. Filtered Water Particle Volume Concentration

The lack of correlation between particle counts and turbidity, shown earlier for raw water sources, is even more evident for filtered water, as shown in Figure 7.4.2(e). In the bracket between 0,1 NTU and 0,2 NTU, for example, there is three-log variation in particle counts! However, if plants are viewed individually, a correlation between turbidity and particle counts is again evident. This reflects the fact that the differences in particle nature, which was demonstrated for the raw water sources, persist through the entire treatment process.

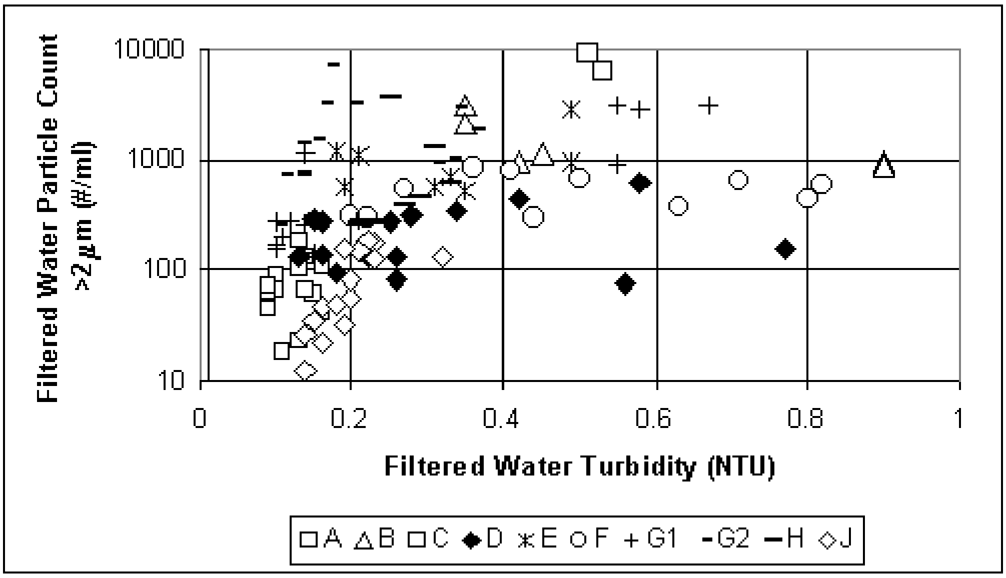

Figure 7.4.2(e) : Filtered Water Turbidity vs. Particle Counts

7.4.3 Treatment Effects

In general, it was found that all parameters discussed clustered fairly tightly for all the raw water sources and the filter plants. The median value of each parameter is thus a reasonable single-point descriptor for each sampling point, and will be used in this section.

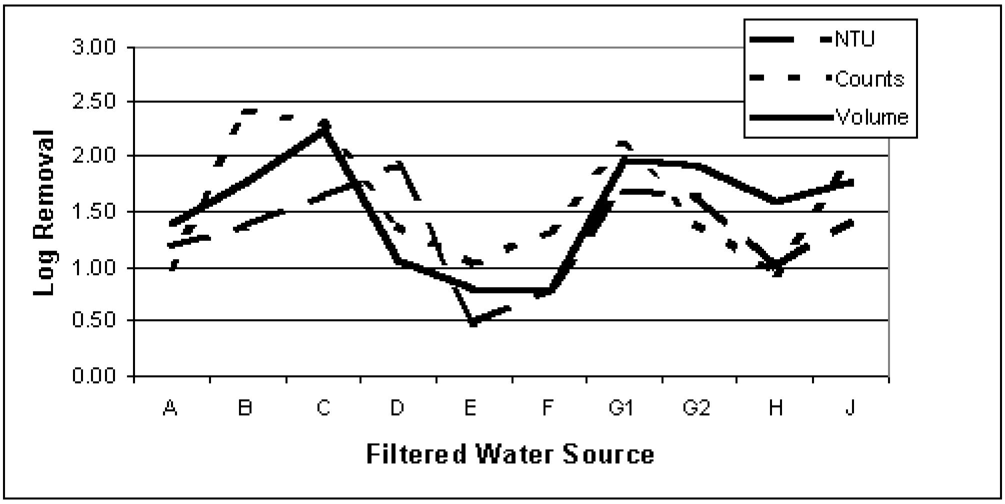

Figure 7.4.3 : Log Removals Based on the Three Water Quality Measurement Parameters

The log-removal was calculated for each plant, using turbidity, particle count and particle volume respectively. This is shown in Figure 7.4.3. Generally, removal for all parameters range between 1 and 2 logs. Where the log-removal for turbidity seems low for plant E, it can be explained by the very low turbidity at which this plant started out. Treatment performance is also a function of the specific treatment plant. Comparison of plants G1 and G2 show that the same raw water, dosed identically, will be treated to different levels due to small differences in settling tank and filter design.

7.5 Conclusions and Recommendations

A number of conclusions can be drawn from the brief survey presented. A comparison of the nine raw water samples indicate:

- There are only small differences in raw water turbidity, mostly relatively low. This demonstrate the inherent limitations of turbidity as a means of classification, as nothing of the character of the suspended material is revealed.
- Particle counts, and especially the volume-averaged particle diameter, in contrast, show that there are significant differences in particle suspensions. Even in winter, the particles in the running river sources are small and probably colloidal clay, while some other sources show large, probably organic particles.
- There is no clear correlation between particle counts and turbidity, although a relationship is found when only one source is viewed at a time.
- Particle counts ranged from 500 to 810 000 particles/mℓ > 2 µm with median 16 800 particles/mℓ > 2 µm.
- Particle volume ranged from 0.06 to 240 mm^3/ℓ with median 2.3 mm^3/ℓ.
- The volume-averaged diameter ranged from 4 to 33 µm with median 12 µm.
- Turbidity ranged from 0.4 to 109 NTU with median 6.2 NTU.
- The pH ranged from 6.8 to 9.7 with median 7.5.

A comparison of the filtered water from ten filter plants indicate:

- Particle counts ranged from 13 to 77 000 particles/mℓ with median 440 particles/mℓ.
- Particle volume ranged from 0.0022 to 2.4 mm^3/ℓ with median 0.056 mm^3/ℓ.
- The volume-averaged diameter ranged from 3 to 33 µm with median 13 µm.
- Turbidity ranged from 0.09 to 2.9 NTU with median 0.25 NTU.
- The pH ranged from 6.3 to 9.6 with median 8.6.

A comparison between the raw water quality and the filtered water quality indicate:

- The significant differences in raw water particles size, perhaps surprisingly, persist throughout the treatment process
- The log removals, whether expressed in terms of turbidity, particle counts or particle volume, are approximately the same.

The results reported in this chapter do not bring conclusive results to any one specific question, but is meant to demonstrate how particle counting opens an entirely new door to the characterisation of particle suspensions and the assessment of treatment performance. Not only can operators assess how good their plant performance is, but they can also gain more insight into *why* the performance may be non-optimal at certain times. There are numerous leads to further research following from the survey presented above, of which two have already started within the RAU Water Research Group:

- Correlating the treatment performance with more operational variables to determine for example whether flocculation pH and particle size play a determining role in particle removal.
- Assessing not only the overall treatment performance, but also looking at the contribution of individual unit processes within each treatment plant.
- Extending the survey to include a broader range of raw water types.
- Repeating the tests at the plants discussed above, but at a time of higher suspended solids during summer.

Chapter 8

THE RELATIONSHIP BETWEEN PARTICLE SIZE, AND *CRYPTOSPORIDIUM* OOCYSTS AND *GIARDIA* CYSTS IN FILTERED WATERS FROM WATER TREATMENT PLANTS

8.1 Background

Most water treatment plants rely on the effective dosing of coagulants and flocculation to achieve particle removal by dissolved air flotation, sedimentation and rapid gravity filtration. Jar test procedures are commonly used as a means to establish an effective coagulant dose. Traditionally, a particulate measurement is made using either turbidity or suspended solids. Both are indirect measurements. Turbidity is the measure of the light scattering properties of particles in the water, whereas suspended solids is a measure of the mass of particulate matter present. If particle sizes or densities vary, these cannot be related to particle numbers.

Particle counting is becoming more popular worldwide as a more accurate means of measuring final water quality. Many studies have shown that *Giardia* and *Cryptosporidium* can be removed effectively if the process produces filtered water at or below a specific turbidity value. This varies however depending on the process, the raw water quality and the type of coagulant used. However, further work has shown that *Giardia*- and *Cryptosporidium*-sized particles may well pass through the treatment process with no observed increase in the filtered water turbidity.

The objective of this study is to determine whether the sensitivity and accuracy of particle counters is sufficient to be able to detect *Cryptosporidium* and *Giardia* in the filtered water from water treatment works.

It is important to stress at the outset of this study that the number of other particles occurring in natural water samples will be far in excess of the number of oocysts and cysts measured. Particle counters can not be used as a reliable monitoring mechanism for the cysts and oocysts in natural waters. The aim of this study is purely to determine the efficiency and accuracy with which *Cryptosporidium* oocysts and *Giardia* cysts can be measured using particle counting techniques.

8.2 Literature Study

Giardia and *Cryptosporidium* are waterborne protozoan parasites, which are associated with diarrhoeal illness in many parts of the world (Fayer & Unger, 1986, quoted by Kfir et al. 1995). *Cryptosporidium* is an oval shaped parasite and appears to cause disease in man and livestock (Badenoch, 1990). *Cryptosporidium* has a complicated life cycle, which goes through many forms, the most important being a 4-6 µm diameter oocyst, which contains the infective sporozoites. *Cryptosporidium* oocysts are released from infected hosts at a rate of up to one million per day (Daniel, 1995). Oocysts are the dormant form that can survive adverse environmental conditions and are responsible for disease transmission from host to host (Badenoch, 1990).

The life cycle of *Giardia* is simple with two morphological forms, namely non-invasive trophozoite and an environmentally resistant cyst which is excreted in the faeces (Smith, Robertson & Ongerth, 1995). The cysts are much larger than oocysts and are 8-12 µm in length and 7-10 µm in width. Transmission routes for both organisms are by the resistant oocysts or cysts and can include animal-to-person, person-to-person and water-to-person.

The use of particle monitors is a simple, cost effective and suitable technique to measure particle removal for particles in the *Cryptosporidium* and *Giardia* size range in settled and final treated water (Veal & Riebow 1994) .Particle monitors can be used for continuous measurement of filter performance. Conventional turbidity monitoring is not sufficiently sensitive for monitoring the removal of *Cryptosporidium* and *Giardia* (Gregory, 1994).

However, it should not be assumed that particle counters will be able to detect *Cryptosporidium* and *Giardia* directly. One reason may be that their concentration may be too low for reliable detection, bearing in mind that there will be many other particles in a similar size range. Another factor is the effect of particle properties (e.g. refractive index) on the optical response of particles. It was shown (Lewis 1991 reported by Gregory 1994) that *Giardia* cysts are counted in the 1-5 µm range even though their true size is greater than 10 µm. The reason for this is that particle counters are generally calibrated with latex spheres of a know size which have a fairly high refractive index and, hence, scatter (or block) more light than biological particles of the same size. The author also observed that *Cryptosporidium,* which is around 4 -6 µm, have the same scattering power as 2 µm latex particles.

The overall level of particulate contamination in treated water should be maintained as low as possible to effectively safeguard against the presence of oocysts and other pathogens (Gregory

1994). The monitoring technique therefore needs to be selected carefully. There is evidence that conventional turbidity measurement is not sufficiently sensitive to particles in the oocyst size range. Methods based on particle counting should give more reliable on-line monitoring.

In the USA the drinking water regulations have a maximum contaminant level goal (MCLG) of zero *G lamblia*, with a maximum contaminant level (MCL) of 99.9% (3 log) removal or inactivation of cysts. This was promulgated in 1989 in the Surface Water Treatment Rule (EPA Current Drinking Water Standards, 1998). The Interim Enhanced Surface Water Treatment Rule introduced in November 1998 (EPA Microbial and Disinfection Byproduct Rules 1998) adds the following key additions which apply to all systems using surface water or groundwater under the influence of surface waters, that serve 10000 or more persons:

- MCLG of zero for *Cryptosporidium*; a 2 log removal requirement for systems that filter;
- strengthened combined filter effluent turbidity performance standards with individual filter turbidity monitoring provisions;
- disinfection profiling and benchmarking provisions (to ensure microbial control and minimising disinfection by-products);
- requirement introduced for covers to be placed on new treated water reservoirs
- sanitary surveys to be conducted on all surface water systems regardless of size.

In South Africa the South African Bureau of Standards specification (1999) specifies that *Cryptosporidium* and *Giardia* should not be detected in 95% of samples analysed with a maximum of 1 count per 100 ℓ and 10 counts per 100 ℓ for the remaining 4% and 1% of sample analysed respectively. The Department of Water Affairs and Forestry guidelines (1993), for domestic and full contact recreational use, list *Cryptosporidium* oocyst and *Giardia* cyst levels of less than one per 10 ℓ. Rand Water in their 1992 potable water criteria list recommended limits of zero cysts and oocyst per two ℓ sample, with a low risk attached to five *Giardia* cysts per sample. Similar levels were given for *Giardia* in the 1994 Department of Health guidelines for drinking water. *Cryptosporidium* levels were not specified.

8.3 Materials and Methods

A standard addition technique was used to examine the accuracy of the particle counters to detect a spike of concentrated particles when injected into the water sample. A sample of water containing as few particles as possible (typically final filtered water) was used as a blank, resulting in a baseline count. The method of injection and data recording described in Paragraph 10.3.2 was used to determine the number of particles in the actual spike. Three separate standards were used to assess the accuracy of the particle counter viz. NIST traceable latex spheres, standards containing *Cryptosporidium* and *Giardia* and a NIST reference material 2806 “Medium Test Dust”.

8.3.1 Particle Counting Instrument

An ARTI on-line particle counter supplied by Optima Hydraulics was used for the study. The instrument was connected to a constant head flow control device which regulated the flow of water through the sensor. The particle counter was calibrated before delivery and connected to a computer that collected data every minute from the sensor. The flow through the sensor was maintained at 100 mℓ per minute.

8.3.2 Standard Reference Materials

Three standard reference materials were used during the experiments.

EZY-CALä Particle Size Standards – consist of suspensions of certified particle size standards in a range of diameters from 2 to 70 µm. The microspheres are packaged in water at a concentration of 2 000 particles /mℓ. A magnetic stirrer bar is included in each bottle for clean, convenient and direct sampling by optical particle counters. The particles are composed of polystyrene or polystyrene divinyl benzene. The aqueous suspension medium contains a combination of dispersing agents which helps to keep the particles from clumping or sticking to flow surfaces in particle counters.

***Cryptosporidium* and *Giardia* Standards** – Samples of concentrated *Cryptosporidium* and *Giardia* were purchased from USA. These are commonly used as samples for confirming the detection and analyses in the Analytical Services Laboratory at Umgeni Water. The samples were diluted in the Umgeni Water Laboratory to approximately 10 000 per mℓ.

Standard Reference Material® 2806 - consists normally of a poly-disperse, irregularly-shaped mineral dust suspended in approximately 400 mℓ of hydraulic fluid at a normal concentration of 2.8 mg/ℓ. SRM is intended for use in the calibration of instrument response to Medium Dust suspended in hydraulic fluid. The dust was obtained as a dry powder and was made up in water at a concentration of 2.8 mg/ℓ.

8.3.3 Standard Addition Technique

Water is fed to the particle counter through 6 mm nylon or Teflon tubing. An injection fitting was manufactured and inserted in the tubing before the sensor. The injection fitting comprised a T-piece where the branch was sealed using a Teflon coated rubber septum through which a sample of standard solution could be injected (Figure 10.3.3). A "Hamilton" accurately calibrated syringe was used to measure the volume of standard solution injected into the water. A "50 step" injection device was used to slowly inject the solution into the water in 50 equal components over the desired time. (i.e a 5 mℓ syringe was used and each injection comprised 0.1 mℓ; alternatively when a 10 mℓ syringe was used each incremental volume injected was 0.2 mℓ).

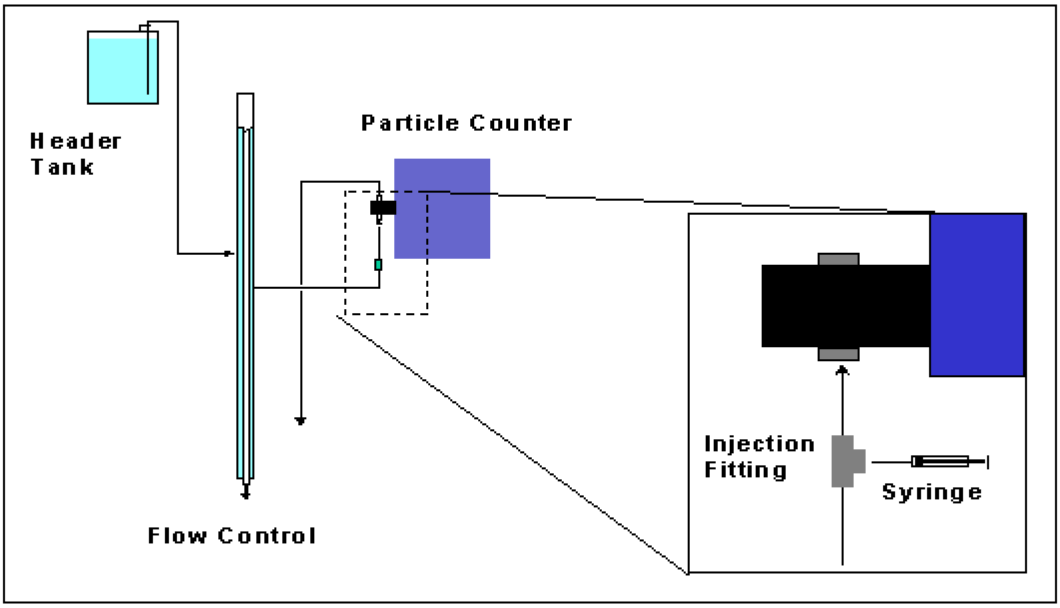

Figure 8.3.3(a) : Injection of Standard Solutions for Calibration Verification

Prior to the addition of standard solution, the header tank was filled with filtered water with as low a particle count as possible. Flow to the particle counter was started and the counts were allowed to stabilise. This measurement then formed the baseline of background counts in the water. The particle counter was set up to count for a minute and display the count every minute. The flow was set to measure 100 mℓ/min.

