

Authors: Ms Roshani Shrestha, Dr Ian Brodie

Presenter: Ms Roshani Shrestha

Position: PhD Candidate

Organisation: University of Southern Queensland

Contact Details: Ph +61 7 4631 1940 Fax +61 7 4631 5581 Email:

Roshani.Shrestha@usq.edu.au

Title: A Protocol for the Use of Particle Counting in Stormwater Suspended Solids Analysis

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Abstract: Particle count technology that has been used for more than two decades in potable water testing is finding increasing application in stormwater as well. Particle count is a two-dimensional measurement of particle number by size, therefore it can potentially produce accurate and precise information on water quality, system performance and stormwater treatment efficiencies compared to conventional TSS, SSC and turbidity measurement methods.

In this study, a protocol for using PAMAS S4031, a particle counter based on light obscuration has been developed. Most of the modern particle counter instruments currently being adapted for liquid analysis are designed with the narrow spectrum analytical requirement in mind (for example, counting blood cells and fuel spray droplets size), so it should be noted that these instruments have limitations. In this paper, the limits and capabilities of available instrument technology will be presented in detail to make the best use for analysing suspended particles in stormwater.

A silica powder LANG LANG SILICA 60G, obtained from highly pure sand with known particle size distribution (PSD) in term of percentage of mass and uniform density of 2.66 gm/cm^3 was used for the verification of the particle counter. Initially two different methods, namely sieve analysis and settling column, were used to confirm the given PSD of silica. Wet and dry sieving were performed to fraction particles in the range between $45\mu\text{m}$ to $250 \mu\text{m}$. Settling column analysis was used to fraction particles larger than $2\mu\text{m}$ whereas the pipette method was used for smaller particles between $2 \mu\text{m}$ to $62.5 \mu\text{m}$. The PSD obtained from these methods were compared with the given PSD. Based on

particle density information, particle counts were calculated mathematically for given PSD and were compared with particle count obtained from PAMAS particle counter. It was found that although the measurement range was between 1 to 100 μm , the instrument was most effective in measuring particles smaller than 10 μm only.

The protocol will thus mainly focus on fine particles < 10 μm . This study comprises of series of monitoring trials, conducted at different concentrations, flow rates, instrumental set ups etc. The protocol is expected to be fast and easy to use and can be used to evaluate stormwater quality and treatment performance. It can also assist in the selection of treatment systems on a more consistent basis.

1. INTRODUCTION

Suspension of solids in stormwater has been identified as a cause of water quality deterioration within receiving waterbodies as it can lead to loss of aquatic habitat, block stormwater infrastructure, modify flow conditions and transport harmful pollutants (Furumai et al. 2002; Brodie 2007; Roesner et al. 2007). Presence of suspended solids makes water turbid which limits light penetration affecting aquatic life and reducing the aesthetic value of waterbodies (Bhargava & Mariam 1994; Osmond et al. 1995; Wood & Armitage 1997).

A large number of treatment systems are commercially available to treat suspended solids in stormwater. Substantial differences are evident in the way the performance of these treatment systems are evaluated and presented. Various measures to indicate treatment performance are in use and this makes a comparison between different systems difficult. However, the most commonly used indicator in evaluating treatment systems is the suspended solid measurement as suspended solids itself is a pollutant of concern and is easy and inexpensive to measure. Total Suspended Solids (TSS) and Suspended Solids Concentration (SSC) analysis methods are commonly used (Clark et al. 2008). In addition, indirect methods such as turbidity are also being used as this is a convenient instrument-based measurement. However, problems exist with all of

these methods. For example TSS and SSC methods include all the solid material into a single class (Roesner et al. 2007). 80% TSS load removal of large particles would not have the same water quality outcome as 80% removal of fine material. Fine particles can have a significant impact on the transport and fate of chemicals due to their higher specific surface area and surface reactivity. Hazardous pollutants are found to be associated with fine particles (Chebbo et al. 1992; Pechacek, 1994; Pitt et al. 1995; Sansalone and Buchberger, 1997; Furumai et al. 2002). In addition to this TSS and SSC are tedious to measure, extraction of a representative subsample is a major concern in case of TSS. Turbidity is a measure of light scattered by suspended particles but does not relate information regarding the concentration or physical size of the suspended material (O'Leary et al. 2003).

