Cover Page

Paper for ICWH 2002 – September 23, 2002

Title: Beyond Turbidity – A Quantifiable Analysis of Solids in Drinking Water

Abbreviated Title: A Quantifiable Analysis of Solids in Drinking Water

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Abstract

Achieving a comprehensive understanding of the solids content in raw and finished water provides a basis for continued improvements in water treatment processes and reduction in biological hazards. Most instrumentation used for solids analysis uses optical technology. The interaction of optical beams with suspended solids in water depends on the size distribution, shape, refractive index and optical absorption spectrum of different particle populations present in the sample. Although standards-based turbidity measurement is a fundamental tool for water treatment regulation and plant optimization, it makes no assumptions on individual particle properties but compares, empirically, the light scattered and absorbed in a sample volume to a similar volume containing a reference material. Particle counters, primarily based on light obscuration or scattering from individual particles, have been introduced to estimate particle size and distribution. A primary motivation for particle counting has been to isolate particles similar in size to pathogens such as Cryptosporidium. Particle counters base their size estimations on calibration material with specific optical properties. In practice, both source and treated water contain particles with unknown sizes and a distribution of optical properties. As a result, difficulties are encountered in correlating results both between different instruments measuring the same sample and from the same instrument used in different locations. The paper describes research aimed at developing a particle imaging technology which can identify specific sub-populations of particles using any combination of size, shape, structure and optical properties. Initial results show that particle imaging can operate on-line and provide sufficient sensitivity to detect all solids including those which are virtually invisible. The paper describes the challenges encountered in developing the technology and presents initial results obtained from calibration samples and from raw and treated water.

1.0 Introduction

Measurement and characterization of suspended solids is a basic component of water treatment. The solids content in raw input waters largely determines treatment requirements. Activity in Ontario to identify, through the presence of larger particles, groundwater under the direct influence of surface water is a recent example (*Ref: O'Connor 2002, Gehrels, 2002*). Characterization at various in-process treatment stages such as coagulation, flocculation, filtration and disinfection is fundamental to the design, operation and quality control of treatment facilities. Measurement and regulation of solids content in finished water is an essential element of the standards which protect public health and safety. Despite the importance of solids characterization to water purification, the range of instrument technologies available to assist in this task this has been limited. This paper is concerned with the development of a new instrumentation design utilizing digital imaging technology for application in the characterization of solids in drinking and wastewater.

2.0 Solids in Water

Solids in water consist of particles in suspension. In addition to their obvious aesthetic impact on taste and appearance, particles are well known to have a considerable range of adverse effects *(Ref: DWS Secretariat, 2001)*. These include effects on water chemistry in processing, staining and corrosion, increased disinfection demand and byproducts as well as adsorption and transmission of toxins and diseases. Individual particles may be composed of one or more of a wide range of materials. The particular effect of the particle will depend on the material, physical properties and concentration of the particle involved. Table 1 provides a summary of some of the particle materials and properties which might be found in surface water. Components in the mixture will vary widely with location, season and weather. Since this raw water "soup" is the starting point from which clean and verifiably safe drinking water must be produced, it is desirable to have its components characterized quickly and economically.

Particle Type	Possible Size range	Density Range	Optical Absorption	Typical Refractive Index*	Scattering Power
Inorganic (silt, clay, natural precipitants)	Sub-µm to 5 µm **	2 to 4	Very high to very low depending on elements present	1.4 to 1.8	High
Organic Detritus	Sub-µm to 100s of µm	~ 1	Generally high	Various	Low
Biological Organisms	Sub-µm to 100's of µm	~ 1	May be very low	May be close to 1	Various
Mixed organic/inorganic		Various	Non-uniform	Non-uniform	Various

** Refractive Index of water is approximately 1.33 at 15°C*
*** Larger particles settle quickly*

Table 1 - Representative Properties of Particles Typically Found in Surface Water