Once the count had stabilised a specific volume of standard solution was injected. Normally 0,6 mℓ per minute. (i.e. one injection on the stepper every 10 seconds. Over a period of between 3 and 6 minutes the fill sample of standard would be added and the background measurement of the filtered water would return to a similar level as that before the trial. Figure 8.3.3b.

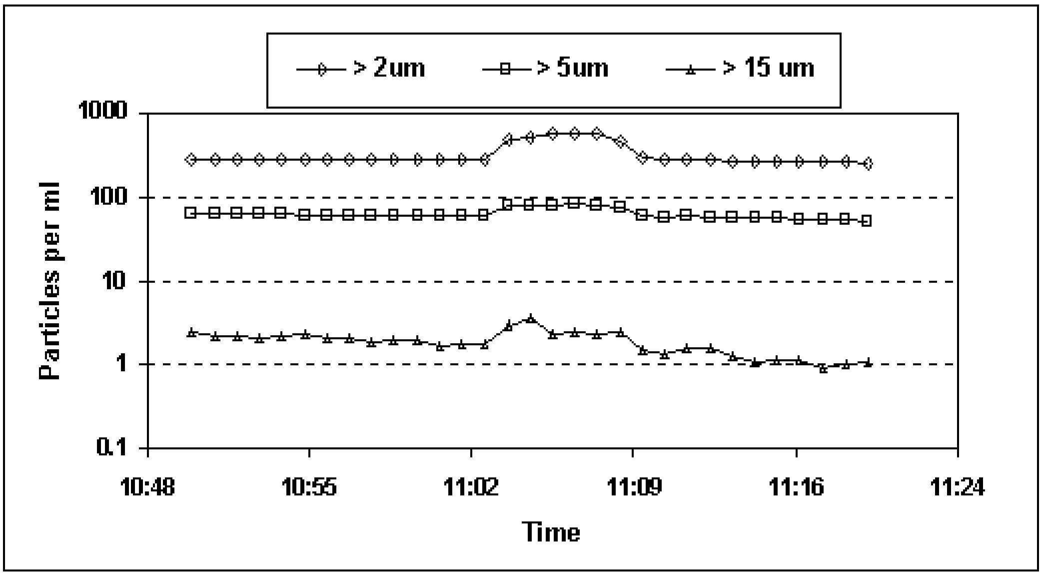

Figure 8.3.3(b) : Particle Counter Response to the Injection of Particles

8.3.4 *Data Analysis*

The use of this technique is to establish whether the particle counter is able to detect particles in the water in specific size ranges. The particle counter was set up to detect sizes greater than the specific channel size. (i.e. >2 µm; >3 µm; >5 µm; >7 µm; >10 µm; >15 µm; >25 µm; and >50 µm;)

The cumulative count SN_i is measured for each time period t_x. Before the injection the particle count is recorded as the background count SN_{ib}. The background count is subtracted from each individual measurement, resulting in the actual number of particles measured for each individual time period.

$$\sum N_i(t_x) = \sum N_i - \sum N_{ib} \text{ (for each time interval)} \qquad \textit{8.3.4(a)}$$

The total particles added are then calculated by totalling all the individual increments for each time period. Thereafter, the Incremental or Discrete Counts can be calculated as shown in Paragraph 5.4.3. The particle concentration of the standard used can be determined by dividing by the volume of standard sample injected.

8.4 Results and Discussion

The experiments were performed in three separate phases. Firstly, an attempt was made to determine the accuracy of the particle counters using National Institute of Standards & Technology (NIST) traceable monodisperse latex spheres. Latex spheres of nominal diameter 5 µm and 10 µm were obtained from Duke Scientific Corporation. (Actual sizes were 4.991 µm ± 0.035 µm and 9.975 µm ± 0.061 µm). Some form of reliability was obtained, but the latex particles have different optical properties to *Cryptosporidium* and *Giardia* and the experiments were repeated using non-viable *Cryptosporidium* and *Giardia* particles in solution. Following discussions with ARTI (manufacturers of the particle counter) a sample of Mineral Test Dust (MTD) was obtained and the results of the particle counts obtained were compared to results published from participating laboratories in a "round robin" interlaboratory exercise.

8.4.1 Spiking with NIST Latex Spheres

When performing the experiments, it was expected that the instrument should detect half the particles above the actual size and half below the actual size as described in Paragraph 4.3.2. The instruments were set-up differently from normal operation to be able to detect different size particles in a narrower range on either side of the expected mean. Figure 8.4.1 shows a consistency in the calibration of the ARTI instrument as opposed to the MetOne instrument when this technique is used. It was also noticed that when comparing data from the two instruments, the numbers were comparable for particle sizes down to 7 µm, but below this the MetOne instrument tended to measure higher counts than the ARTI instrument.

Figure 8.4.1 shows that the particle counters measured particles in the spike both above and below the actual size as expected. The ARTI instrument appeared to measure more consistently on either side of the mean measuring 41% and 59% of the standard particles in the size ranges above the actual value. During preparation and injection of particle counting standard, the entrainment of bubbles as well as the possibility of bubble formation in the sample tubing at the point of injection could result in inaccurate counts. Specific care should be taken during injection to minimise any turbulence which may result and thereby decrease the accuracy of the particle count detected by the instrument.

Results from the Met-One instrument indicate that more of the particles added to the water were detected in a size range above the actual size. This may indicate that the electronic calibration of the instrument is incorrect and may need re-calibrating.

Although only 5mℓ of sample was injected during the experiments (NIST standard containing 2 000 particles/mℓ; i.e. 10 000 particles) the number of particles detected by the particle counters exceeded this. The recovery of particles was poor and other disturbances (e.g. air bubbles or turbulence during injection) resulted in this technique not being totally effective. Ideally, both the particle size and count should be accurate to verify calibration of the instrument. This procedure only serves to give an indication of whether the calibration is acceptable or not.

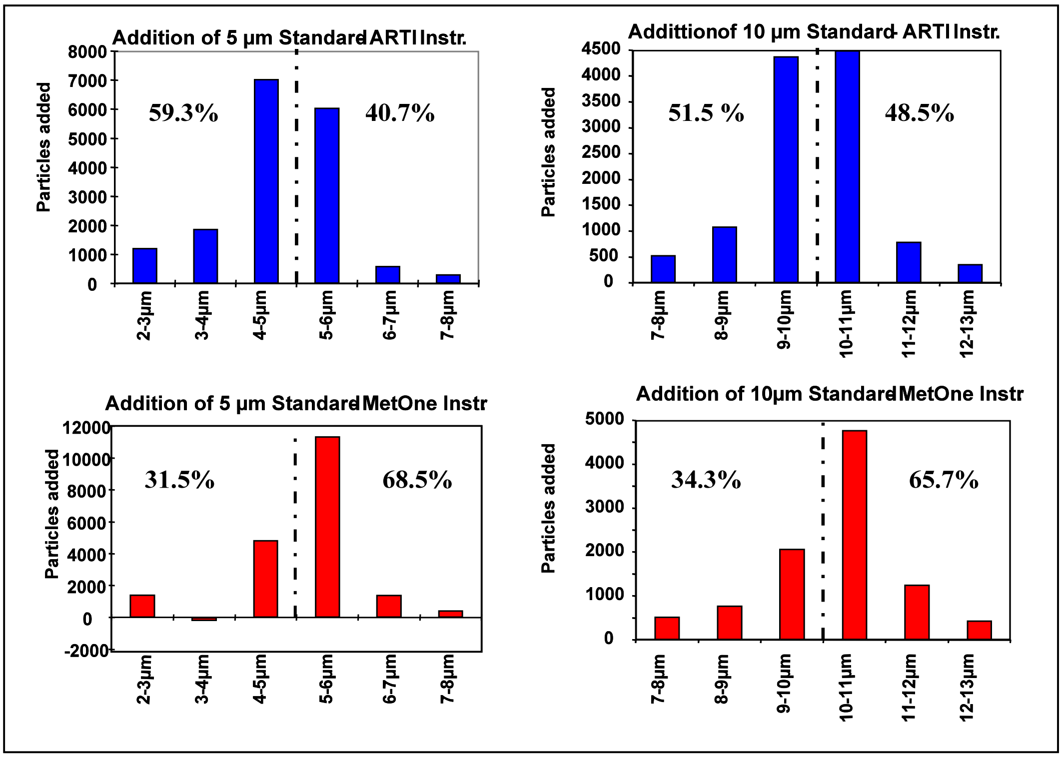

Figure 8.4.1 : Results of a Calibration Exercise Using Two Different Particle Size Standards.

8.4.2 Addition of *Cryptosporidium* Oocysts and *Giardia* Cysts

The *Cryptosporidium* standard was diluted by the Umgeni Laboratory with Ultrapure water, producing a solution containing approximately 10 000 particles/mℓ. During each injection a total volume of 3.6 mℓ was injected. The data obtained from the particle counter indicated that additional particles were added in significant quantities that the total particles/mℓ in the filtered water increased from approximately 225 particles/mℓ to over 400 per mℓ. The data was then analysed as described in Paragraph 8.3.4.

The assumption is made that the baseline (or the particle count in the filtered water) is constant throughout the experiment. The total number of particles in each bin size can be calculated and is shown in Figure 8.4.2a. The average concentration of standard suspension particles added was 34 616 particles/mℓ. This is significantly higher than the expected concentration of 10 000 particles/mℓ. Most of the particles (86%) were detected in the size range 2 to 5 µm and a further 11 % were detected in the size range 5 to 10 µm. This indicates that the particle counter may not necessarily detect *Cryptosporidium* accurately in the expected channels between 4 and 8 µm, but the presence of *Cryptosporidium* may well be detected in the total particle count (i.e. >2 µm).

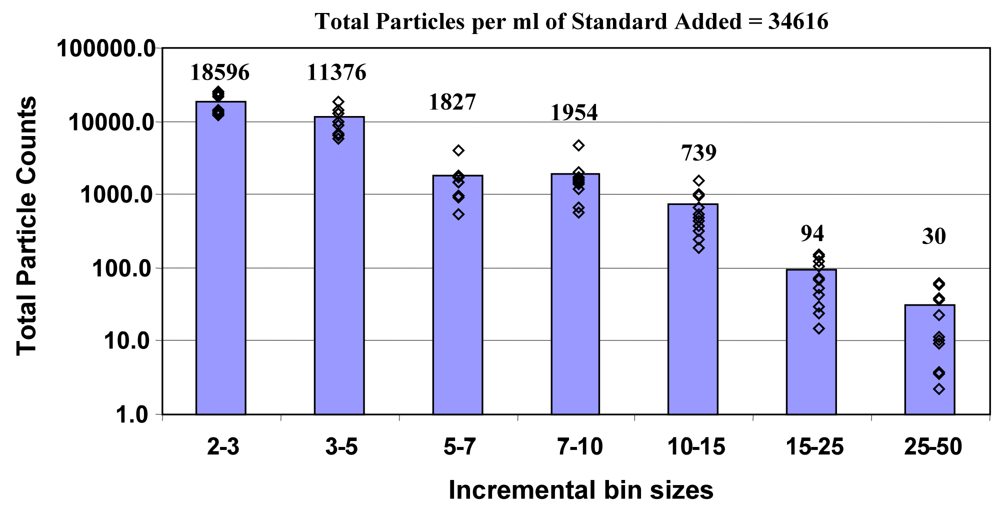

Figure 8.4.2(a) : Detection of *Cryptosporidium* Oocysts by Injection

A sample of standard containing *Giardia* particles was prepared in the same way as the *Cryptosporidium* standard producing a solution containing approximately 10 000 particles/mℓ. During each injection, a total volume of 7.2 mℓ was injected. The response of the particle counter to the injected sample was significant, showing an increase in the particles per mℓ in the filtered water from approximately 40 per mℓ to over 270 per mℓ. The data was then analysed as described in Paragraph 8.3.4.

During these experiments a water filtered through an ultra filter membrane was used. The baseline counts of 40 per mℓ were significantly lower than the baseline used for the *Cryptosporidium* experiments. The total number of particles in each “Incremental Bin Size” was calculated and is shown in Figure 8.4.2b. The total number of particles added on average per mℓ of standard solution was 33 071 particles/mℓ. As with the addition of *Cryptosporidium*, this is significantly higher than the expected concentration of 10 000 particles/mℓ. Most of the particles (89%) were detected in the size range 3 to 10 µm. This is lower than the expected range of 8 to 12 µm. This may be as a result of the shape of *Giardia* being elongated with a width in the range of 7 to 10 µm.

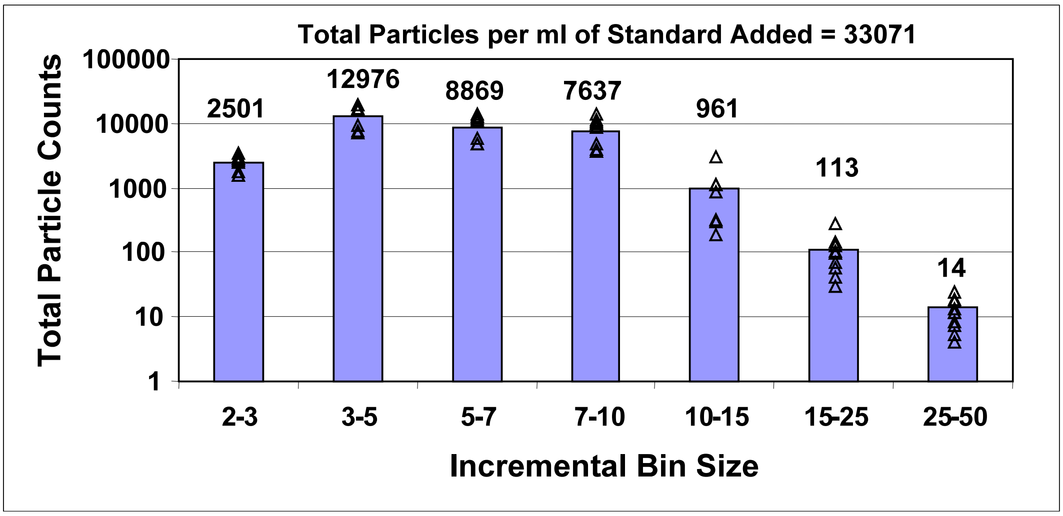

Figure 8.4.2(b) : Detection of *Giardia* Cysts by Injection

This shows however that there is a reasonable chance that should there be *Giardia* particles in the water the particle counter is like to be able to count the particles and register their presence.

8.4.3 Spiking the Water with Standard Reference Material 2806

A solution of Standard Reference Material was made up in Ultrapure Water as describe in the product brochure from the National Institute of Standards & Technology (NIST). The sample was mixed and injected in a similar manner to the procedure described in Paragraph 8.3.4. The data from the particle counter was refined and the Cumulative Counts are shown in Table 8.4.3(a).

Table 8.4.3(a) : Cumulative Counts for the Injection of Medium Test Dust SRM 2806 (Number of Tests = 22)

	Particle Concentration (Counts/mℓ)							
	>2µm	>3µm	>5µm	>7µm	>10µm	>15µm	>25µm	>50µm
Average	5 366	3 864	1 294	855	471	282	167	80
Std Dev	626	435	224	146	122	73	46	22

An example of a laboratory round robin exercise is detailed elsewhere (National Institute of Standards & Technology, 1997). Three companies utilizing five instruments and five different sensors were evaluated to compare the counter and sensor response. The instruments were all calibrated in accordance with the existing ISO 4402:1991 Standard. Table 8.4.3(b) shows the results of the respective instruments.

Table 8.4.3(b) : **Results of Round Robin Comparison (Extracted from Reference Brochure NIST – Standard Reference Material 2806)**

	Counts/mℓ Greater Than Indicated Size						
Particle Counter	**>1µm**	**>2µm**	**>5µm**	**>7µm**	**>10µm**	**>15µm**	**>20µm**
Laboratory 1	5 624	4 429	2 039	1 215	576	191	77.9
Laboratory 2	5 351	4 482	1 977	1 139	521	188	88.6
Laboratory 3	-----	4 208	1 898	1 116	525	182	82.1
Laboratory 4	5 313	-----	1 993	-----	541	182	82.5
Laboratory 5	5 350	-----	1 992	-----	517	174	80.9

It should be noted that the particle counters used in the round robin study were specifically used for counting particles in hydraulic oil samples and not water. It may not be totally accurate to compare the numbers, none-the-less the particle numbers indicated in Table 8.3.4(a) are of similar magnitude to the numbers obtained during the participating laboratories. The results are sufficiently similar to indicate that this type of reference material could be used as a standard for confirmation of the calibration of an instrument on a plant.