As an alternative, particle count as a water quality indicator can be an appropriate alternative to TSS, SSC and turbidity. Kavanaugh (1980) and Mortiz and Hoffman (1993) mention the use of particle count as a more sensitive measure of particle removal efficiency than the turbidimetric measurement technique. As particle counting is a two-dimensional measurement of particle numbers and sizes, it can produce accurate and precise information even below the range of turbidimetric sensitivity. Filtration showing identical turbidities could be distinguished easily with particle count analyses (Mortiz and Hoffman 1993).

In spite of the extensive application of particle counting in drinking water and wastewater treatment (Greenberg et al. 1995), its use in stormwater is relatively new. Minimal research has been done regarding particle counts in stormwater, which includes the work done by Pechacek 1994; Characklis et al. 1997; Sansalone et al. 1998; Li et al. 2006 and others.

Researchers have utilized particle counting to elucidate first flush effects, contribution of particle number based on land use and size distribution of particles in stormwater. Sansalone et al. (1998) and Li et al. (2006) used particle counting to demonstrate first flush in runoff from highway pavements. Similarly, Characklis et al. (1997) studied the change in particle number in surface water during storms and found that particle numbers smaller than 2.5 μm increase by 0.4-log scale over background levels. Although particles in this fraction do not constitute a large proportion of the mass of suspended solids, they contribute significantly to the total surface area of solids (Cristina 2001). Li et al. (2006) monitored particles ranging between 2-1000

μm in runoff from highway and reported that more than 90% of particles were less than 10 μm in diameter which is comparable to the results indicated by Pechacek (1994). In Li's research, particle number concentration was correlated with total suspended solids and turbidity, suggesting that these parameters could be useful surrogates for each other (Li et al. 2006).

At present there are three particle counting techniques applied in water industries, namely light scattering, light obscuration and electrical resistance - also referred to as Electrical Sensing Zone (ESZ) or Coulter counter. Particle counters both count and size individual particles as they flow through a sensing zone (Hargesheimer et al. 1995). The most commonly applied technique in water industries is the light obscuration, or light extinction, type (Hargesheimer 2000, Bridgeman 2002). Light based techniques are an indirect measurement of particle count, based upon the theories of light scattering and light absorption, both of which remove energy from the incident light, decreasing the intensity upon the receiving photo detector (Bridgeman 2002). By comparison, ESZ devices are true particle counters.

Not all particle counters measure correctly and accurately. Several studies show that the same test repeated within different countries yield varying results (Van Gelde et al, cited in Cerenio et al.2002, p.3-1).This is due to the variation in the way the counters are constructed and calibrated. In order to address this issue several standards have been published. But there is data variability generated by an individual counter because of the different ways that the instrument can be setup, and also the experimental procedure used by the operator. This paper will discuss the development of a protocol for the use of PAMAS S4031 particle counter for the analysis of suspended solids . This will assist to obtain reproducible data and a meaningful comparison of the data generated by various operators.

2. METHODOLOGY

Methodology consists of two steps, firstly the confirmation of the PSD of the silica sample and finally the development of protocol for PAMA 4031 particle counter.

2.1 Confirmation of PSD of Silica

Silica powder LANG LANG SILICA 60G, obtained from highly pure sand with known PSD in term of percentage of mass and with uniform density of 2.66 gm/cm^3 was used for the verification of the particle

count by PAMAS S4031 instrument. Mean values of particle size provided by the manufacturer are presented in the Table 1. Use of silica powder for a similar purpose has been reported by various researchers. For example SIL-CO-SIL 106 was used for laboratory performance testing of various stormwater treatment systems, e.g. Hydroworks used it for testing Hydroguard for TSS removal efficiency and hydraulic head loss (Bryant n. d). It is also used by Washington State as TSS Bench-Test Evaluation Standard (Guidance for Evaluating Emerging Stormwater Treatment Technologies 2008).

Table 1: Given mean values of particle size of SILICA 60G

Particle size in μm	<250	<150	<106	<75	<45	<20	<10	<2	<1
% Finer	99.0%	94.1%	86.2%	65.0%	60.0%	33.7%	19.1%	5.9%	5.1%

A curve was fitted to the given PSD to interpolate particle sizes in between so comparisons can be made with the adopted sizes used in settling tests. The comparison between given and interpolated particle size distribution is shown in the figure 1.