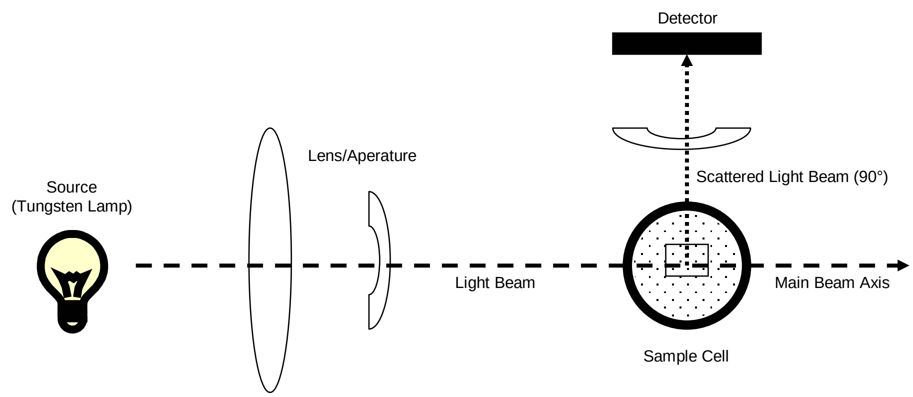
3.0 Present Analytical Techniques and Tools

Direct methods of particle characterization such as micro-filtration and manual microscopic examination are laborious, difficult to interpret and impractical for process applications where rapid measurements are needed. As a result, test instrumentation which automatically measures suspended solids content is required.

3.1 Turbidity

The turbidity parameter has been used for many years as the basis of a rapid and simple test which provides an approximate indication of solids content. Turbidity test methods and equipment measure the intensity of scattered light resulting from the interaction between an optical beam, derived from a tungsten lamp or laser, and the sample under test. The intensity of the scattered light, which depends both on absorption and scattering in the optical beam path, is measured perpendicular to the main beam axis (*Ref: Sadar 1998*) The instrument is calibrated with a reference material of standard turbidity. Notwithstanding its empirical basis, standards-based turbidity measurement continues to serve as the fundamental tool for water treatment regulation and plant optimization.

Figure 1 shows the principles of turbidity measurement and lists some strengths and limitations *(Ref: Sadar 1998 1999).*



Advantages/Strengths	Disadvantages/Weaknesses
• Widely accepted in Water Treatment Industry • Standards-based • Simple, rapid measurement • Economical • Single parameter result	• Qualitative measurement only • No information on particle size, distribution • Equivalent results can be obtained from samples having widely different particle sizes and distributions • Oriented to submicron particles and less sensitive to small or anomalous concentrations of larger particles (~10 µm)

Figure 1 – Turbidity Overview

The interaction of optical beams with suspended solids in water depends on the size distribution, shape, refractive index, concentration, wavelength and optical absorption spectra of different particle populations present in the sample. Since turbidity measurement takes no quantifiable account of these properties, samples having quite different concentrations of solids, but of different materials, can give the same turbidity value. Turbidity measurement is also less sensitive to small but anomalous concentrations of larger particles which are of particular concern if they are present in water having otherwise low particle content and turbidity.

Although the relationship between turbidity and particle concentration is highly indirect, it is of interest to note that the existing Canadian turbidity guideline of 1 NTU could be met by water having as many as 6000 particles per cc greater than 2 µm. The newly proposed Canadian limits for finished water produced by chemically assisted filtration are < 0.3 NTU or < 0.2 NTU for input water > 1.5 NTU and < 1.5 NTU 90% of the time *(Ref: DWS Secretariat 2001).* Water of 0.2 NTU can have approximately 20 particles per cc > 10 µm, 100 particles/cc > 5 µm and 1000 particles/cc >2 µm diameter.

3.2 Particle Counting

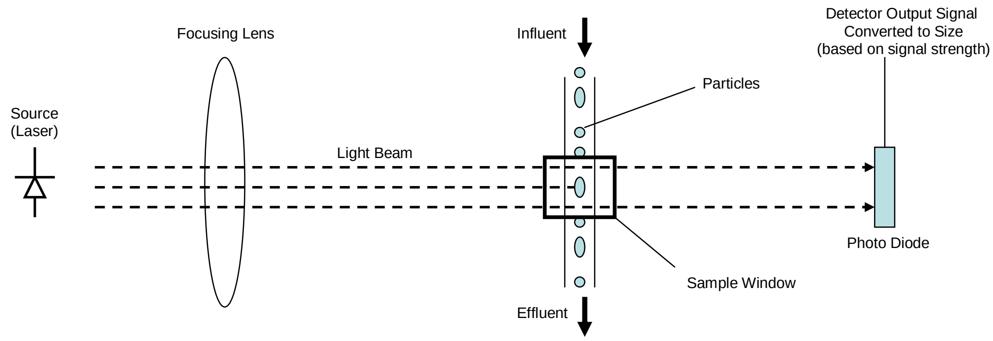
To overcome some of the limitations of turbidity measurement, particle counting technology originally developed for other industries has, more recently, been applied in water treatment *(Ref: Hargesheimer et al 2000)*. The particular technology most commonly employed relies on light obscuration by individual particles as they pass, one at a time, through an optical beam. Size estimations are based on the fact that each particle will block or scatter optical power from the beam by an amount which is proportional to the particles equivalent diameter. The proportionality factor is derived and periodically recalibrated using a calibration material containing particles of known size. The material most commonly used is polystyrene latex with a refractive index of 1.59 and a transmission ratio (average) of 85.2%. Equivalent diameter is defined by each instrument manufacturer.