8.5 Conclusions and Recommendations

The method standard injection can be used with limited accuracy to determine the accuracy of on-line particle counters. Once the spike is injected, the data should be extracted and manipulated to indicate the accuracy within a particular size range. The particle counters used during this trial showed that the instruments were sensitive to particles in the size range of 5 to 10 µm respectively, but a count recovery could not be obtained with sufficient certainty.

When *Cryptosporidium* and *Giardia* were added to the filtered water, a response was obtained which measured *Cryptosporidium* mainly in the size range 2 to 5 µm and which measured *Giardia* in the size range 3 to10 µm. This is encouraging, but it should be remembered that *Cryptosporidium* and *Giardia* are likely not to be in the water in a high enough concentration for the instrument to be able to detect spikes in the counts of these particles. It is possible that should there be a sudden increase in particles in the respective size ranges that there may also be *Cryptosporidium* and *Giardia* present in the water.

A comparison of the detection of a Standard reference test dust using the on-line particle counters showed similar results to those obtained from other laboratories using different instruments for the measurement of particles in hydraulic oils. The results indicate that when a reference dust is used for water particle counters, an adequate response can be obtained and that this could be used as an alternative to the monodisperse latex spheres used commonly for calibration checking of the instruments.

8.6 References

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Chapter 9

THE EFFECTS OF PRE-TREATMENT ON THE TURBIDITY AND PARTICLE CONCENTRATION OF FILTERED WATER

9.1 Background

The design and operation of flocculation processes and the evaluation thereof have been based mainly on recommended G values and the selection of a suitable coagulant based on laboratory scale jar test procedures. In more specific cases a history of the raw water quality as well as results from pilot scale experiments are used to finalise a suitable design. A suitable design will normally include pre-treatment processes such as ozonation, floc aid (bentonite) addition, pH adjustment, pre-chlorination or the use of dual coagulants.

The floc shape and size can be affected by pre-treatment conditions and, as such, the turbidity of a sedimentation effluent or a filtered water quality may not fully represent the behaviour and dynamics of particles in the process. Time delays through the process may also prevent immediate response to changes.

In this chapter the experiences at the Wiggins Water Treatment Plant are reported to indicate how particle counters can identify non-optimum operating conditions and to demonstrate the effect pre-treatment has on the filtered water particle counts and turbidity.

9.2 Literature Study

The design of a flocculation vessel for Dissolved Air Flotation (DAF) was studied by Valade et al. (1996), who found that the flocculation conditions affected the particle counts after DAF and also contributed to significant improvement in filtered water turbidity. Valade compared flocculation conditions at low and high G values and also for different arrangements of impellers. It was found that the coagulant type and flocculation time had the greatest effect on filtration performance. A 5-min flocculation time produced generally a lower filtered water turbidity than 20-min flocculation, even though the particle count was slightly higher. However ,there was a more rapid headloss development on the filters.

The effect that ozone and peroxone have on the performance of direct filtration showed differences in head loss development and filter breakthrough (Tobiason et al., 1992). In general the turbidity and particle count results from these studies indicate that pre-oxidation slightly improves the filtered water quality. Reduced particle counts have also been reported by others (Georgeson, 1988; Chang and Singer, 1991 and Dunn et al.,1988). The post oxidation particle counting suggests that the oxidants induced coagulation, the so-called micro-flocculation effect. The results could not explain the observed benefits of delayed filter breakthrough or reduced headloss development.

9.3 Materials and Methods

9.3.1 Particle Counting Instrument

An ARTI WPC (Water Particle Counter) on-line instrument supplied by Fluid Systems Partners GmbH was installed at the "City Outlet" sampling point on the pipeline from the on-site storage reservoir at Wiggins to the City of Durban. The instrument was set up to monitor particles in eight size bins and was connected to a PC loaded with the "Aquarius" software supplied by Fluid System Partners.

9.3.2 Data Analysis

Data were collected by the Aquarius software and stored on the PC. Graphical display of the continuous particle counts enabled the on-line observation of particle counts. A manual report of data was generated using the software. Data were extracted to a text file and then imported into Microsoft (TM) Excel for graphing and presentation. The software logs the data every minute but allows reduction of the database whilst reporting. The operator is able to extract a report of average particle counts every 5, 10, or 15 minutes. When analysing data from the on-line measurement of particle counts, averaged data every 5 minutes was extracted for reporting in this project.

9.4 Results and Discussion

Particle counters were installed at the Wiggins Water Treatment Plant to observe more closely the quality of the water delivered to the City of Durban. This followed discussions within Umgeni Water as to whether the water treatment processes are adequate for the removal of *Cryptosporidium* at Umgeni Water. Further work is planned to investigate the headloss development on a filter at the plant and to compare the performance of the plant for periods when ozonation is operating to periods when ozonation is off-line. At the same time the particle counter will be compared to the latest "low-range" turbidity meters available recently in South Africa.

9.4.1 Filter Performance at the Wiggins Water Treatment Plant

During the monitoring period the raw water turbidity was less that 5 NTU and a coagulant aid (bentonite) was dosed into the raw water to assist with flocculation.

The total particle count > 2 µm was initially found to be approximately 20 particles per mℓ. The final water turbidity at the time was <0,1 NTU. On 12 December 1999, the bentonite dose was increased to 1.9 mg/ℓ to improve the results for the floc formation tests. This obviously influenced the coagulation and flocculation conditions and resulted in an increase in the final water particle count. An increase in turbidity was not observed. The plant performance improved however and between 13 December and 16 December the particle count in the final water of particles > 2 µm improved to approximately 10 particles per mℓ.

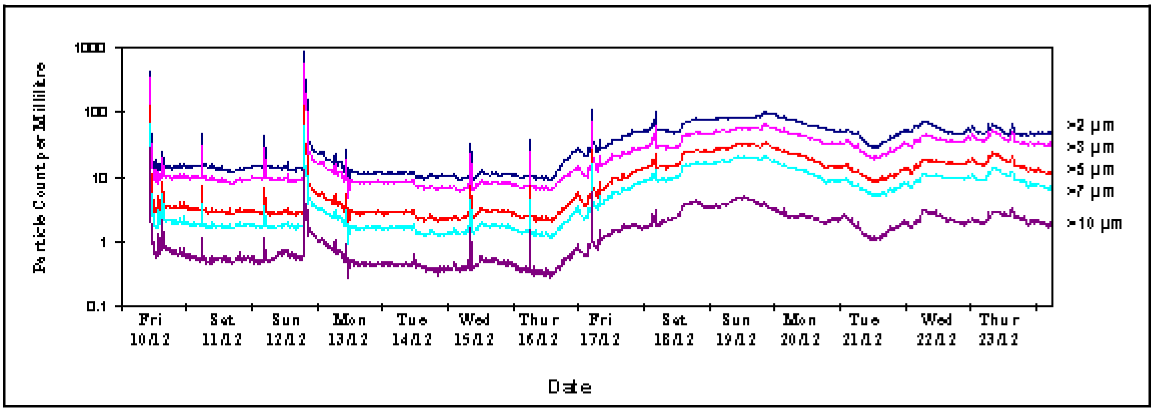

Figure 9.4.1: Operating data from the Umgeni Water Wiggins Water Treatment Works

On 16 December 1999 the bentonite dose was decreased from 1.9 mg/ℓ to 1.5 mg/ℓ. The particle count began to rise. On further investigation, problems with the effective control of the coagulant dose were found. Although the streaming current detectors at the plant are maintained according to the manufacturer's instructions, occasional additional calibration and maintenance are required. The signal drifts from time to time and regular coagulant dosage rate confirmation tests are performed by operating staff to determine the actual coagulant dose. When the signal drifts and the operating tests do not detect an abnormally low or high coagulant dose the performance of the plant can become non-optimal for particle removal. During the observed period the turbidity increased to 0.13 NTU and the final water particle count increased 10-fold to approximately 120 particles per mℓ (particles > 2 µm).

During the period of monitoring, it was accepted that the plant could achieve a consistent final water total particle count (particles > 2 µm) of less than 50 per mℓ.

9.4.2 *The Effect of Pre-ozonation on Filtered Water Quality*

An investigation was initiated at the Wiggins Water Treatment Plant in Durban to assess the effects of ozone on the quality of filtered water with regard to particle counts. An on-line particle counter was installed at the outlet of a single filter and the filter was operated and backwashed manually during this period. After three weeks of operation with pre-ozonation, the ozonation was stopped and the plant was operated and optimised without ozone addition. During the investigation, the filter was operated for as long as possible in an attempt to identify whether particle breakthrough might occur.

9.4.2.1 Operation with Pre-ozonation

Initially, the particle counts were relatively high, and remained high after backwashing the filter on 22 November 2000. Once the particle counting sensor had been cleaned on 24 November, the particle counts reduced and remained consistent for the remainder of the investigation. This emphasises the need to regularly check the calibration of the particle counter and at least check the zero count (with no flow) as described in paragraph 4.3.1.3.

The variation in plant operating conditions is shown in Table 9.4.2. Although the flow varied between 80 and 190 Mℓ/d and the filtration rate may be affected by other filters being taken off-line for backwashing, the particle count in the filtered water remained relatively constant. The filtration rates calculated between 1.6 and 4.1 m/hr are low when compared to other rapid gravity installations. Under these operating conditions, breakthrough of particles from the filter was not observed after 10 days of continuous operation (between 27 November and 7 December). It should however be noted that the plant was being operated at under half its design capacity.

Table 9.4.2 : Plant Operating Conditions During the Investigation

Determinant	Minimum	Maximum	Average
Raw Water Turbidity (NTU)	1.25	15.4	3.7
Raw Water pH	6.73	8.32	-
Pre-ozone Dose (mg/ℓ) [1]	1.1	2.0	1.5
Bentonite Dose (mg/ℓ) [2]	2.0	2.0	2.0
Lime Dose (mg/ℓ)	0.9	1.9	1.2
Pre-clarifier pH	7.33	8.54	-
Coagulant Dose (mg/ℓ) (Blended Polymeric Coagulant DADMAC / Polyaluminium Chloride)	1.5	3.2	2.1
Post-clarifier Turbidity (NTU)	0.17	1.21	0.64
Post-clarifier pH	7.63	8.52	-
Plant Flow rate (Ml/d)	80	190	140
Filtration Rate (m/hr)	1.6	4.1[3]	2.9

[1] During investigation using pre-ozone

[2] During investigation without pre-ozone

[3] Calculated with one other filter off-line for backwashing

A daily pattern of increase and decrease in particle count was noticed. It is unlikely that this pattern can be linked to the hydraulic conditions under which the filters were operating. Filter washes are controlled by PLC and take place throughout the day, and not only during the day shift. Also, the patterns have a recurrence period of 12 to 24 hours, whereas one filter wash could only affect the filtration plant for 1 to 2 hours. A more significant change in filtration rate during the experiment was effected by the variation of flow from 80 Mℓ/d to 190 Mℓ/d and this also did not affect the particle count at all.

The only variable that the daily pattern could possibly be related to is water temperature. Under normal operating conditions, the water temperature in the impoundment will increase during the day and decrease again at night. The effect of temperature is noticed on the plant approximately 10 hours later due to the time it takes to transport the water to the treatment plant. The colder water temperatures normally reach the plant between 10 am and 12 pm. There appears to be a corresponding increase in particle count reaching a maximum at approximately 11 am each day. The performance of the treatment plant is affected either by a reduction in efficiency of coagulation or a carryover of small particles from the clarifiers and an increased breakthrough of small particles from the sand filter. The viscosity of water (which increases with a reduction in temperature) can result in increased headloss through filters as well as poorer settling characteristics of flocs in clarifiers. The turbidity meters after the clarifiers were not able to detect a significant change in turbidity. Even though the particle counter detected a variation in particle count, the total counts greater than 2 micron varied between 9 and 15 per mℓ.

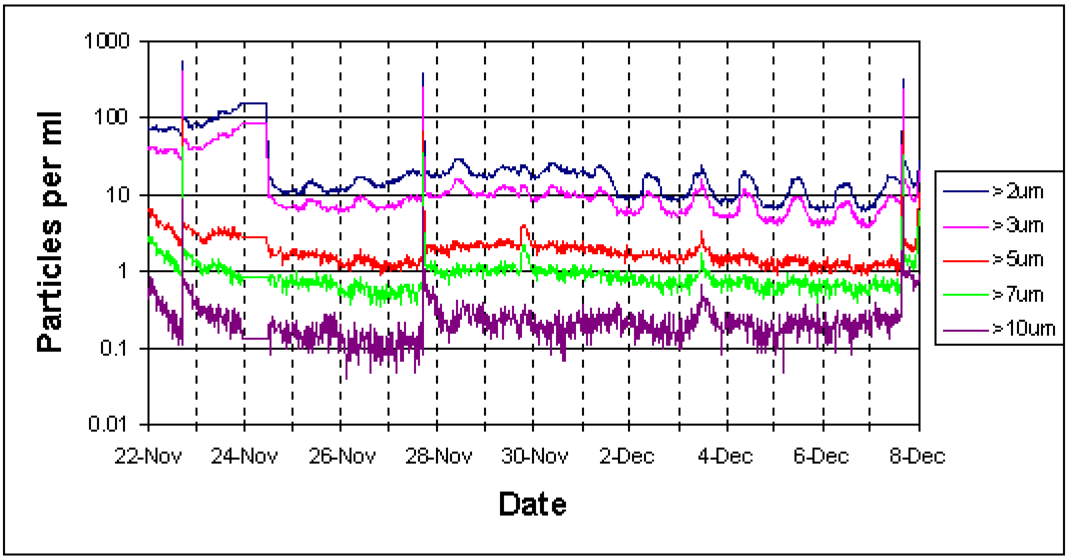

Figure 9.4.2(a) : Particle Counts of Filtered Water at the Wiggins Water Treatment Works while Operating with Pre-ozone.

9.4.2.2 Operation Without Pre-ozonation

On 12 December the pre-ozone dosing at Wiggins Water Treatment Plant was discontinued and a new investigation was initiated to assess the performance of the plant for particle removal. A sudden increase in particle count was observed on 14 December where particles > 2 micron increased to 80 per mℓ. On 15 December the floc formation tests on the plant started showing deterioration in the settled and filtered water turbidities. Bentonite dosing was started immediately on the plant to assist in improving the coagulation and flocculation processes.

After closer investigation, the streaming current set-point for coagulation control required optimisation, but due to very low turbidity in the water, this could only be effectively achieved using bentonite as a coagulant aid. During the optimisation process, the particle count started improving.

The operation with bentonite required higher doses of coagulant. On 19 December the coagulant dose was decreased from 2.4 mg/ℓ to 2.0 mg/ℓ in order to reduce chemical operating costs on the plant. This resulted in a significant increase in particle count although the final water turbidity remained below 0.05 NTU. Further optimisation continued and the streaming current set-point was adjusted to achieve a coagulant dose of 2.2 mg/ℓ. This was found to be adequate to reduce the particle count to less than 10 particles per mℓ.

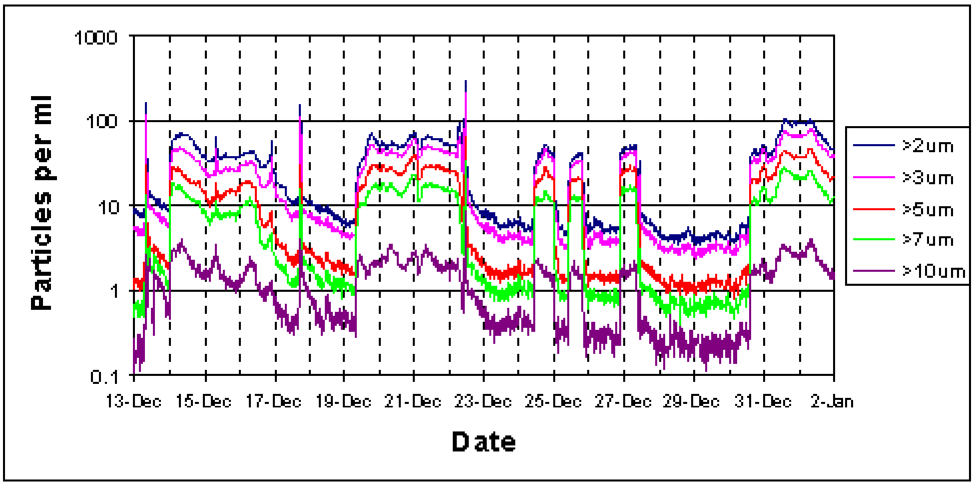

Figure 9.4.2(b) : Particle Counts of the Filtered Water at the Wiggins Water Treatment Works while Operating without Pre-ozone.

In the period between 23 December and 30 December 2000, the streaming current set-point for coagulant dosing was kept constant, yet variations in the particle count were still observed. The coagulant dose is automated to achieve a streaming current goal after coagulation based on the streaming current set-point. There are a number of reasons as to why the particle removal had been affected during this period :

S Raw water particle characteristics may have changed, which resulted in a larger or lesser dose of coagulant to achieve the required streaming current set-point.