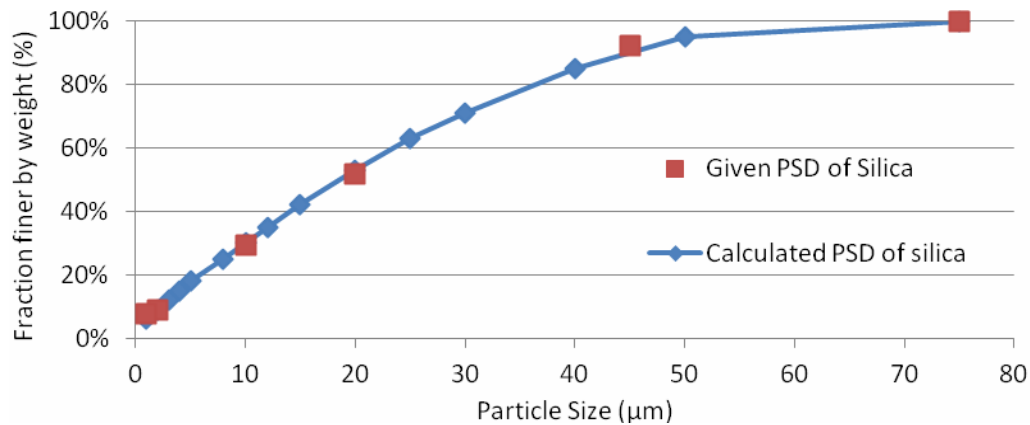


Figure 1: Comparison between given and interpolated PSD of SILICA 60G

Two different methods namely sieve analysis and settling column were used to confirm the given PSD of SILICA 60G. Wet and dry sieving were performed to fraction particles between 45 μm to 250 μm . Settling column was used to fraction particles larger than 2 μm whereas settling column (pipette method) was used for

a smaller particle range of between 2 μm to 63 μm . The PSD obtained from these methods were compared with the given PSD. These methods are briefly described below.

Sieve method: ISO 2591-1 (1988) guidelines were followed for dry and wet sieving technique. In the case of dry sieving around 20 gram of air dry silica powder was transferred to the top of a sieve stack containing the sieves of 150 μm , 75 μm , 63 μm 53 μm and 45 μm respectively. Mechanical sieving was used during the process. After 60 minute of sieving, residue on each sieve was collected and weighed to obtain the PSD. For wet sieving about 10 gram of silica sample was passed through each sieve. Water-spray was used to enhance the sieving process. The rinsing is carried out until the liquid discharged through the receiver is clear. Residue on each sieve was dried, this dry weight gave the cumulative mass of PSD.

Settling column: A top entry settling column (Figure 2) of 1.8 m long with 60 mm diameter based on the design presented by Hairsine and Mc Tanish (1986) was used. When sealed at the top, the tube contains a column of water, provided the open bottom end of the tube immersed in water contained in a turntable (Figure 2). Samples were introduced to the top of the tube in an injection barrel. Prior to the initiation of testing, the lower end of the tube was rested on and sealed by a rubber pad in the turntable.

Electronic motors raise and lower the column, and turn a sampling tray as required to separate the various settling velocity classes. Once the injection barrel is in place and sealed onto the top of tube, the tube is raised off the pad, and the pressure change causes the injector to open, allowing the sample to enter the tube and commence settling (Loch R.J. 2001). The instrument is controlled by computer program which allows the user to input sampling (rotation) times.



Figure 2: Settling column, Turntable with sampling tray

For settling test (column and pipette), silica was sieved through sieve no. 200 with pore size of 75 μm using a mechanical shaker. 10 gram of oven dry silica powder < 75 μm was used for the settling column, filled with tap water. A fraction of particles >73 μm , < 73 - > 52 μm , < 52 μm - > 42 μm , < 42 μm - >22 μm , <22 μm - >10 μm , < 10 μm - < 2 μm were collected in the tray at the bottom in 371seconds , 726 seconds, 1132 seconds, 20000 seconds, 77560 seconds and 49183 seconds respectively . Settling times were calculated based on Stokes Law assuming spherical particles. Dry weight of collected sample in each tray gave the PSD of silica in terms of mass.