However, unlike the particle materials encountered in other industries, both source and treated drinking water contain particles having a broad distribution of optical properties which are quite different than those of the calibration particles. If, as a result of these differences, a particular particle interacts with the beam more strongly or more weakly than the calibration particle of equivalent diameter, the instrument will provide a size estimation which is too large or too small respectively. As a result, sizing data on practical samples is unreliable *(Ref: Hargesheimer et al, 2000, Sadar 1999)* and difficulties are encountered in obtaining consistent results both between different obscuration particle counters measuring the same sample and from the same instrument used in different locations *(Ref: Haught 1999)*. In reported particle counting studies *(Ref: Huck et al 2001)* which compare log removal rates of particles and cryptosporidium oocysts, the observed differences may be, in part, due to differences in optical properties of particle families.

Notwithstanding these difficulties, obscuration particle counting technology does provide valuable additional insight beyond turbidity testing. The technology, when correctly applied, has allowed significant improvement to be made in treated water quality *(Ref: Hamilton et al 2001).* The use of particle counters now forms part of the compliance regulations in a number of jurisdictions (i.e. Alberta, New Zealand) and is being actively evaluated in others.

However, due to the technical shortcomings highlighted above and the difficulty in formulating a regulatory standard and treatment process in response to particle count data, introduction of particle based regulations has been slow. In many instances, particle counters are deployed in water treatment plants (WTP's) but since there is no regulatory standard on maximum particle count/size and no associated operational procedures triggered by particle counts, their data is not used in active management of the WTP.

Figure 2 shows the operation of obscuration particle counting and lists the strengths and limitations of the technology.



Advantages/Strengths	Disadvantages/Weaknesses
• Quantitative measurement • Extensive research to support application in water treatment industry • (Some) regulatory acceptance	• Indirect measurement technique • Measurements (count, size) are imprecise since actual particle properties differ from calibration material • Particle concentration limits due to coincidence error • No information on sub-populations • Fouling and high maintenance • Inconsistency of results between different instruments • Stable flow rates are required for accurate concentration measurements

Figure 2 – Particle Counting (Obscuration) Overview

4.0 Value of Particle Characterization

The question may be asked as to whether, in view of the uneven industry response in adopting obscuration particle counting, an increasingly detailed characterization of the suspended particle populations is likely to be of practical value in water treatment. In fact, studies relating particle size (as measured by obscuration or microscopy) to observed effects in filtration performance, disinfection and biological safety testing have shown valuable and even dramatic correlations between properties and apparent particle diameter. These could not be observed using turbidity measurement. Examples include:

- Small but abnormal concentrations of larger (5 to 12 µm) particles in ground water of low turbidity indicate contamination by surface water *(Ref: Hamilton et al)*
- Measured log removal of surrogate particles of similar size as surrogates for removal of Cryptosporidium oocysts *(Ref: Huck et al 2001).*
- Larger (7 µm approx.) organic and mixed inorganic/organic particles mask pathogens from UV inactivation *(Ref: Jolis et al)*
- Particles of similar size mask chemical inactivation *(Ref: Silverman et al)*
- Organic particles mask more effectively than inorganic particles *(Ref: DWS Secretariat)*
- Larger particles reduce measured coliform counts by masking *(Ref: Parker et al)*
- Presence of larger particles indicates filter breakthrough *(Ref: Hamilton et al)*
- Particles adsorb metals, herbicides and viruses *(Ref: DWS Secretariat)*