S The streaming current sensor may have gradually become fouled, resulting in a marginally incorrect signal and, hence, a larger or lesser coagulant dose, thereby affecting particle removal, or

S Other plant and water quality conditions may have contributed to variation in the conditions at coagulation. The final water pH was controlled by increasing and decreasing the lime dose prior to coagulation. During periods where the lime dose was higher, the particle removal was found to be poor. This can be explained by considering that the point of zero charge on the surface of particles is dependent on the pH. The required coagulant dose, theoretically, therefore also changes with varying pH and, whilst the plant is operated at a constant streaming current set-point, the automated coagulant dose will change with varying pH.

It was found that for particle removal, the plant was very much more sensitive to small changes in operating and environmental conditions whilst operating without ozone. The investigation showed that with continual optimisation, the same or better particle counts could be achieved without pre-ozonation as previously obtained whilst dosing ozone.

9.4.3 Comparison of Particle Counts to Turbidity Measurement

During the on-line particle investigation at the Wiggins Water Treatment Plant, a "low-range" Hach 660 turbidity meter was installed in parallel to the particle counter to compare the sensitivity of this instrument to variations in particle count in the filtered water.

Figures 9.4.3a and 9.4.3b show that changes in turbidity are detected during periods where there is a more significant change in particle count. Typically, at the particle count and turbidity levels experienced during the investigation, a 10-fold increase in total particle count may be detected as a 2-fold increase in turbidity.

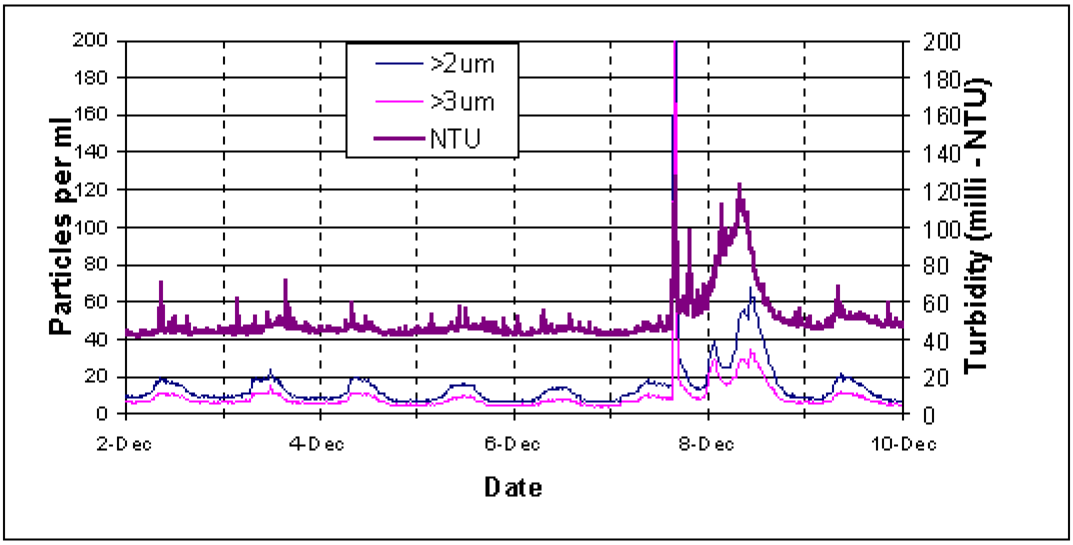

Figure 9.4.3(a) : Comparison of Particle Counts with Milli-NTU Turbidity Measurements While Using Pre-ozone

During the period when ozone was being dosed (Figure 9.4.3a), a greater response to turbidity was observed than whilst the plant was being operated without ozone (Figure 9.4.3b). This may be due to ozone having an affect on the surface properties of the particles. There is not a true relationship between particle count and turbidity. Figure 9.4.3a shows a turbidity of 100 mNTU (0.1NTU) and a total particle count > 2 µm of only 70 particles per mℓ, whereas in Figure 9.4.3b the turbidity hardly changed when there was a significant change in particle count from 5 particles per mℓ to 100 particles per mℓ.

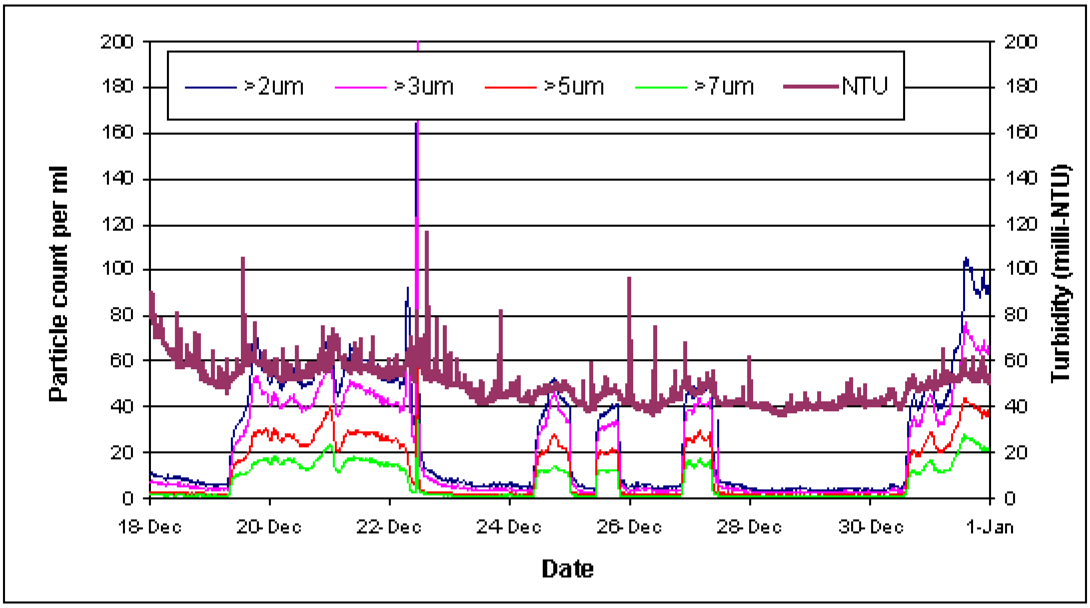

Figure 9.4.3(b) : Comparison of Particle Counts with Milli-NTU Turbidity Measurements While Not Using Pre-ozonation

The "low-range" turbidity instrument, although measuring turbidity in "milli-NTU" was not significantly more accurate than the normal range turbidity instrument, except that additional significant digits are provided by the instrument. (i.e. 42.38 mNTU vs 0.04 NTU).

The results indicated that not only was the particle count information more sensitive than turbidity measurement but far more information is obtained from a particle counter regarding the particle counts at different particle sizes. This can then be analysed further if required.

9.5 Conclusions and Recommendations

The following were concluded from this study:

- Whilst treating water with a low raw water turbidity, bentonite is sometimes required to improve the coagulation and flocculation of particles. The bentonite dose was found to have an impact on the quality of the filtered water. During treatment of water at the Wiggins Water Treatment Plant, it was possible under the prevailing raw water and plant operating conditions to reduce the total particle count >2micron to less than 10 particles per mℓ.

- Although regular backwashing of the sand filters is required to maintain the quality of the filtered water, it was found that particle breakthrough did not occur after 10 days of continuous operation. It should be noted that during this period the filters were being operated at a third of their design flow.

- The particle count in the filtered water was found to be affected not only by changing coagulant and bentonite dose, but by other operating parameters. The chemical and physical conditions at the point of coagulation will affect the particle characteristics and, therefore, the streaming current measurement after coagulation. As a result of changing conditions, the coagulant dosing control is affected. It was noted that small changes in pH possibly caused significant changes in particle count in the filtered water.

- Particle counting of low turbidity filtered waters was found to be more sensitive than "low-range" turbidity measurement. During the investigation, the filtered water turbidity remained consistently below 0.1NTU, whereas total particle counts >2 micron varied between 5 per mℓ and 100 per mℓ.

- The particle count after filtration was not found to be significantly affected by pre-ozonation when compared to operation without ozonation. Although the filtered particle count was more consistent and the quality of the water was more easily maintained using pre-ozonation, when the plant had been optimised for operation without ozone, the filtered water particle count achievable was found to be lower.

9.6 References

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Chapter 10

PARTICLE COUNTERS, ZETA POTENTIAL AND STREAMING CURRENT DETECTION AS CONTROL PARAMETERS FOR THE OPTIMISATION OF WATER QUALITY

10.1 Background

Most water treatment plants rely on the effective dosing of coagulants and flocculation to achieve particle removal by dissolved air flotation, sedimentation and rapid gravity filtration. Jar test procedures are commonly used as a means to establish an effective coagulant dose. Traditionally, particulate measurement is effected using either turbidity or suspended solids. Both are indirect measurements. Turbidity is the measure of the light scattering properties of particles in the water, whereas suspended solids is a measure of the mass of particulate matter present. If particle sizes or densities vary, these cannot be related to particle numbers.

Particle charge, indicated by either zeta potential or streaming current, correlates with turbidity removal especially when charge neutralisation is the dominant coagulation mechanism. Umgeni Water uses a measurement of streaming current to control the coagulant dose on most of the treatment plants. This has been found to be an effective control parameter for the particular raw water source and the coagulants used on the works. The objective of this study is to determine whether streaming current can be used as an effective control parameter to optimise the concentration of particles in the filtered water, or whether the coagulant dose can be set using the particle counts of filtered water as a primary measure for plant control.

10.2 Literature Study

The removal of particles in water treatment is accomplished by coagulation, settling and filtration processes. The primary objective of water treatment is to destabilise and condition particles in the water chemically and physically for settling and filtration. The particle destabilisation is caused by coagulant addition. Primary coagulants are dispersed into the raw water during a “rapid” or “flash” mixing stage in a fast and uniform manner to make the coagulation as effective as possible. Hydrolysis takes place during the addition of the coagulant and is almost instantaneous. Complete floc formation may take several minutes. The subsequent increase in the size of particles results in the development of floc and is called flocculation. These are critical processes in the removal of particles and the effectiveness is determined by the amount of coagulant added and the extent of destabilisation (AWWA, 1992).

Colloidal particles in water are electro-negatively charged and in stable suspension. Aggregation of these particles results from the addition of positively charged ions to the suspension resulting in destabilisation of the particles i.e. coagulation. The destabilisation of surface charge and the interaction of ions in solution, effects the “zeta potential” which is related to the thickness of the electrical double layer which surrounds a colloid (Van Duuren, 1997). Streaming Current Detection (SCD) is related to the concepts of zeta potential and electrophoretic mobility, and is a measure of the current generated by particles in the water after destabilisation (AWWA, 1992).

During a study by Hutchinson (1984), on-line particle counters were used to monitor the efficiency of the treatment process, and indicated the need to backwash filters. A laboratory-scale particle counter was also used to compare the number and size of particles in the pre-treated and finished water in order to establish optimal chemical dosage. The direct result of implementing this procedure was to reduce chemical costs by up to 32 percent. Additional savings were also realised by a reduction in filter backwash frequency and a corresponding load reduction on the sludge handling system. The conclusion from this study was that particle counters give the operator the information to be able to optimise coagulation and filtration.

In another study a pilot filter was again used to simulate the full-scale plant performance. A laboratory particle counter was used to determine the optimum coagulant dosage (Monscvitz and Rexing, 1983). The remaining particle count in the filtered water within a particular size range was the criterion for optimising the dosage. By performing 5 experiments, fitting a quadratic and setting the derivative equal to zero, the minimum dose could be established. This procedure was also used for a dual coagulant system.

Zeta potential was used to optimise the coagulant dose (Tunison, 1985) where the particle counts in filter effluent were measured and compared for different filters. Tunison found that although a new filter was designed and constructed similar to the old filters, the particle counts in the final water from the new filter were higher than the counts from the old filters. However, the filter run was longer. This showed that, although every attempt was made to reduce the final water particle count, the filter also contributed to the number of particles in the filtered water.

10.3 Materials and Methods

An automated pilot plant consisting of coagulant dosing, pulsator type clarifier, and rapid gravity sand filtration was used during the evaluation. The water flow rate and coagulant dose were controlled by a PLC and the streaming current was measured after flash mixing. The clarifier was operated at an up-flow rate of 1.5 m/hr and a sand filter with 600 mm of 0.95 mm silica sand was operated at a filtration velocity of 5.2 m/hr.

10.3.1 Streaming Current Detector

A simplified cross section of an SCD sensor chamber is presented in Figure 10.3.1. Water flows through the chamber which contains a small piston that reciprocates vertically. The current is generated by electrically charged particles in the water being tested. These particles momentarily attach to the piston and the induced current is measured by electrodes in the outer cylinder.

10.3.2 Particle Counting Instrument

An ARTI on-line particle counter supplied by Optima Hydraulics was used for the study. The instrument was connected to a constant head flow control device which regulated the flow of water through the sensor. The particle counter was calibrated before delivery and connected to a computer that collected data every minute from the sensor. The flow through the sensor was 100 mℓ per minute. The set-up of the particle counter is shown in Figure 10.3.2.

10.3.3 Control of the Coagulant Dose

The coagulant dose was set manually to achieve variability in the streaming current measurement on the pilot plant. A blended polymeric coagulant (DADMAC) was used as the coagulant best suited for the economic operation of the Wiggins Waterworks in Durban. By increasing the coagulant dose, the SCD became more positive.

10.3.4 Sampling and Dilution

Samples of raw, settled and filtered water were taken and individually fed to the particle counter. The particle counts in the respective size ranges were allowed to stabilise before the average particle count was determined. During sampling of the

Raw Water (where the count exceeded the concentration limit of the instrument) the sample was diluted with ultrafiltered water (low particle count) to determine the total number of particles in the water. In order to ensure that the filtered water samples were comparable, the sand filters were backwashed once per day and sampling was performed after an hour of filtration.

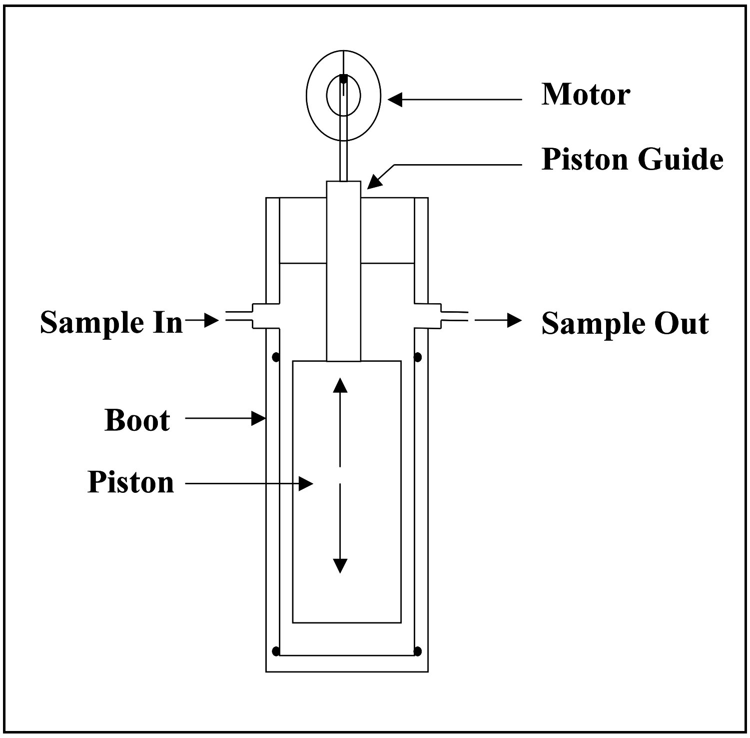

Figure 10.3.1: Schematic of the Streaming Current Detector

10.4 Results and Discussion

Raw water from the Inanda Dam is delivered to the Wiggins Water Treatment Plant through a series of aqueducts and tunnels. This water is piped from the “head of the works” to the pilot plant testing facility where the trials were performed. During the period the raw water turbidity was between 10 and 20 NTU and particle counts > 2 µm between 2 500 and 4 000 per mℓ (Figure 10.4a). The particle index of the average raw water quality was calculated to be 38 : 33 : 19.

The coagulant dose used on the plant was adjusted to between 2 and 7 mg/ℓ, thereby simulating a range of conditions for comparison. The streaming current was measured for both the raw water and the water after charge neutralisation. Figure 10.4.b shows the effect of neutralisation through the addition of coagulant on streaming current. At a dose of approximately 5 mg/ℓ the streaming current became zero. This is not an indication of total neutralisation of surface charge on the particles. To determine this the zeta potential measurement should be used. The streaming current of the raw water during the trial period was relatively consistent between –2,3 and –3,4 mV.