Pipette method: Pipette method of particle size analysis is a sedimentation procedure which utilizes pipette sampling at controlled depths and times (Day 1965). Particles having a settling velocity greater than the ration of the depth and the elapsed time period will settle below the point of withdrawal after the elapsed time period. The sediment concentration at a certain depth can be determined by withdrawing samples at that height. Usually eight or nine samples are withdrawn (Rijn, L. C. Van, 1986). Settling times were determined based on Stokes Law and ranged from 30 minutes to 4 hours. The pipette method determines the weight percent of sample. There are two critical assumptions: the particles all have same density and the particles are spherical.

The method that is outlined in - Methods of Soil Analysis, Part 1, (Day, 1965) was followed during the experiment. Ultrasonic energy was applied for the dispersion of the sample. 20 gram of oven dry sieved silica < 75 μm were used for the tests. The fractions of <62.5 μm , <31 μm , <16 μm , <8 μm , <4 μm , and <2 μm were collected using 10 mL volumetric pipette at 10 cm, 5 cm and 2 cm depths from the surface at 22 °C.

The proportion of solid material in each size fraction was calculated using the oven dried masses, the volume removed by pipette sampling and the volume of suspension in the sedimentation cylinder.

An electronic microscope (Nikon ECLIPSE E600) was employed to view the particle shape. Particles smaller than 75 μm were analysed under 20 and 40 times magnification.

2.2 Protocol for using PAMAS S4031 particle counter

PAMAS S4031 particle counter (Figure 3) is a compact analysing system for liquids like raw, waste and drinking water, organic and corrosive fluids. It is a portable self contained instrument capable of analysing and reporting contamination, from a bottle or online. It is operated on the single particle counting system using the light blockage principle with a laser based sensor system calibrated with latex spheres according to ANSI/NFPA and ASTM. It can measure a size range from 1–100 μm , with 16 freely adjustable size channels. The instrument is configured to measure up to a limit of 200,000 p/mL, however, the optimum range of performance ranges between 1-30 μm (PAMAS S4031 manual). The instrument was used by Jenderek (2005) to monitor the filtrate of a membrane unit for waste water processing. Silica smaller than 75 μm was used for the test, though the instrument is capable of measuring particle size up to 100 μm .



Figure 3: PAMAS S4031 Particle Counter

Important Setups for Analysis

Several aspects are important in the setup of the PAMAS particle counter some of which are:

Pre-run time: Time to rinse the instrument with the sample to be run.

Number of Measurements/Run: Number of measurements of a sample that can be taken.

After Measurement Period: Time (second) taken for each measurements of a sample. Measurement Vol: Volume of sample analysed.

The instrument needs to be rinsed with MilliQ water before the analysis of each sample. Time to rinse the instrument depends upon the quality of sample analysed. Milli Q water was run until the total count of particle reached as low as 100p/ml.

Silica solutions of 10 ppm, 20 ppm, 40 ppm, 80 ppm, 400 ppm were made by diluting stock solution of 1000 ppm. Stock silica solution was prepared adding 0.5000 gram of silica powder in 500 mL of Milli Q water, and further dilution were done to get 50 mL silica solution of required concentrations. Milli Q water used for the process had particle count less than 100/mL according to the PAMAS S4031.

Two methods to inject the sample for analysis were tried. Initially a magnetic stirrer was used for sample mixing and samples were pumped in. An average of ten readings was taken for a sample using default setup. It was found that the larger particles settled to the bottom of the inlet pipe. To avoid settling a flask with an opening on the base was used and was kept above the instrument to allow gravity flow in addition to pumping.

Around 40 ml of sample was analysed in single measurement, pumped at rate of 10 ml/min during the measurement. The instrument has the option of running up to 10 measurements for a sample and take the average of only reliable results. However, in order to eliminate the error due to non homogenous solution, the entire sample was measured in a single run. The advantage of using single measurement instead of taking average of several measurements is that it prevents the bias of low particle count of big particles that are recorded at the beginning of the measurement, especially when gravity flow is permitted. The pre run time was set at 30 seconds to make sure that the early portion of sample with large particles were not flushed out during the rinse of the instrument.