- Presence of particles greater than membrane cutoff size indicative of leaks in membrane systems *(Ref: Colvin et al)*

Based on such observations, it appears that a treatment strategy which involves measuring and maintaining an acceptable particle distribution "signature" in a facility and reacting quickly to any anomalous increase in the number of larger particles would make more sense than relying solely on turbidity measurement. To demonstrate the relative insensitivity of turbidity measurement to larger particles, we performed an experiment where a sample of water measuring 0.06 NTU was spiked with 1% additional concentration of raw water from a small area river with a turbidity of 3.85 NTU and containing many large biological particles. The resultant mixture measured an identical 0.06 NTU.

In order to be of value, particle measurements must be accurate, consistent and repeatable. In view of the remaining limitations of obscuration particle counting, a further evolution of instrument design remains desirable. The balance of this paper will describe development efforts made by the authors towards this objective.

Some key characteristics for an analyzer which could provide comprehensive analysis of solids in water are summarized below.

- High sensitivity and resolution for accurately characterization and measurement of the equivalent diameter of ALL particles in sample
- Ability to differentiate and count sub-classes of particles having different properties such as diameter, transparency or shape
- Volume sampling rate which provides adequate statistical accuracy and fast response
- Capability for in-line application
- Ability to consistently characterize both raw water samples having very high solids and finished water having very low solids with the same device
- Capability for data analysis and output in a simple form suitable for pass/fail measurement
- Acceptable cost for widespread application
- Rugged, easy to use and insensitive to fouling

To date, no available instrumentation technology is able to fulfill all of these criteria.

5.0 Particle Image Analysis

Particle analysis and particle counting are a longstanding and active science. A variety of instrumentation technologies employing a broad range of physical principles have been applied to the characterization of different types of particles in different environments and industries. In reviewing recent developments for potential application to a new instrument for water particle analysis, it became apparent that techniques based on optical imaging were becoming increasingly powerful and economical.

In principle, particle imaging analysis technology appears to have considerable potential for water solids analysis. In practice, the specific requirements listed above provide many technical challenges. Since none of commercially available analyzers developed for other applications meets the desired performance and cost requirements, we have been carrying out experiments to evaluate the feasibility of using this technology.

The principles of operation of a particle analysis system based on imaging are shown in Figure 3.