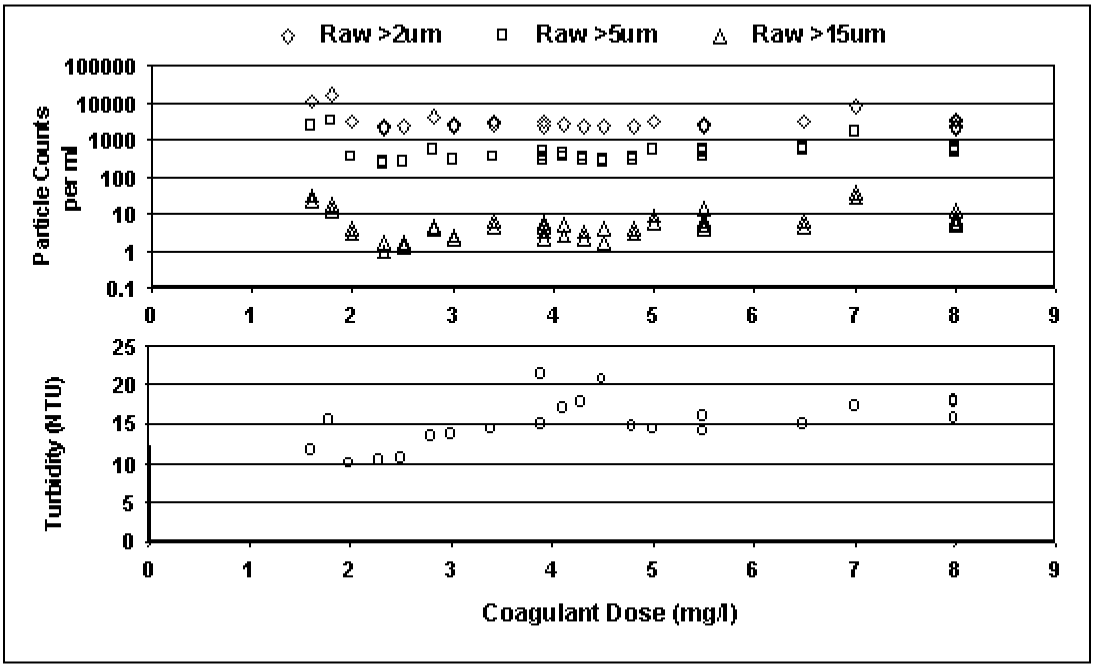

Figure 10.4(a) : Raw Water Turbidity and Particle Counts

The particle counts of the coagulated, flocculated and settled water were reduced from the raw water, as can be seen in Figure 10.4c (2500 particles per mℓ > 2 µm, in the raw water to between 300 and 800 per mℓ in the settled water). At very low coagulant dosages the performance of the clarifier was poor and flocs were visible in the overflow. It was apparent that the distribution of particle size did change between the raw water and the settled water where the number of particles in the size range >15 µm increased from an average of ~2 per mℓ to ~10 per mℓ in the settled water. This is as a direct result of flocculation. There appeared to be a broad range of coagulant dose within which a similar performance was observed. This is not surprising as the plant was being operated below its design capacity and the turbidities of the raw water were relatively low.

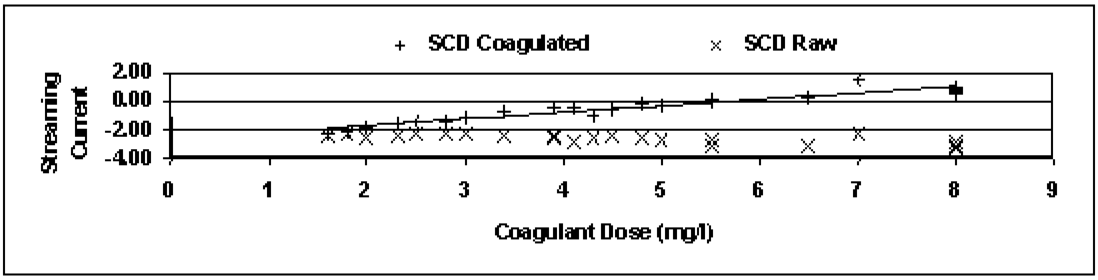

Figure 10.4(b) : The Effect of Coagulant Dose on Streaming Current

When one compares the particle counts and the turbidity of the settled water, a trend may be evident that the lowest particle counts and turbidity are between a coagulant dose of 3 and 5 mg/ℓ.

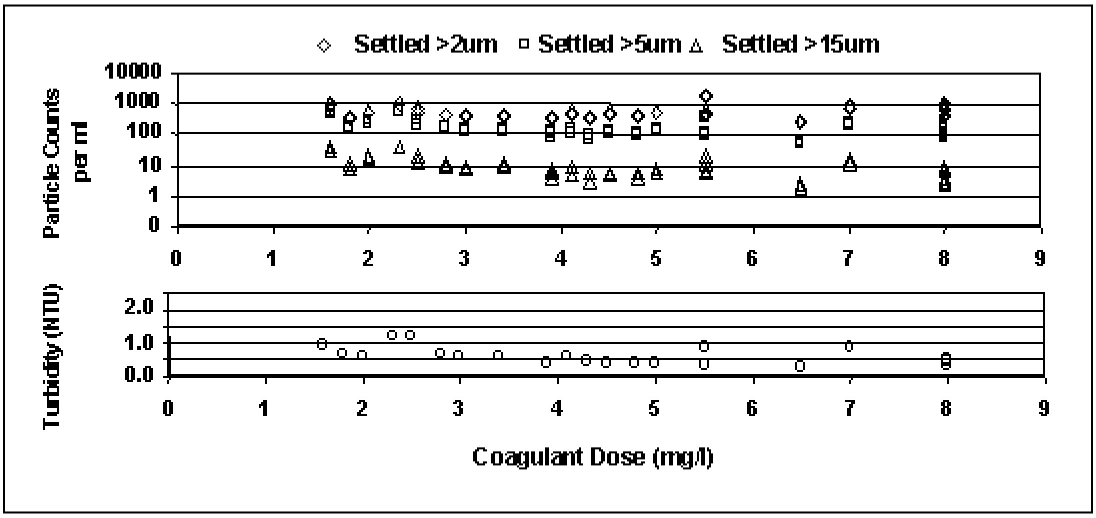

Figure 10.4(c) : Particle Count and Turbidity of Settled Water at Various Coagulant Dosages

Unfortunately, it was not possible to connect the particle counter to the pilot plant on-line as there was insufficient head available to obtain a reliable operation of the counter. It was also not possible to determine the length of filter run before breakthrough nor the average particle count over a full filter run for each coagulant dose.

Particle breakthrough at the beginning of a filter run (during “filter ripening”) will also contribute a high count which has no link to the coagulant dose and optimisation of the coagulation processes. In light of these facts, the use of “filtered water particle count” as a parameter by which the coagulant dose should be controlled would lead to increased effort and probably higher operating costs.

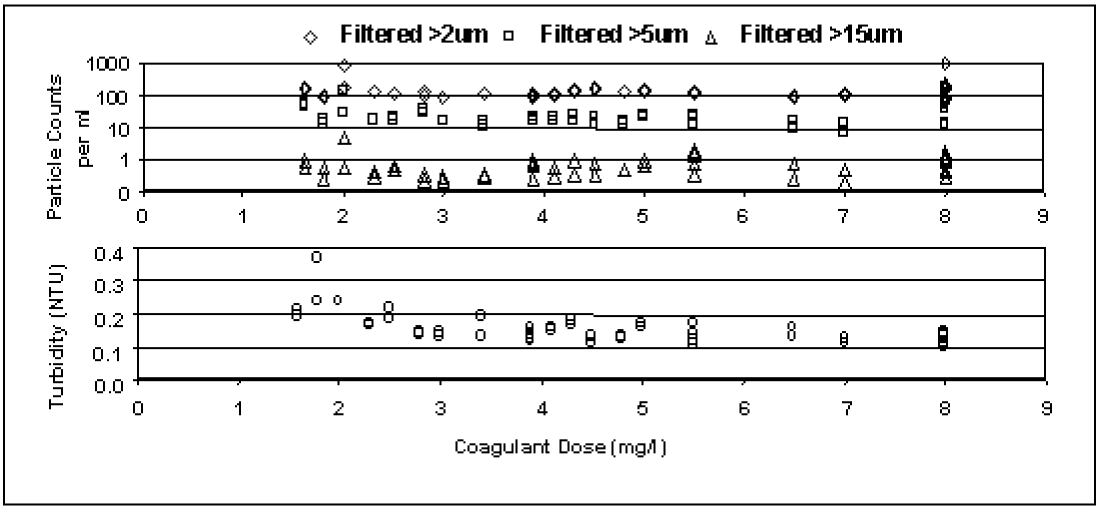

Figure 10.4(d) : Particle Count of Filtered Water at Varying Coagulant Dosages

The settled water particle count, although also relatively stable over a large range of coagulant dosages, would be a more appropriate parameter than the filtered water particle count to use as a direct measure of the effectiveness of coagulation and flocculation. The streaming current (zeta potential measurement) varies most consistently with a changing coagulant dose. It is believed that this is a more appropriate method for controlling the coagulant dose as it is a direct measurement and can be installed directly after coagulation. In this way, a faster response to changes in the raw water quality can be obtained, whereas there may be a delay time when monitoring a particle count after settling or filtration.

The previous study of filtration performance indicated that there may well be a relationship between the conditions at coagulation and the filtered water particle count. This could not be concluded during the pilot plant trials. The large delay time between coagulant dosing and measurement of particle count after sedimentation or after filtration detracts for using particle counting as a control parameter.

10.5 Conclusions and Recommendations

The pilot plant study concluded that the use of particle count as a primary measure of plant performance and coagulation efficiency is not advised. The particle count does provide a more rapid response to changes in plant performance and will be a more sensitive warning of abnormal plant operation. This included pre-treatment, coagulation, sedimentation as well as filtration.

The particle count after settling may be a more direct measurement, but a long delay time between coagulant addition and particle measurement also implies that a poor response to plant operating problems may be obtained.

The streaming current or zeta potential measurement shows the most consistent response to changes in coagulant dose and is therefore a better parameter to use for control of coagulation processes. This can also be applied close to the coagulation point and a faster response to changes in the water quality can be obtained.

10.6 References

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Chapter 11

THE EFFECT OF FLOCCULATION REGIME ON FINAL WATER QUALITY

11.1 Background

South African plants, in general, do not reach the extremely low turbidity ranges (commonly less than 0.15 NTU) which are currently reported from the state-of-the-art treatment plants from Europe or northern America. The most likely explanation is that there is no legislative or consumer pressure to reach these very low values, and operators do not find it necessary to further optimise treatment performance once their own operational goal (commonly 0.5 NTU or lower) is reached. In some cases, however, the authors have observed serious operational effort to minimise filtered water turbidity to the lowest possible level, but even then turbidity of less than 0.15 NTU is seldom or never reached.

It is our, at this point untested, hypothesis that this may be due to a different flocculation regime encountered at many South African treatment plants. Elementary flocculation theory differentiates between the *sweep floc* flocculation regime, and the *charge neutralisation* flocculation regime. In the first case, smaller particles are forcibly swept out of suspension by large chemical precipitates, even if there would be some electrical repulsion between particles. In the second case, electrical repulsion is eliminated by surface charge neutralisation, and particles can agglomerate spontaneously and grow into removable flocs. Intuitively, it seems that the charge destabilisation regime will be more effective in removing the last remnants of unflocculated particles.

These two regimes are controlled by the pH at which flocculation is carried out. At low pH values (approximately between 5.5 and 6.5), the charge neutralisation regime will predominate, while the sweep floc regime will dominate at pH values higher than say 7.5. Many of the raw waters overseas have lower buffer capacity and lower pH than the well-buffered, high pH (and often eutrophic) waters found in inland South Africa. If overseas plants do not already operate at the low pH values required for charge neutralisation, the pH is easily dropped by the acidic nature of the coagulant used. This opportunity does not exist in South Africa, where buffer capacity is high and salinity is already a problem. The hypothesis is that the sweep flocculation regime, encountered more often in South Africa, is inherently the less effective of the two regimes.

To test the hypothesis, one should study the flocculation kinetics at a range of pH values which cover both the sweep floc and the charge neutralisation regimes. The work described in this chapter boils down to a measurement and quantification of flocculation kinetics. It is the objective to use particle counting as a possible way to better calibrate the so-called Argaman-Kaufman model, a potentially powerful method to describe flocculation mathematically.

11.2 Literature Study

11.2.1 The Argaman-Kaufman Model

The Argaman-Kaufman flocculation model can potentially be used as an extrapolation tool to generalise the flocculation behaviour of a particular coagulant/raw water combination from a small number of simple batch experiments. In other words, having only a few data points available, the designer can predict the flocculation behaviour at other flocculation times, at other velocity gradients, and even for other reactor types. Despite the potential power and convenience of this method, it has not been widely adopted in general design practice, probably due in part to the following potential difficulties:

- Uncertainty about the general validity and scalability of the model;
- No generally accepted procedure for the determination of the flocculation constants; and,
- The difficulty of expressing flocculation performance in terms of the Argaman-Kaufman flocculation performance parameter.

The Argaman-Kaufman flocculation model, as it is generally known, encompasses most of the progress that had been made this century in unravelling the mechanisms of orthokinetic flocculation. The mathematical foundations had been laid by the often-quoted work of Von Smoluchowski, developed in 1916 for the orthokinetic flocculation of colloidal particles under laminar flow conditions. In 1943 Camp and Stein introduced the RMS velocity gradient G in the place of the laminar velocity gradient, thereby extending the model to include turbulent flow regimes. The assumption was made that all particle collisions were permanent, and no floc breakup was considered. In 1966, Harris and Kaufman modified the previous models to include the concepts of floc breakup and non-lasting collisions. These concepts were incorporated into a turbulent diffusion model, with the assumption of bimodal floc size distribution, by Argaman and Kaufman in 1968. With a number of simplifying assumptions, they developed and verified a working equation for flocculation in a single completely mixed reactor. By assuming that the model constants remained constant in consecutive tanks, the model was extended to and verified for a number of flocculation tanks in series. In 1977 Bratby and co-workers demonstrated and verified a theoretical link between flocculation in completely mixed and batch reactors, which brought the calibration of the Argaman-Kaufman model within practical reach of relatively simple batch laboratory procedures. A more detailed review of the above developments, along with the concomitant mathematical developments, can be found elsewhere (Bratby, 1980; AWWARF, 1991).

The Argaman-Kaufman equation for batch reactors, which will be used in this paper, is:

$$\frac{n_1}{n_0} = \frac{K_b}{K_a}.G + \left(1 - \frac{K_b}{K_a}.G\right) e^{-K_a.G.T} \quad \ldots\ldots\ldots\ldots\ldots\ldots \quad 11.1$$

Where:

n_1 = number concentration of particles remaining after flocculation period

n_0 = number concentration of particles before flocculation period

K_a = aggregation constant

K_b = breakup constant

G = root mean square velocity gradient (1/sec)

T = flocculation period (sec)

The flocculation performance parameter is defined as the ratio n_1 / n_0.

11.2.2 *Measurement of Flocculation Performance n_1/n_0.*

The measurement of supernatant turbidity, after flocculation and settling in a standard jar test apparatus, is a universal and time-tried yardstick of flocculation performance. This principle, of substituting turbidity as a measure of particle concentration, had also been used for the expression of flocculation performance in Argaman-Kaufman testing. The proposed method of sample withdrawal (Bratby, 1980) used a bent pipette for drawing a sample horizontally 30 mm under the water surface - the same method used by the authors. There are three complications when turbidity is used to quantify flocculation performance:

S If a flocculated sample is left to settle, the flocculated particles as well as the unflocculated primary particles will settle from the suspension, albeit at different rates. The turbidity measured after settling (representative of n_1) will actually measure less primary particles than those present immediately after flocculation stopped. The accepted way to compensate for this error (Bratby, 1980), is to measure the initial turbidity (representative of n_0) only *after* a sample has been coagulated and *allowed to settle for the same settling period*, without any flocculation at all. In this way, the n_0 measurement will also reflect less primary particles than those present immediately after flocculation, thereby, hopefully, leading to a true n_1/n_0 ratio.

- How long should samples be left to settle before n_0 and n_1 are measured? One qualitative guideline is that the settling time should correspond to a time slightly greater than that beyond which no significant improvement in supernatant quality is evident (Bratby, 1980). In theory, the settling time should not be all that critical if the reasoning in the previous paragraph is valid.
- The derivation of the Argaman-Kaufman model is based on the removal of primary particles from a suspension. Turbidity has been used in the past as a substitute for the determination of the actual number of particles in suspension.

Particle counting holds the promise of overcoming these difficulties posed by using turbidity for measuring flocculation performance. If the primary particles could be measured directly with the particle counter, then there would be no need for settling, as the samples can be analysed directly after flocculation. The tricky question of settling time would not enter into the problem at all.

11.3 Materials and Methods

It stands to reason that flocculation constants cannot be compared or generalised unless the method and apparatus are standardised. For this reason, apparatus and a methodology were recommended by Bratby et al.(1977), also reproduced in Bratby (1980). This included the exact geometry of the apparatus, along with a calibration curve for stirrer speed *versus* velocity gradient G. These recommendations were closely followed for the experiments reported here.

Two flocculation constants (K_a and K_b) are derived from the experimental data. The degrees of freedom is equal to the number of tests n minus two. Moreover, the effect of the aggregation constant K_a is especially evident early in the flocculation process, while the effect of the breakup constant K_b is only evident later in the flocculation process, and especially at high velocity gradients. A previous study showed (Haarhoff et al., 1996) that:

- a total of nine batch tests is sufficient for reliable estimation of the flocculation constants, by including three different velocity gradients and three different flocculation times,
- sufficiently short flocculation time of 6 minutes or less is included to demonstrate the effect of the aggregation constant, and

- a sufficiently high velocity gradient of 100 /s and long flocculation time of 12 to 18 minutes are included to demonstrate the effect of the breakup constant.

The experimental matrix used in this project accordingly used twelve batch tests, namely three velocity gradients and four flocculation times, which leaves ten degrees of freedom after estimation of the flocculation constants.

The least-square fitting of the flocculation constants was done in terms of n_1/n_0 instead of n_0/n_1, as it varies linearly with the experimental or measurement error. If this is not done, a small measurement error at low turbidity will be greatly amplified if it is used below the line, and will dominate the sum of squares used for the best fit.

11.4 Detailed Analytical Procedure

The experimental procedure for the Argaman-Kaufman test is detailed below. The procedure is the nett result of numerous attempts at the test. Due to the sensitivity of the result to experimental variation, it is crucial that the test is performed consistently and that the floc is handled with care. The flocculation kinetics are monitored by following both turbidity and particle counts at the same points throughout the experiment, allowing a comparison of the two techniques.

11.4.1 Pre-test Preparation

- Switch on the particle counter and turbidimeter approximately half-an-hour before starting any test.
- Clean all glassware to be used thoroughly with detergent and rinse with tap water followed by ultra pure water.
- Ensure that the environment in which the tests are being conducted is dust-free as far as possible in order to minimise contamination of the test samples.

11.4.2 Preparation of the Coagulant

- The coagulant chosen in all tests performed thus far has been an inorganic metallic coagulant, iron (III) chloride or ferric chloride ($FeCl_3$).
- The coagulant dose is expressed as $FeCl_3$ rather than Fe^{3+}. Thus the dose solution is prepared as “grams $FeCl_3$ per ℓ of solution”.
- The concentration of the dose solution prepared is 1 g/ℓ.

11.4.3 Preparation of the Kaolin Suspension

- Weigh approximately 5 g of kaolin powder in a weighing bottle and transfer to a 1 ℓ volumetric flask.
- Make up to the mark using ultra pure water.
- Mix this solution thoroughly by swirling and inverting to ensure that all the powder is dispersed in the water. This solution will now be used as a stock solution to prepare the test samples by dilution.

11.4.4 Preparation of the Test Samples

- The test samples should have a kaolin concentration of approximately 55 mg/ℓ.
- To ensure uniformity in concentration, a 20 ℓ volume of solution is prepared for each flocculation G value.
- Pour 220 mℓ of the stock solution (thoroughly mixed using a magnetic stirrer) into a measuring cylinder and transfer to a 25 ℓ aspirator bottle. Make up to the 20 ℓ mark using ultra pure water.
- Mix thoroughly by inverting and swirling.

11.4.5 The Test

- Disperse 3 ℓ of the test sample into a reactor jar. Lower the impeller and mix at a low speed.
- The mixing conditions under which the tests are to be performed are tabulated in Table 11.4.5
- A maximum of 3 tests can be performed at a time, owing to the limitations of the equipment.
- The coagulant to be dosed is dispersed into the sample using a syringe.
- Using the optimised dose (12 mg/ℓ in this instant), obtain the correct volume of coagulant into a syringe, one for each reactor.
- Start the rapid mix and disperse the coagulant as close as possible to the impeller. Assistance might be needed in this regard since the three syringes are discharged simultaneously.
- Particle counts are measured at the point when each flocculation time expires.
- Dilution is necessary since the counts were over the concentration limit of the counter.

Table 11.4.5 : Coagulation and Flocculation Parameters for the Argaman-Kaufman Tests

Coagulation		
G value (sec^{-1})	**Corresponding rpm**	**Mixing time (sec)**
250	100	15

Flocculation		
G value (sec^{-1})	**Corresponding rpm**	**Mixing time (min)**
30	22	3; 6; 10; 15
60	41	3; 6; 10; 15
100	46	3; 6; 10; 15

- To dilute, collect a volume of sample using a pipette with a 90° bend. The tip might need to be cut in order to widen the hole to avoid break up of the floc. Draw up slowly, to avoid floc breakage, from a depth of about 30 mm below the liquid surface.
- Disperse the collected sample slowly beneath the surface of the dilution water. Note that the sample is added to the water and not vice versa.
- Mix only by a swirling motion of the beaker. Cover with a clear polyethylene plastic wrap and measure the counts.
- Turbidity measurement is conducted after the optimised settling time (30 min in this instant) has expired for each sample. The settling time was selected after settling tests indicated that longer settling times did not improve the water clarity significantly.
- In addition to the determinations for the four flocculation times, a value for n_0 is determined from each 20 ℓ sample prepared. This is achieved using the exact same procedure above with the omission of the coagulant and flocculation i.e. particle counts are measured after rapid mix and turbidity after settling.

11.4.6 pH Adjustment

- In addition to the standard tests where pH is constant, a separate set of tests was conducted at varying pH levels.
- The pH is adjusted using HCl and NaOH.
- The test outlined above was now conducted at chosen pH levels.
- The tests were performed at pH 6, 7 and 8 in order to view the effect of different flocculation regimes.

11.5 Results and Discussion

Figure 11.5(a) illustrates a typical result returned by the Argaman-Kaufman test. Each of the tests return a co-ordinate on a n_1 / n_0 vs. mixing time graph. Where n_1 / n_0 denotes the ratio of particle count (or turbidity) at the end of the specific flocculation time tested to the particle count (or turbidity) directly after coagulation. The Argaman-Kaufman model is then fit to the data by tweaking the K_a and K_b values until the least squares curve is found. The flocculation (K_a) and break-up (K_b) constants are then taken to describe the flocculation characteristics of the suspension.

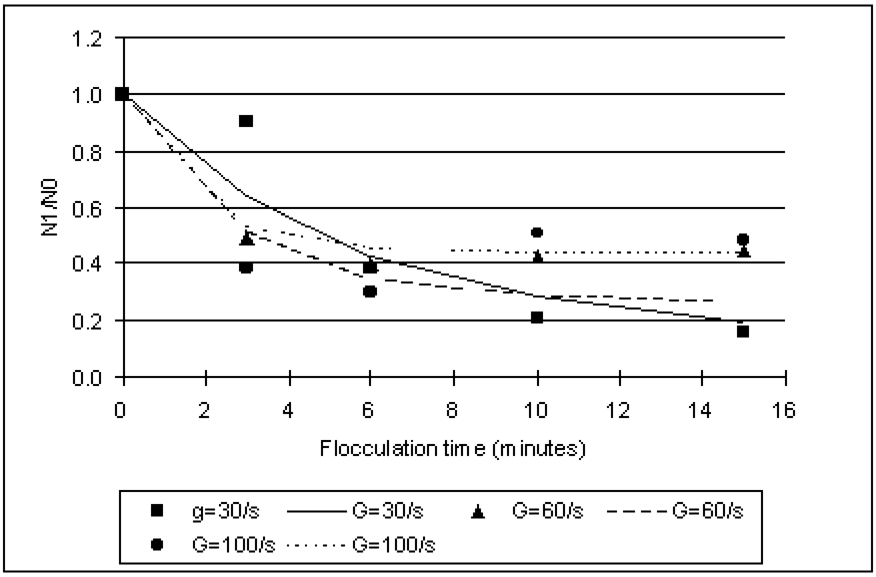

Figure 11.5(a) : A Typical Result Returned After Performing the Argaman-Kaufman Test.

When considering the Argaman-Kaufman results returned for tests at various pH levels, the results are expected to indicate an improved final water quality due to increased aggregation at lower pH values and/or a decreased break-up in the same region. When the results are considered from the test performed at various pH levels, this is not found. Figure 11.5(b) shows the results for the particle counting tests and Figure 11.5(c) show the results for the same test based on turbidity. Both tests indicate that the aggregation constant remains approximately constant at the three pH levels. The break-up constant, however, illustrates unexpected tendencies. The break-up constant using particle counting techniques is unstable, indicating no trend, whereas the turbidity methodology indicates a break-up constant following a trend contradictory to what is expected. The results obtained from these tests are inconclusive.

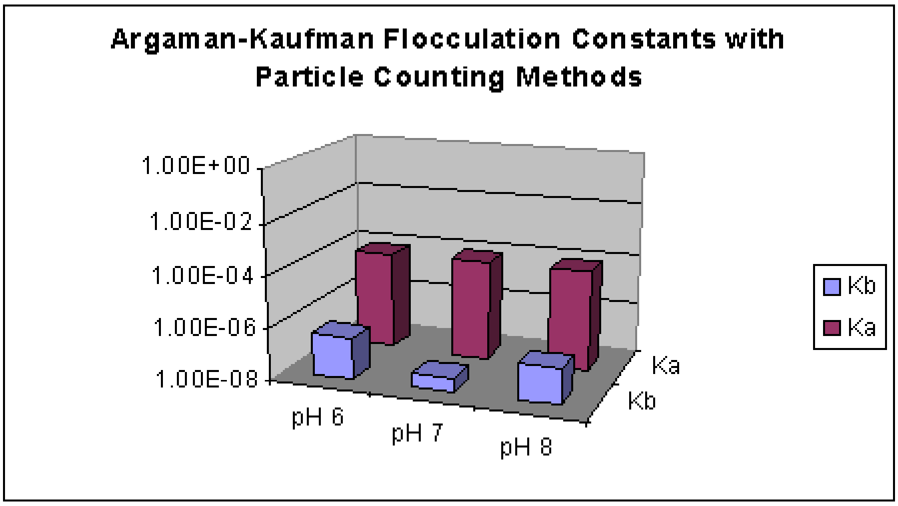

Figure 11.5(b) : The Results from the Argaman-Kaufman Tests Based on the Particle Counting Technique.

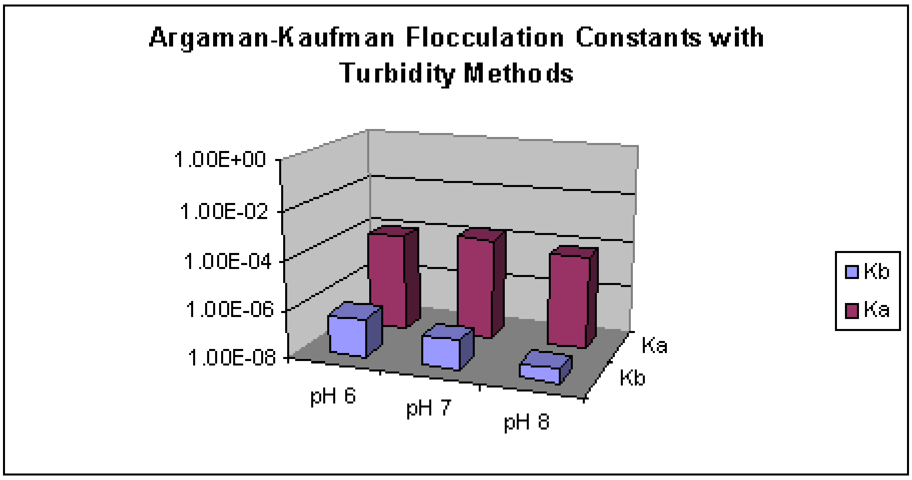

Figure 11.5(c) : The Results from the Argaman-Kaufman Tests Based on the Turbidity Technique.

11.6 Conclusions and Recommendations

In this study, the suitability of particle counting was assessed as a means to measure flocculation kinetics. If this were successful, it would be a significant stride forward to introduce the Argaman-Kaufman flocculation model as a practical methodology for measuring and predicting flocculation kinetics. Despite repeated attempts and the introduction of numerous precautions and improvements to the experimental method, this study did not provide the precision and repeatability required by the researchers.

Does this reflect negatively on particle counting in general? The researchers believe not. The processes of coagulation and flocculation are, after almost a century of research as summarised in the literature survey, still poorly understood even at a conceptual level. To measure such a complex, transient phenomenon as flocs in the process of growing and breaking up, is the hardest possible test to which particle counting could be put. Specific questions that could be raised in this case, include:

- Concerns regarding the amount of flocculation or break-up that might occur in the feed line to the particle counter sensor. Although it might be argued that an acceptable comparative result between various flocculation regimes might be derived from the admittedly "flawed" measurements, it cannot be guaranteed that all flocs are affected similarly during transport. Large fluffy flocs are sure to be affected differently than more compact flocs. It is virtually impossible to distinguish between the affect of mixing intensity and floc transport to the sensor.
- The affect of the comparatively turbulent flow when the floc enters and passes through the sensor also give rise to similar questions as above.
- There is a significant time delay from sampling the particles to measuring the particles due to the transfer of the sample along the sampling tube. The counter used in this study had a delay of approximately 60 seconds. It is not clear what the affect of this delay on the nature of the floc is.
- The response of particle counters to floc has not been studied to date. Since light obscuration particle sensors essentially detect the reduction of light falling on it , it is quite conceivable that large fluffy flocs might elicit the same response as smaller more dense flocs, essentially negating the affect of various flocculation variables such as mixing intensity and flocculation regime.

Most of these concerns fall away when particle counting is applied to relatively stable suspensions which change over tens of minutes or hours (such as filter cycle suspension changes), and when the processes of floc formation and breakup have been mostly arrested. Therefore, for tracking flocculation kinetics with the type of experiment conducted here, particle counting is not a reliable tool, but this does not preclude its successful application to many other drinking water applications.

11.7 References

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Chapter 12

FILTER PERFORMANCE UNDER VARIOUS HYDRAULIC CONTROL REGIMES

12.1 Background

Particle breakthrough in filter operation is most likely in the first hour of operation. Cranston and Amirtharajah (1987) pointed out that the transport of potentially pathogenic micro-organisms might occur in this initial period of degradation. Alternatives that had been suggested to reduce the ripening peak include filter-to-waste and the injection of poly-electrolytes to the backwash water in the final stages of backwash.

These alternatives, although effective, are not ideal. The filter-to-waste procedure can lead to the loss of significant quantities of product water, particularly if the ripening period is long. Ripening can take up to two hours. In addition to this, both filter-to-waste and addition of poly-electrolytes require significant and costly infra-structural changes to plants that have not been designed with these alternatives in mind.

Other suggestions include (Cranston and Amirtharajah, 1987):

- discharging backwash water standing above the media in the filter basin, but again not all plants are provided with the infrastructure required for this alternative and this alternative will also lead to a further wastage of water, and
- extending backwash runs for some time after the bed had been washed at lower than minimum fluidisation rates which also leads to excessive water wastage.

In contrast to this, all filters allow a measure of hydraulic control in their start-up procedures. If it can be shown that alternative hydraulic control regimes can lead to significant reductions in the peak and duration of the ripening peak, this alternative can be employed at the most basic plants. In the South African context, this is of particular importance.

12.2 Literature

12.2.1 Filter Ripening

Amirtharajah and Wetstein (1980) studied the initial degradation in effluent quality in detail and concluded that the poor quality of the effluent was caused by the remnants in the backwash water that was still in the filter basin after backwash. A detailed analysis of the ripening peak was presented and this is shown in Figure 12.2.1.

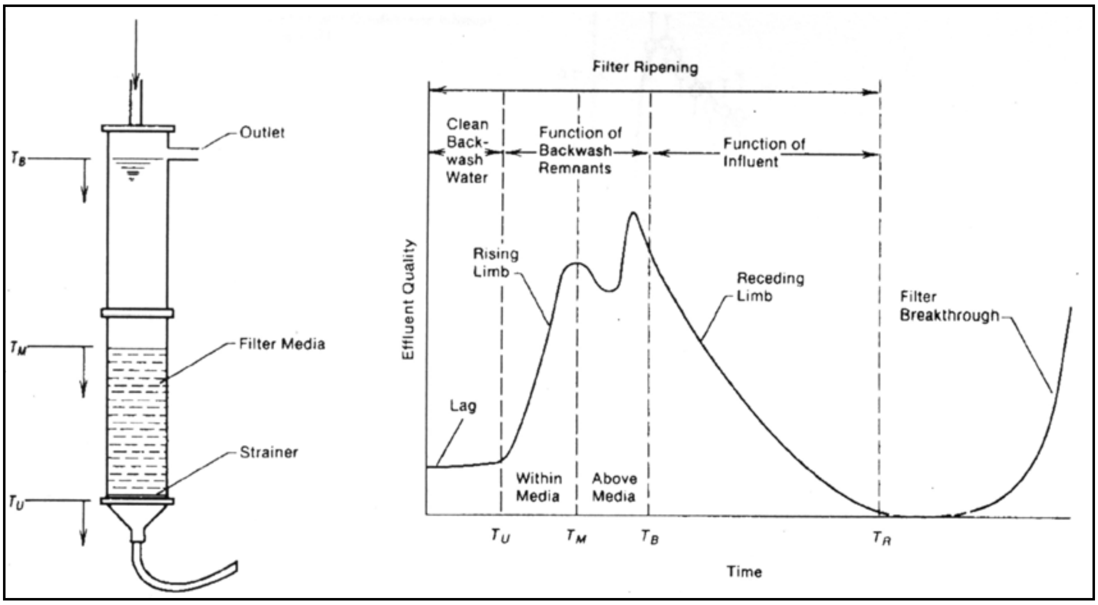

Figure 12.2.1: Detailed Analysis of the Ripening Peak. (Amirtharajah and Wetstein, (1980))

The analysis shows several distinct phases in the ripening peak. These stages can be traced to the position of the water at the end of the backwash process.

The first stage is fairly clean as it originates from the backwash water that is still in the underdrain system at the end of the backwash cycle. The time at the end of this stage is denoted as T_U.