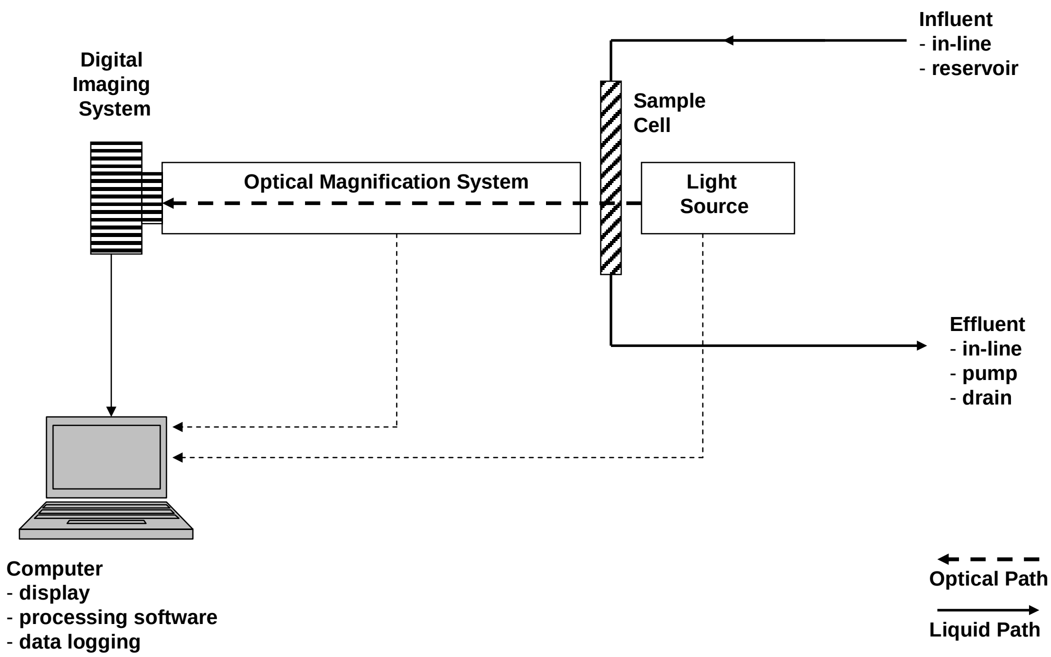


Figure 3 - Imaging System Overview

The sample volume is illuminated by an optical beam which interacts with the particles. The resultant beam is imaged by an optical system with selected magnification onto a pixel detector array in an electronic camera. Provided the particle interacts sufficiently strongly with the beam, an image will be produced on the pixel array. Every pixel within the image will provide a signal which is different from that of pixels which experience only the background illumination. The signal from each pixel in the array is collected and analyzed by the algorithm contained in the system software. The equivalent diameter of the particle is calculated from the number of pixels within the image area and the known magnification. Unlike obscuration particle counters, the computed equivalent diameter does not depend on the strength of interaction of the particle with the optical beam as long as the particle can be optically resolved. Stable flow rates are not required for accurate measurement. No calibration is required. The technique should also be capable of differentiating, on the basis of image contrast, between particles with different optical properties such as strongly absorbing carbon or weakly absorbing silica. Particle images may also, in principle, be segmented on the basis of shape. Since each exposure frame can contain many particles, analysis of samples with high solids concentrations is possible.

6.0 Technical Considerations

Key technical challenges to be overcome in the design of the imaging analysis system for drinking water and wastewater applications include

- The analyzer must process particles with the full range of optical properties of the different particulate materials which are present in water samples.
- To allow an adequate volume sampling rate for statistical accuracy, analysis must be made on particles in dynamically flowing samples.
- For the same reason the largest possible volume of water must be processed in each frame acquired by the digital camera.
- Particles with diameters ranging from 2 µm to 500 µm must be accurately measured. A broader range from submicron to millimeter would be desirable.
- The design must use components which would allow the system to be sufficiently economical.

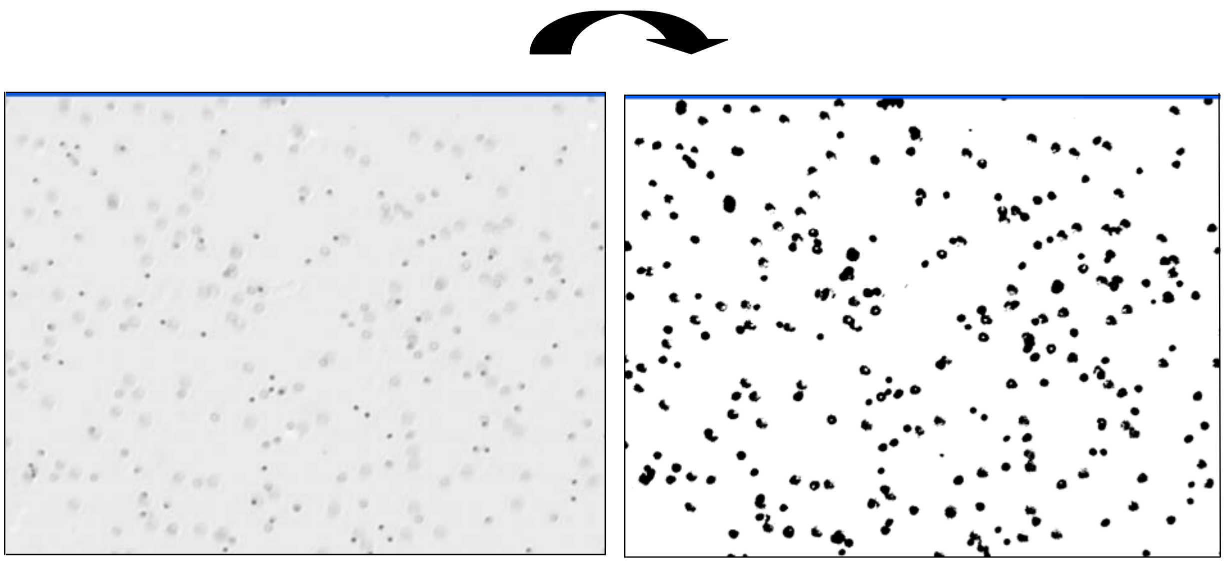
These requirements, in turn, pose challenges to the main functional elements of the system.