The second and third stages are functions of remnants in the backwash water that is in the media and in the filter basin above the media respectively, at the end of the backwash cycle. The times at the end of these stages are denoted with T_M and T_B respectively and both times correspond with peaks. The first peak is not always noticeable due to the difference in peak size and the time resolution of the measurement technique employed. It is also directly related to the rate of filtration. T_B generally lasts from 1 to 10 minutes.

After T_B, the water quality improves as new water enters the basin. The receding limb of the curve describes the stage in which the effluent quality improves and this ends at T_R when the characteristic production phase of a filter starts where a water of constant quality is produced. T_R may extend from 1 to 2 hours.

12.2.2 Mechanisms in Filter Ripening

Several mechanisms are responsible for the improvement of effluent quality during filter ripening. Cranston and Amirtharajah (1987) made mention of the following two.

- *Dendrite and particle chain formation*
  As particles attach to media they effectively increase the size of the media grain. The increased "surface area" of the media grain then allows for more efficient transport of particles to the grain as well as for more effective attachment. This leads to an improved filter effluent quality.
- *Pore clogging*
  Pore clogging leads to an increase in local capture efficiency. After backwash no pores are clogged. During the initial stages of filtration clogging becomes more prevalent and this leads to increased filtration efficiency.

12.2.3 Factors Influencing Ripening

The following variables have been found to influence filtration efficiency in general and filter ripening specifically.

- *Filtration rate*
  The negative impact of increased filtration rate on particle is expected from filtration theory (Clark et al., 1992). This was also found to be the case in work done by Darby et al. (1991).

- *Suspended particle size*
  The physical characteristics of particles are thought to play the dominant role in the transport of the particle to collector sites (Darby and Lawler, 1990). It was suggested that larger particles are transported to their collection sites on the media by sedimentation and fluid flow, while smaller particles are captured more efficiently by Brownian movement (Darby et al., 1991). Clark et al. (1992) compared the ripening of particles of various sizes and found that ripening occurred at different rates for different particle sizes. The ripening effect on smaller particles (1 to 6 µm) continued long after the ripening of larger particles (6 to 13 µm) had stopped. It was suggested that the poorer removal might be caused by surface chemical effects or by floc break-off.

- *Suspended particle size distribution*

  Vignesvaran and Ben Aim (1985) suggested that retained coarse, with retained fine particles, acted as additional particle receptors and that this could account for the improvement of the removal efficiency of finer particles in the presence of coarser particles. Chang and Vignesvaran (1990) have already developed a mathematical model to incorporate particle size distribution.

- *Particle concentration*

  Higher particle concentrations lead to faster rates of ripening. This is because the rate of ripening depends on the number of particles captured previously (Clark et al., 1992). The total number of particles available (concentration) to be captured has a significant effect on the removal in a filter bed.

- *Particle destabilisation*

  The degree of stabilisation of the particles to be removed, plays a significant role in the removal of particles during ripening and filtration in general (Tobiason et al., 1996). This is due to the role that chemical conditions play in the attachment of particle to collector sites (Darby and Lawler, 1990)

Apart from filtration rate and particle destabilisation, the plant operator has no control over the factors that govern the removal of particles during ripening as well as during normal filtration. As stated earlier, particle destabilisation involves complicated operational procedures and sometimes fairly expensive plant alterations. This leaves the hydraulic control of the start-up as the only feasible option for amelioration of the ripening stage.

Tobiason *et al. (1996)* did some work on the effect of hydraulic control of filters during start-up at full scale plants and concluded that :

- A reduced initial filtration rate can lower the particle concentration in the ripening peak as well as reduce the cumulative number of particles in the filtered water during the filtration run,
- a higher initial rate causes an increase in the ripening peak but may allow for a shorter time period for filter to waste operation to achieve a desired filter water quality, and
- regardless of initial rate, the peak concentration during ripening occurs at a volume of water associated with the backwash remnants located just above the filter media at the end of backwash.

The work of Tobiason *et al.* (1996) was conducted on full-scale plants and difficulty was experienced in repeating some results. Significant variation was found in the water quality in replicate tests.

12.3 Materials and Methods

The work in this study was carried out on a pilot filtration plant in order to exercise more control over the start-up process. The plant was operated in a direct filtration mode. An artificial raw water was made up using water from a municipal supply to which kaolin was added. The raw water was prepared in a 200 ℓ drum and the kaolin was kept in suspension with a mixer. The water was flocculated using ferric(III)chloride ($FeCl_3$) which was dosed at an optimised rate using a peristaltic pump. A 6 m long 25 mm ϕ pipe running at a super critical angle was used as flocculator. Filtration in the 140 mm ϕ column was carried out at a constant rate of 5 m/h. The media bed was 400 mm deep and consisted of commercially available filter sand with an effective size of 0,85 mm and a uniformity coefficient of 1,4. The filter was operated under constant head and was backwashed from a municipal source. In the latter runs of the investigation the influent water particle counts and turbidity were also monitored to detect whether there were anomalies in the experimental setup that was influencing the outcome of the experiments.

Several runs were monitored. Initial runs were held constant at 5 m/hr. Subsequent runs were increased from 3 to 5 m/hr over a period lasting 20 minutes while others were decreased from 7 to 5 m/hr over a similar period. In order to minimise the effects of variables such as raw water quality, the runs were executed in quick succession. The experiment was a continuous exercise with only two hours allowed for filter ripening and production between the different backwashes. The experimental procedure was improved over time in order to achieve repeatability which proved to be elusive. This report will deal mainly with results generated in the latter part of the study. A more complete record of al the tests done is reported elsewhere (Husselmann, 2000). Theoretical values for T_U, T_M and T_B, as defined by Amirtharajah and Wetstein (1980), were calculated for the three experimental setups and are given in Table 12.3.

Table 12.3 : Theoretical Values for T_U, T_M and T_B

Description of Experiment	T_U	T_M	T_B
Constant Rate Start-up	0.9 min	3.5 min	26.3 min
Decreasing Rate Start-up	0.6 min	2.5 min	21.4 min
Increasing Rate Start-up	1.5 min	5.75 min	31.3 min

The ripening curves were monitored using a Pamas 3316 particle counter which monitored 16 particle size ranges as described in paragraph 5.2 of this report. The particle counter was run in an on-line mode. Turbidity measurements were also taken using a HACH Ratio Turbidimeter.

In addition to this, two full-scale plants were monitored on several occasions. The Pamas 3316 particle counter was used again. Grab samples were taken in the initial runs (those done in 1999) and in later runs (2000 runs) the particle counter was used in an on-line mode. The operators were asked to backwash in the “normal’ way so that repeatability of the backwash procedure could be ensured. In both cases the backwash process was automated with only initiation being triggered manually. Turbidity was also measured occasionally and this was done on grab samples. The purpose of this exercise was to compare pilot plant results with full-scale results.

12.4 Results and Discussion

The results from the three sets of experiments are shown graphically in Figures 12.4(a), (b) and (c) and the various runs are numbered 4.1 to 4.6. Each graph contains both particle counts and turbidity measurements taken during two runs. Figures 12.4(b) and (c) also contain count and turbidity plots of the incoming (“raw”) water for the various experiments.

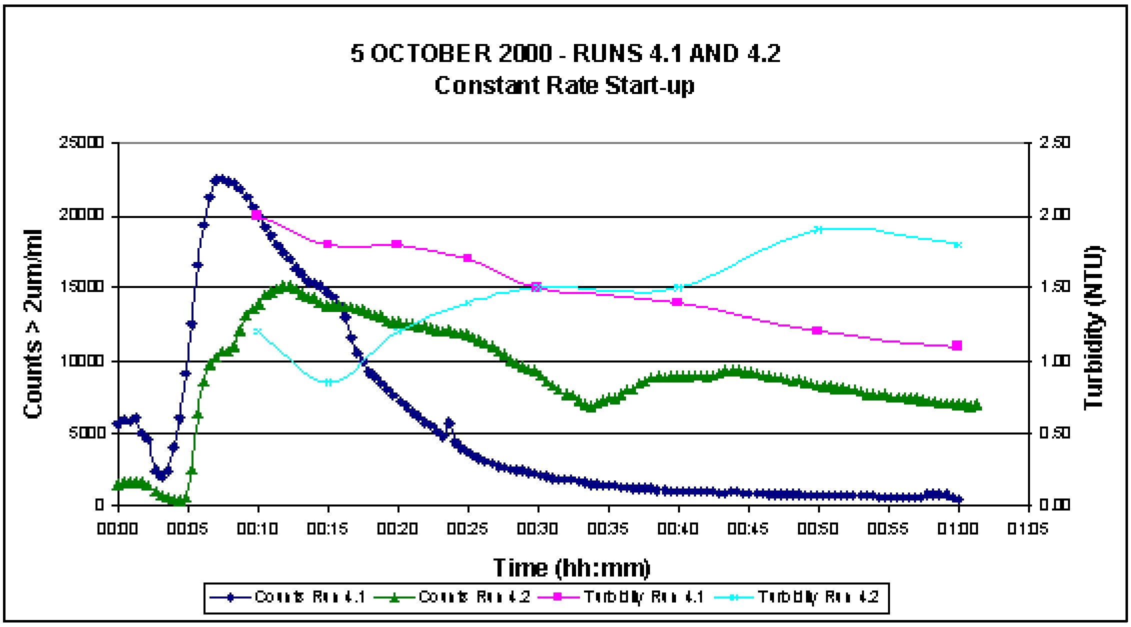

Figure 12.4(a) : Ripening Curves for Filter Runs at 5 m/s.

The particle counts for Runs 4.1 and 4.2 indicate that ripening is taking place. This is, however, not reflected by the turbidity measurements in the same period. The same observation is made in Runs 4.3 to 4.6, indicating that particle counting is the more sensitive indicator of filter performance between the two. Both particle count runs indicate a clear decrease in water quality followed by an improvement. The two graphs are not similar, with a difference in peak counts approaching 50% and also occurring 5 minutes apart. There is also a significant difference in the stabilised counts. When the graphs are compared with the graph presented in Figure 12.2.1, several other observations are also made. These include :

- The lag before the initial degradation in water quality occurs, is visible albeit at different levels. This indicates that the filter was possibly not cleaned to the same level before each run. This must have an influence on the progression of the individual runs but it is not possible to quantify the affect of this at this time.
- One peak, and not two, is visible in the graph. According to Amirtharajah and Wetstein (1980) the first peak is not always visible. However, the peak time does not correspond to the theoretical time for T_B presented in Table 12.3 for this particular experiment. It is, in fact, much closer to the value for T_M. This cannot be explained at this time.
- According to Amirtharajah and Wetstein, the bed should be fully ripened by time T_R. This time is quoted as approximately 1 to 2 hours. The scale model shows some variation in this parameter but the ripening time seems to lie within the range indicated by Amirtharajah and Wetstein.

Runs 4.3 and 4.4 (Figure 12.4(b)) tracked the ripening of the filter during the “Decreasing Start-up Rate” experiment. Also indicated on this graph is the particle counts and turbidity values for the incoming flow. Although some variation is shown in the incoming water flow, this is not considered to be excessive.

The particle counts for the filter effluent show a better comparison directly after start-up than the previous two runs. This, however, does not preclude the two runs shown here, to diverge significantly later in the experiment. In this case, the difference in peak counts approach 100%. Neither of the two runs exhibit a clear peak and ripening continues for the entire run. Again there is a significant difference in the final water quality toward the end of the run.

Runs 4.5 and 4.6 show a closer correlation in the early stages of the run with both the start-up levels showing close resemblance and the peaks occurring at approximately the same time and level. The ripening stages of the two runs are different. Run 4.6 has a more “successful” recovery in the period following the peak than Run 4.5. Again the stabilised water qualities vary substantially.

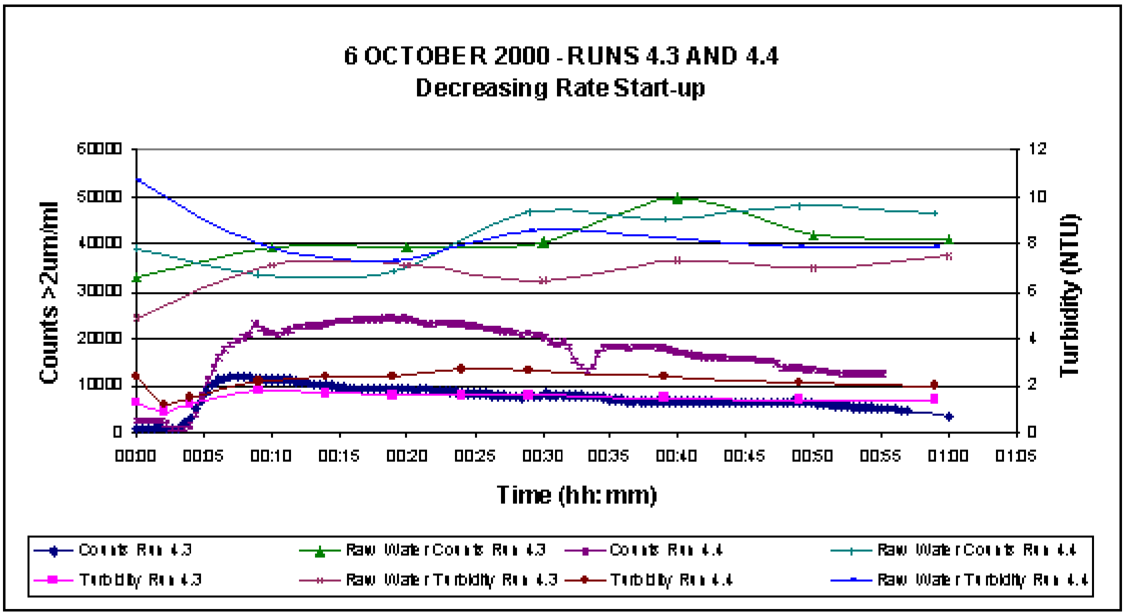

Figure 12.4(b) : Ripening Curves for Filter Runs Started at 7 m/s and Reduced to 5 m/s in 20 minutes.

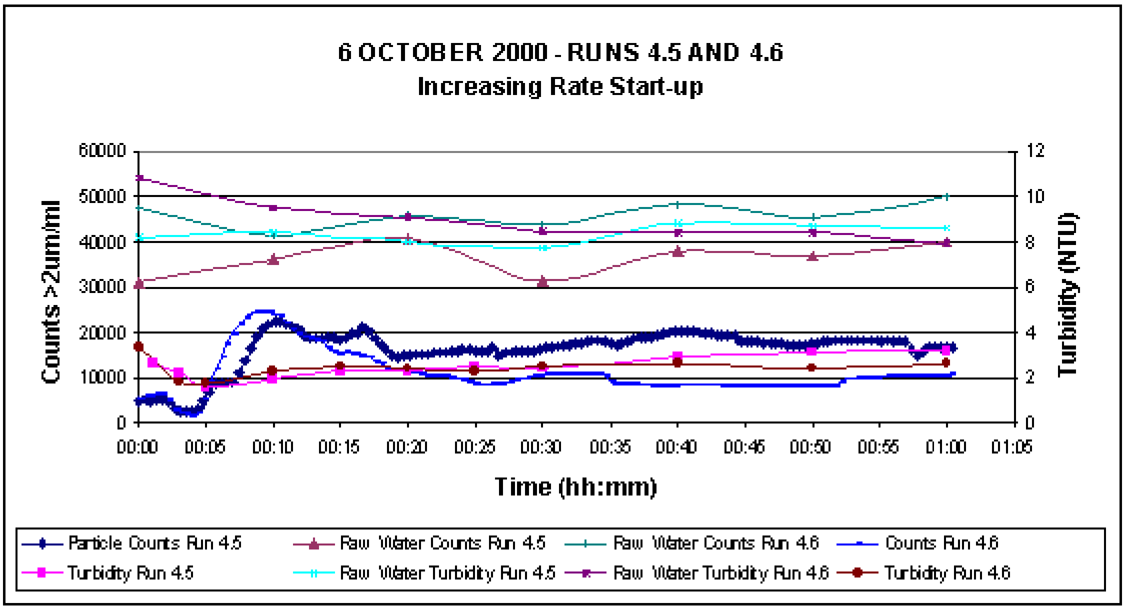

Figure 12.4(c) : Ripening Curves for Filter Runs Started at 3 m/s and Increased to 5 m/s in 20 minutes.

Table 12.3 compares the different filter runs on a theoretical basis. Current theory base the estimate of the occurrence of the peak in ripening, on the amount of water filtered. It follows that the peak will occur faster if initial flow rates are higher. On this basis, the values in Table 12.3 have been calculated. It is not that easy to determine the height of the peak. Some models have been published, but these have not been incorporated in this study as the intention was to measure these peaks. The basic assumption is that the higher the start-up rate is, the higher the peak would be. This is confirmed by the work done by Tobiason et al. (1996).

Table 12.4 summarises the approximate values obtained from the experimental runs for peak counts, T_U, T_M and T_B. The results appear to follow the time patterns as predicted, albeit at different values with the order of peaks following the order of filtration start-up rates. The average peak counts indicate the opposite to what was expected. Here the lowest start-up rate has the highest peak and the highest rate the lowest peak. It is interesting to note that the decreasing rate start-up run took the longest to reach a stable production stage, essentially negating the low peak value reached very early.

Table 12.4 : Measured Values for Peak Counts, Peak Durations, T_U, T_M and T_B

Description of Experiment	Peak Counts >2 µm/mℓ	Peak Duration	T_U	T_M	T_B
Constant Rate Start-up	15 000 - 22 500	25-35 min	3-5 min	7-12 min	
Decreasing Rate Start-up	12 000 - 24 000	50+ min	3-4 min	6-8 min	
Increasing Rate Start-up	22 000 - 25 000	15-20 min	3-4 min	8-10 min	

In order to establish which operating regime provides the lowest risk of particle breakthrough during start-up, peak counts and peak duration have to be considered. In essence, the breakthrough curve has to be integrated over time to give the total number of particles that have broken through. The data presented here is not sufficient to allow confidence in the result that is returned if such an exercise is performed. This exercise has however been performed and is reported as an indication of how it could be done in future studies.

Runs 4.1, 4.3 and 4.6 have been selected as “typical” runs and a base count of 75 000 particles > 2 µm/mℓ is arbitrarily taken to indicate the end of ripening for all three counts. Calculating the number of particles passed through the filter above this value then returns the result as indicated in Figure 12.4(d).

The full-scale test also revealed a significant variation in measurements. The data for Plant 1 and Plant 2 are presented in Figures 12.4(e) and (f). Plant 1 is a 40 Mℓ/d plant treating water from a eutrophic impoundment. The filters are fully automated and washed after a fixed period of operation before breakthrough is experienced. The backwash procedure, which includes air scour, is automated. The first minute of filter water is discarded in a filter to waste procedure. Three different filters were monitored at this plant. Start-up was timed from the start of the filter-to-waste procedure, but no samples were taken from this stream due to accessibility problems. A single sample was taken from the filter outlet as an indication of clear water counts and turbidity as this was the expected point of departure for the curve.

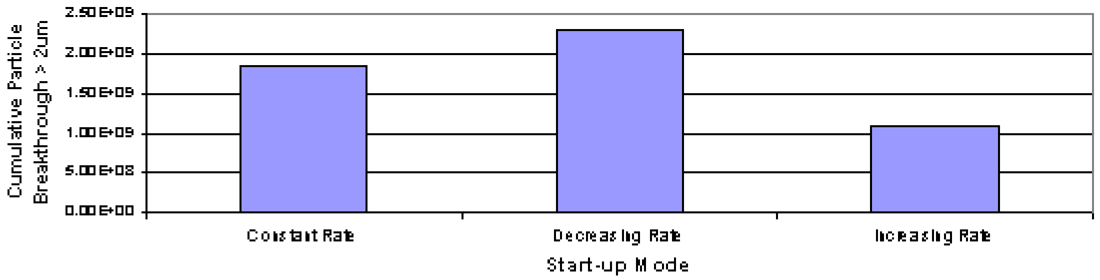

Figure 12.4(d) : Indication of Total Particle Breakthrough for the Three Start-up Modes Investigated

As with the laboratory tests, the turbidity measurements at Plant 1 do not show much variation and do not indicate a distinct ripening curve. However, this is visible for the count data. The three count curves do show some consistency, but this is still not sufficient to analyse the affect of various control options. More runs are required to determine the median response of the filter to the various procedures. The runs also do not show the detail indicated by Amirtharajah and Wetstein (1980). This is possible due to the fact that the first minute of the run is not monitored and also that the response time and resolution of the particle counter is not sufficient to detect the first peak. The peaks measures in Plant 1, and also Plant 2, are much lower than the peaks observed in the pilot plant. This is attributed to the different nature of the suspensions filtered in the different cases.

The turbidity measurement at Plant 2 indicates a more distinct curve than measured previously. However, it is still unsatisfactory due to its unstable nature. The criticism can be levelled at at least two of the particle count runs at this plant. The run of 30 June 1999 posed some particular concern. Plant 2 is a 90 Mℓ/d plant treating similar water to Plant 1. The backwash procedure is similar to that of Plant 1, except that filter to waste is not employed. The peaks show a significant variation in size. This is attributed to all the tests being performed on different days, whereas two of the tests on Plant 1 were performed on the same day. The differing peaks can possibly be attributed to changes in filter influent quality. Again, more tests are required to determine the response of these filters to varying control procedures.

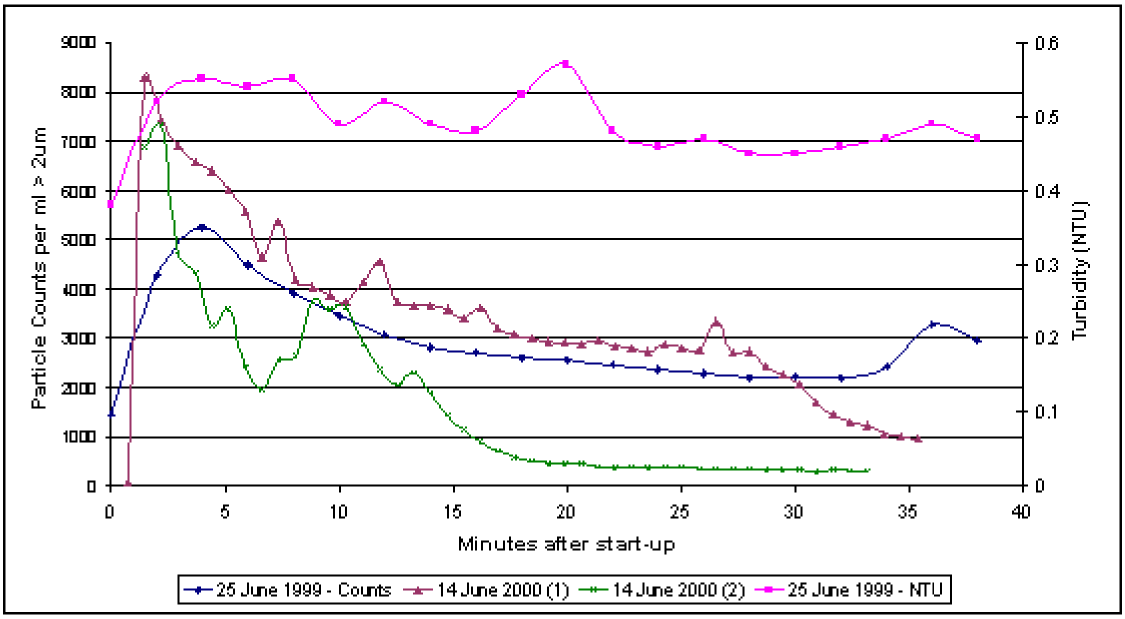

Figure 12.4(e) : Filter Start-up Monitoring at Plant 1

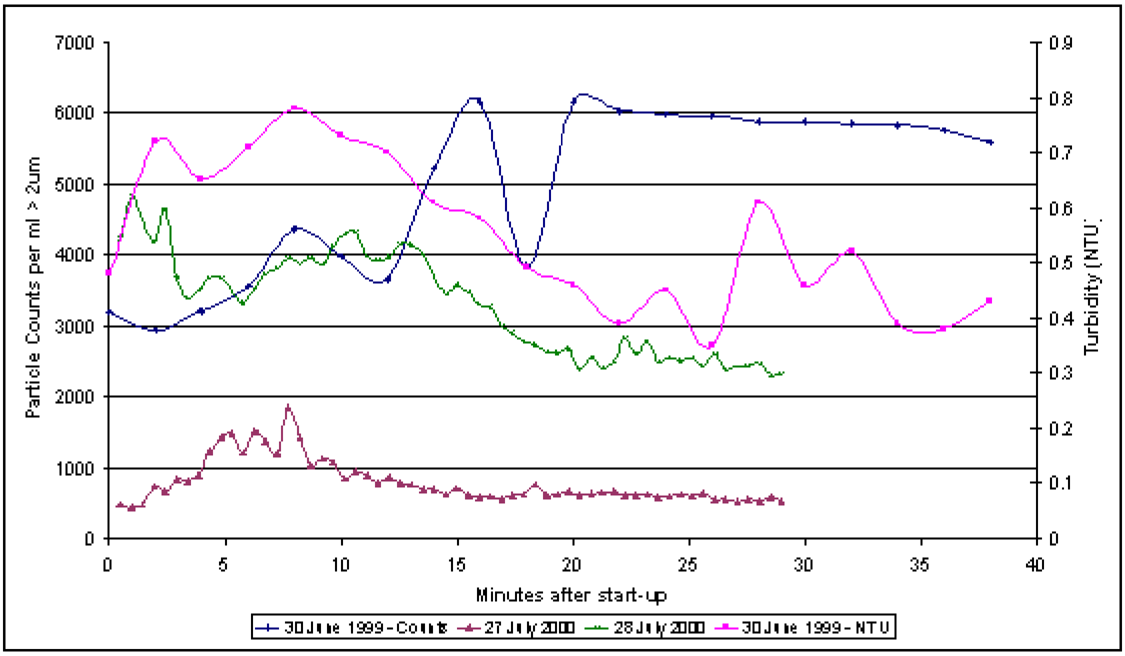

Figure 12.4(f) : Filter Start-up Monitoring at Plant 2

12.5 Conclusions and Recommendations

The results returned from both the pilot plant study as well as the work done at full-scale plants indicate that particle counting is more sensitive, and therefore better suited, to the measurement of filter ripening curves than turbidity.

Maintaining and ensuring repeatable filter runs have proved difficult and led to results that are not entirely satisfactory. This problem has also hampered other researchers (Tobiason *et al.,* 1996). Despite this, the results generated during this study illustrate that some benefit is to be gained from the manipulation of filter start-up. Further study must concentrate on repeat tests over extended periods in order to find the median response of filters to varied control procedures. Repeats of three or four runs are not sufficient due to the inherent sensitivity of filtration processes to small variations in several parameters. The authors still consider pilot-plant tests to be more appropriate than full-scale tests as this allows more control over experimental variables. Other aspects that must be considered are the conditioning of the filter bed before the tests are commenced and the point at which the filter runs are terminated. In order to have repeatable results, it is imperative that the filter run is started with the media in the same state of “cleanliness” as during the previous run. This is a particularly difficult aspect of the experiment as it defies direct measurement.

An aspect of this study that posed particular concern, was the use of water from the municipal reticulation system. The quality of the water changed from day to day and this impacted negatively on repeatability. The use of other water sources was precluded due to the size of the experimental setup. Future workers must attempt to standardise the water quality used in these experiments.

12.6 References

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STANDARDISATION OF THE USE OF PARTICLE COUNTING IN THE POTABLE WATER TREATMENT INDUSTRY IN SOUTH AFRICA WITH CASE STUDIES

PART D : CONCLUSIONS AND PROJECT APPRAISAL

Chapter 13

CONCLUSIONS, PROJECT APPRAISAL AND RECOMMENDATIONS

Several objectives were set at the outset of this project. The level of success of the project in achieving these objectives are presented below.

Evaluation of available particle counters, configuring of the counters and standardising reporting procedures.

This objective has been obtained. A complete summary of particle counters, suppliers and methodology is provided. In addition to this, guidelines are given on the handling of data. To date, this aspect of particle counting has been neglected in literature. The report also shows how the data can be reduced to fewer, more meaningful, data points that can be used in the day-to-day operation of potable water treatment plants. From this discussion, it is clear that particle counters and particle counting technology will have to undergo further development before it reaches the level of standardisation that turbidity measurement technology has reached. Users will have to be aware of the limitations of particle counting until then. This said, careful and conscientious use of particle counting will allow users much more insight than turbidity alone, when water clarity issues are investigated.

Establishing baselines in terms of general process performance using particle counters.

Several plants were selected by the project team and these plants were monitored during several site visits. These visits generated more than 126 process monitoring runs. This ensured that sufficient datasets were available for analysis. Analysis proved to be extremely complex and it was decided to confine the discussion in this report to the characterisation of the raw and filtered waters from these plants. This has been successful and introduced a novel approach in classification of the water. This is particularly true for the raw waters. The results from this study indicate that it is possible to define the character of raw water more succinctly than with turbidity alone. Complete classification of raw waters and further development of process selection criteria based on raw water particle count information must be considered as a next step. An investigation of particle counts during the treatment process is also necessary. This will also provide a diagnostic tool for treatment plant performance evaluations.

Study of the effect of flocculation regime (charge neutralisation vs. sweep floc) on final water quality.

This study has proven to be extremely complex and has served to highlight some of the shortcomings of particle counters, ie. the measurement of coagulated and flocculated particles. These particles are inherently unstable and it is virtually impossible to generate dependable counts on these samples. This is attributed to fundamental shortcomings of particle counters as well as the transient nature of the coagulation and flocculation process that makes the tracking thereof very difficult. This study returned inconclusive results.

Evaluating filter performance under various hydraulic control regimes.

Maintaining and ensuring repeatable filter runs have proved difficult and has led to results that are not entirely satisfactory. This problem has also hampered other researchers (Tobiason *et al.,* 1996). Despite this, the results generated during this study illustrate that some benefit is to be gained from the manipulation of filter start-up. Further study must concentrate on repeat tests over extended periods in order to find the median response of filters to varied control procedures. Repeats of three or four runs are not sufficient due to the inherent sensitivity of filtration processes to small variations in several parameters. The writers still consider laboratory scale tests to be more appropriate than full-scale tests as this allows more control over experimental variables. Other aspects that must be considered are the conditioning of the filter bed before the tests are commenced and the point at which the filter runs are terminated. In order to have repeatable results it is imperative that the filter run is started with the media in the same state of “cleanliness” as during the previous run. This is a particularly difficult aspect of the experiment as it defies direct measurement.

Throughout the experimental work it was found that particle counting was the more sensitive measurement procedure when compared with turbidity. Turbidity failed in most cases to show up the ripening curves. It is suspected that the turbidity curves, along with the particle counting curves will benefit from further refinement of the experimental procedure.

To investigate the use of particle size analysis as a control parameter for the optimisation of water quality, and to compare this to the control using zeta potential or streaming current.

It was found that the filtered water particle count did not change significantly under different coagulant dose conditions. This was primarily due to filtered water samples being taken during a period of "best performance" of the filter. The settled water particle count did change slightly with coagulant dose and an increase in the particle number in the larger size fractions was observed.

There is however normally a long time delay between the point of coagulant addition and the point where particle count would be measured (either after sedimentation or after filtration) which makes it impractical to use this as a response parameter to changes in water quality. Streaming current measurement lends itself as a better tool for optimising coagulant dose as it shows a significant response to changes in dose and can be positioned much closer to the dosing point.

To investigate the relationship between particle size, Cryptosporidium and Giardia in filtered waters from water treatment plants.

It was impractical to monitor filtered water effluent and to try to compare the particle counts to numbers of *Cryptosporidium* oocysts and *Giardia* cysts in the filtered water. Accuracy of analysis and the serious implication of reporting *Cryptosporidium* as being present in final water (if it is actually there at all) meant that these protozoans had to be spiked into samples of filtered water. By spiking though, sufficient numbers could be injected to obtain a response from the particle counter and the size range in which these organisms can be detected was obtained. *Cryptosporidium* oocysts were detected in the 2-5 µm size range whereas *Giardia* cysts were detected in the 5-10 µm size range. The standards of *Cryptosporidium* and *Giardia* that were obtained were however not 100% pure and other particles in the standard resulted in the total counts being more than 3 times higher than what was expected. This confirms that should there be *Cryptosporidium* and *Giardia* in the filtered water, the particle counters may detect an increase in the particle count in a specific size range, but does not indicate that if there are counts in any size range that there definitely is *Cryptosporidium* or *Giardia* present in the water.

To study the effects of pre-treatment processes such as ozonation on the clarity of filtered water by measuring the particle size after filtration.

During an investigation at the Wiggins Water Treatment Works, the particle count after filtration was not found to be significantly affected by pre-ozonation when compared to operation without ozonation. Although the filtered particle count was more consistent and the quality of the water was more easily maintained using pre-ozonation, when the plant had been optimised for operation without ozone the filtered water particle count achievable was found to be lower.

The chemical and physical conditions in the raw water affected the performance of the plant to remove particles from the water. Treating water with a low raw water turbidity bentonite was required to adequately maintain plant performance. The dose of bentonite, the pH at coagulation and temperature of the raw water appeard to affect the filtered water particle count. Pre-treatment and conditioning of the water before coagulation can therefore affect the filtered water quality.

To consider the use of particle size analysis together with Computational Fluid Dynamics for the optimisation of water treatment equipment.

This objective has not been completed. Without the knowledge of particle counting in South Africa and without experience in the use of such instruments, the methodology of how to combine these techniques was not available. CFD is a tool for modelling flow through a plant and can be used to improve inefficiencies in the operation of equipment. Particle counting is probably best applied to filtered water where inaccuracies as a result of coincidence and the limitations of instruments (i.e. concentration limits) are not significant. With hindsight, as the operation of particle counting in South Africa is better understood, there are probably more appropriate analyses which could be used more effectively with CFD for optimising water treatment equipment.

Other work

Over and above these objectives, work was performed in assessing the calibration of the particle counting instruments using monodisperse latex standards from Duke Scientific as well as a Standard Reference Material 2806 used in the calibration of particle counters in the hydraulic fluid industry. These exercises were interesting in that we found it almost impossible to perform both a size comparison as well as a count comparison using the technique of injecting particles into a filtered water. It is proposed that for the objective of checking the calibration of an instrument a special test bench is used where a sample of known particle count and size can be circulated through the particle counter and a reliable consistency in the method and result can be guaranteed.

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