- The optical mapping/magnification systems must provide high contrast images of the many different types of target particles while maximizing imaging volume and minimizing interference due to scattering and stray light.
- The camera must provide low noise, high resolution to minimize pixellation errors, a high pixel reading speed along with a fast readout interface to the analysis computer.
- The wavelength, intensity and geometry of the particle illumination must provide adequate contrast for sub-micron particles ranging from highly transparent to highly opaque.
- The software algorithm must accurately and quickly size the many particles in each sample frame while removing undesirable noise and artifacts
- The overall frame rate, speed of software analysis, sample renewal rate and optical sampling volume must support measurements on flowing liquids with an adequate volume sampling rate.
- The system must be capable of operating on-line and in real-time.

7.0 Preliminary Results

Following a period of component selection and development, we have constructed an experimental analyzer and have started to evaluate the potential of the technology. Our initial experiments have been to analyze suspensions of particles of known and unknown size and materials. Various concentrations of these particles are prepared either by weighing and dilution with particle free water or by direct microscopic counting. Analysis results provided by the system can be compared with the known contents.

The ability of the analyzer to resolve and accurately count mixtures of different particles is demonstrated in Figures 4 and 5 which shows gray-scale, or preprocessed, and processed images of a suspension (a mixture of polystyrene and boro-silicate glass particles) and compares the known concentrations with those measured by the analyzer.



Gray-Scale Image ***Processed Image***

Figure 4 – Images of Polystyrene/Boro-Silicate Glass Mixture

Calculated versus Measured Concentrations

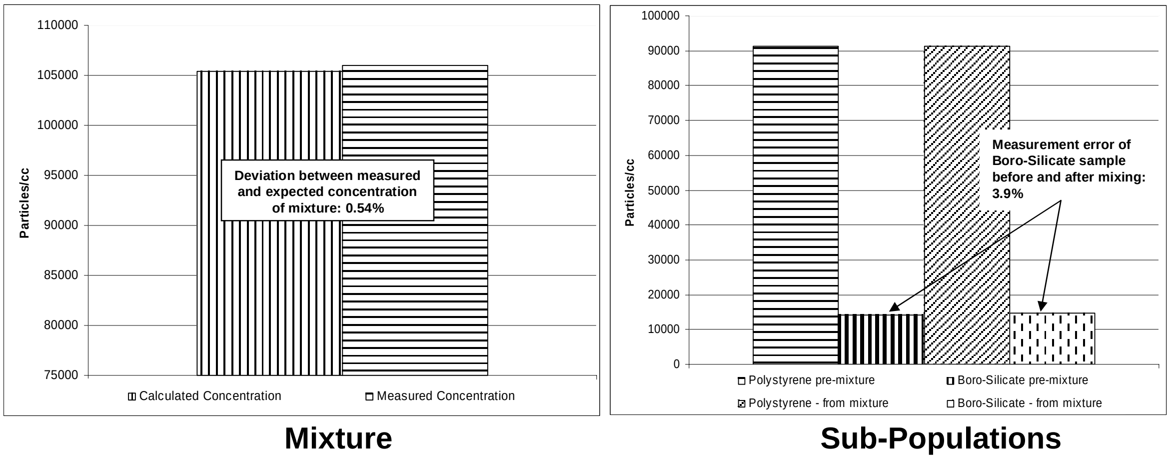
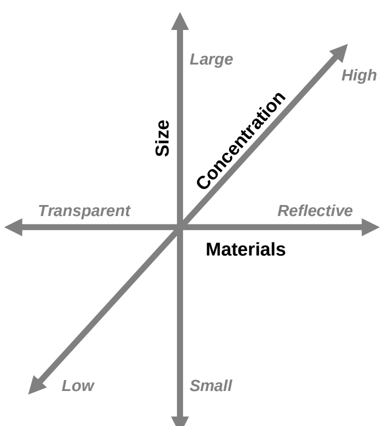


Figure 5 – Mixture of Polystyrene and Boro-Silicate Glass

Figure 6 summarizes the range of particle equivalent diameters, concentrations and particle materials over which we have operated the device.



Observed Particle Analysis Capabilities

Size: <2 µm >500 µm

Concentration: 1/cc 1,000,000/cc

Materials: Silica, various clays, boro-silicate glass, carbon, various organic, polystyrene

Figure 6 – Operating Range of Prototype

Figures 7, 8 and 9 show data collected from tap water (turbidity measured at 0.03 NTU), from a small area river (1.8 NTU) and from a sample prepared from a pottery sink trap (423.0 NTU).

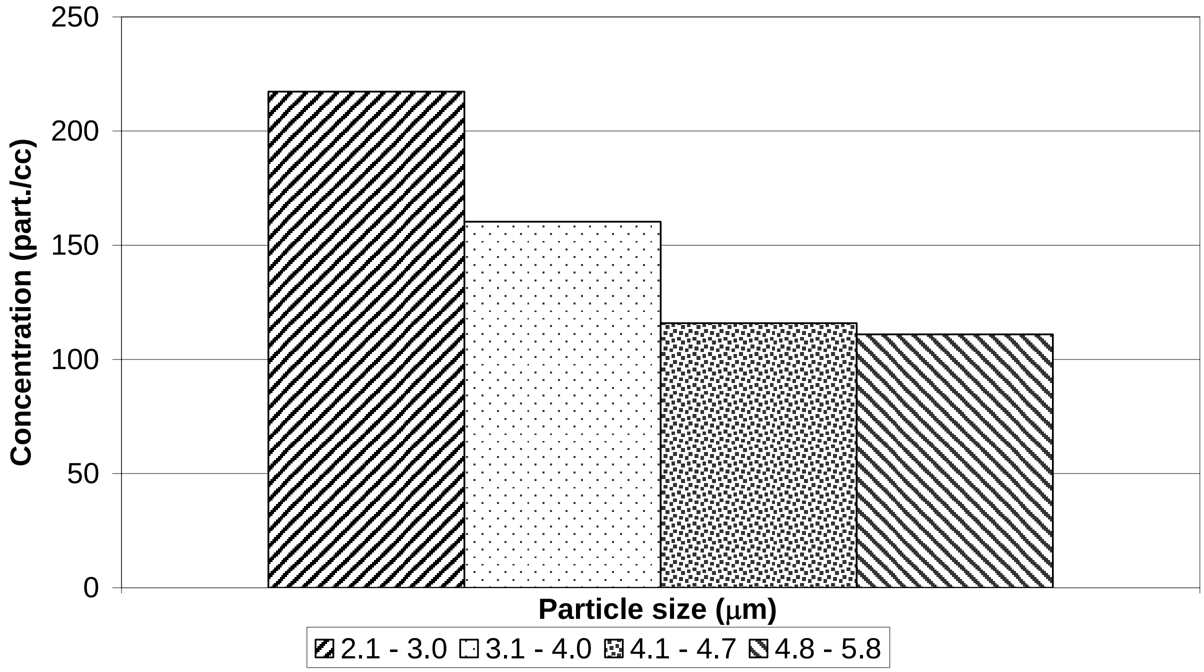


Figure 7 - Tap Water (0.03 NTU)

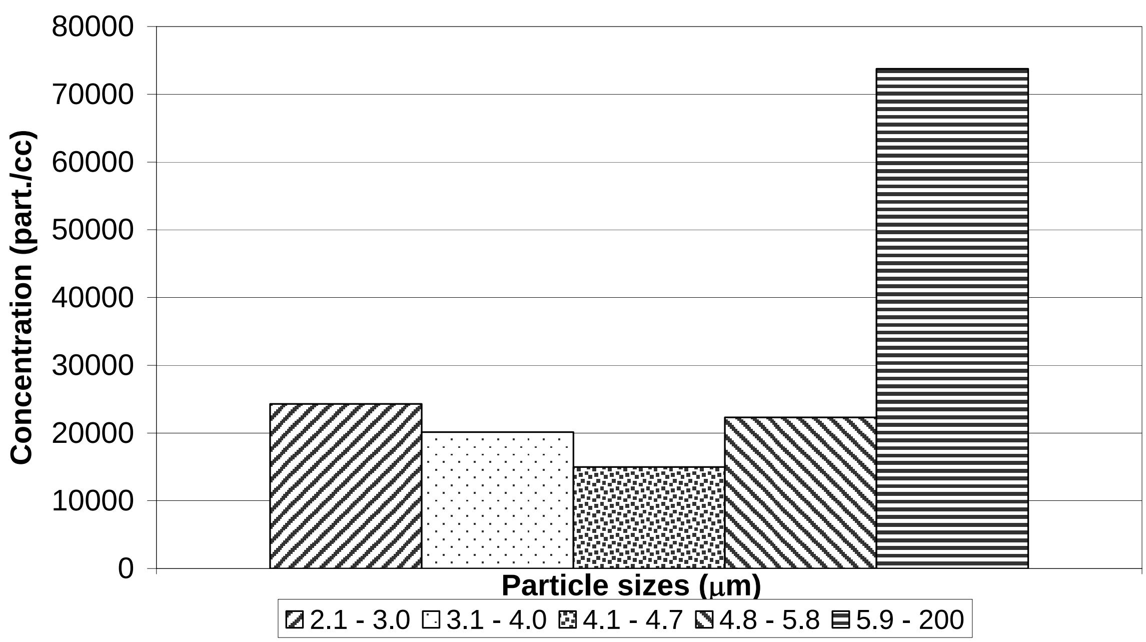


Figure 8 - Jock River Water (1.8 NTU)

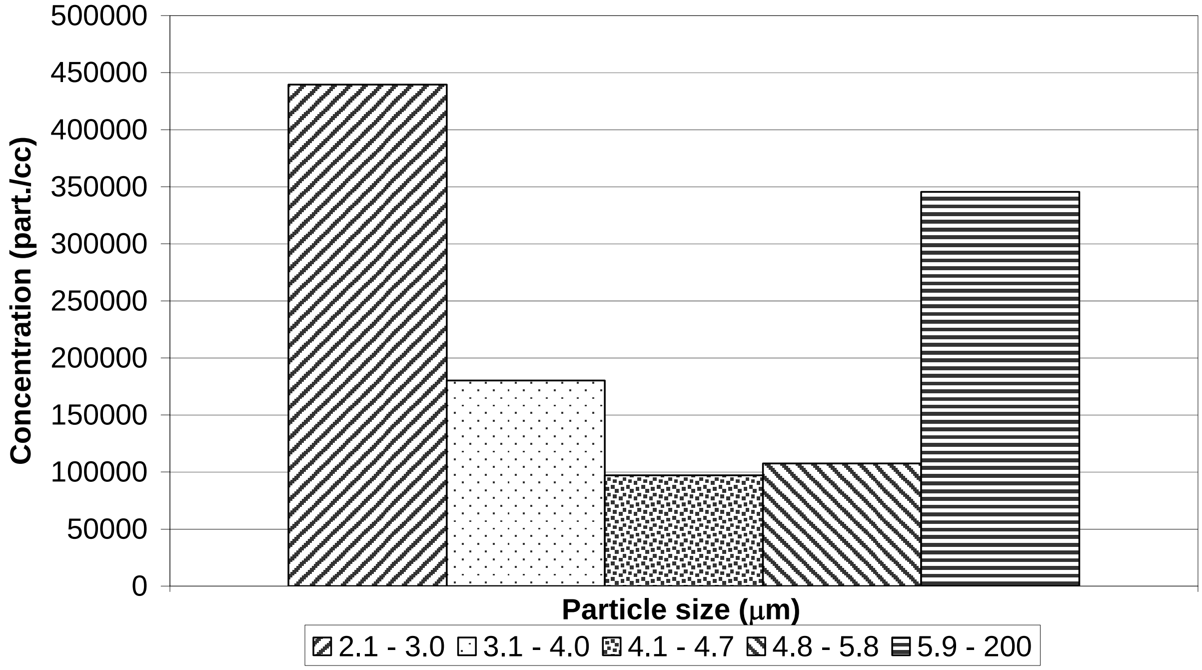


Figure 9 - Pottery Sink Trap (423.0 NTU)

8.0 Conclusions

Our preliminary work has indicated that dynamic particle imaging is a promising technique which can provide new insights into the suspended solids of interest in water treatment. Our prototype device has been shown to be capable of resolving and measuring particles having a wide range of sizes, materials and concentrations encountered in both raw and finished drinking water and in wastewater. The data obtained from the device goes considerably beyond that obtainable by turbidity or particle counting measurement. With further refinement and qualification, we believe that the technology incorporated into the prototype will form a basis of a practical instrument which will be a valuable addition to the range of solids characterization devices available to the industry.

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