The percentage of particle count from the instrument at various concentrations and size range were determined and compared with the expected particle count obtained theoretically using density formula. The suspended solids concentration obtained from particle count data was also compared with the solution of each concentration used for the analysis. The particle count for each particle size increment N_i , Mass of particles in certain size to yield the particle number per aqueous volume (cm^{-3}) for each size increment i can be calculated using following formula

$$N_i = m_i / \rho_{si} V_{si} V_l$$

Where m_i is the mass of the i th particle size, ρ_{si} is the density of particles in the i th particle size increment, V_{si} is the particle volume of known geometry assumed spherical in the i th size increment and V_i is the volume of aqueous suspension containing the particles.

3. RESULTS AND DISCUSSION

3.1 Confirmation of PSD of Silica

Sieve method: Four sieving tests were performed to confirm the given PSD of silica. Figure 4 shows that the PSD obtained from wet sieve for particles between 250 to 45 μm were comparable to the given PSD. The dry sieve PSD poorly matched the given PSD. Silica powder had around 60 % particles finer than 45 μm which was comparable to the results obtained from wet sieve, i.e. 58.1%. The dry sieving showed this fraction to be only around 13%.

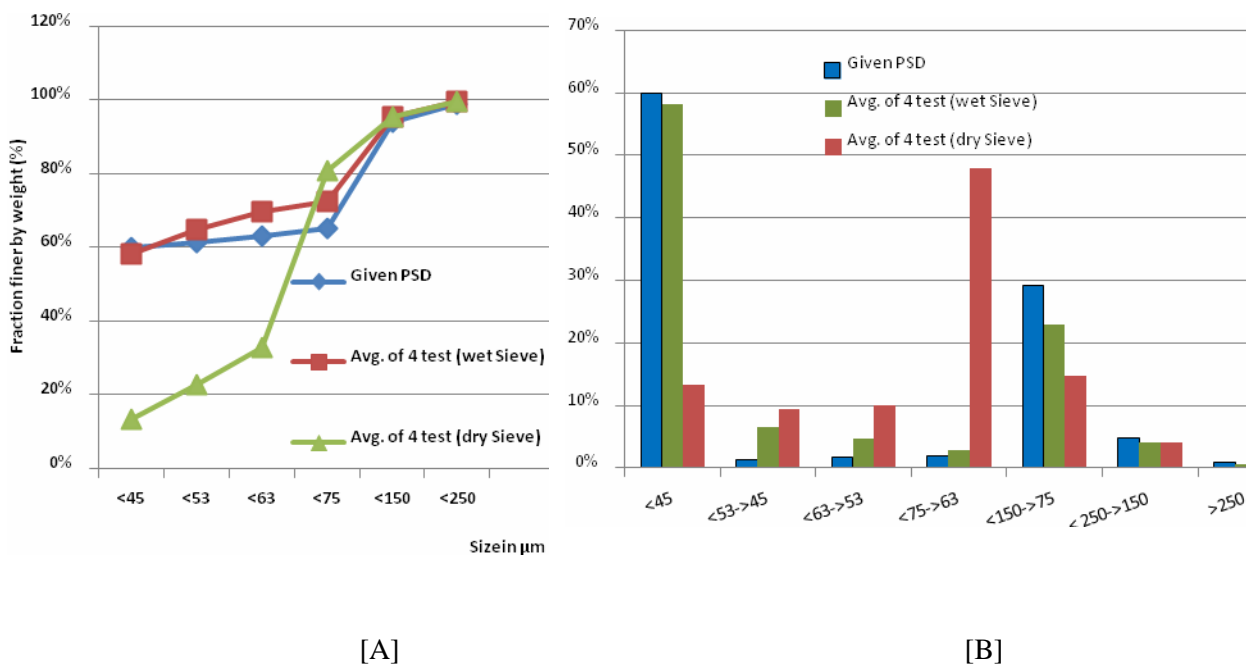


Figure 4: Comparison of given cumulative [A] and non cumulative [B] PSD of Silica with PSD obtained from wet and dry sieve at different size range

As the particle size becomes smaller, the sieving method is less reliable, which was more evident in the dry sieve method. The differences in PSD for all size ranges were higher than 10% except for particle smaller than 45 μm in wet sieve.

Settling column: Figure 5 shows the difference in the percentage of PSD obtained from settling column test and given PSD. The difference is quite significant and is not consistent.

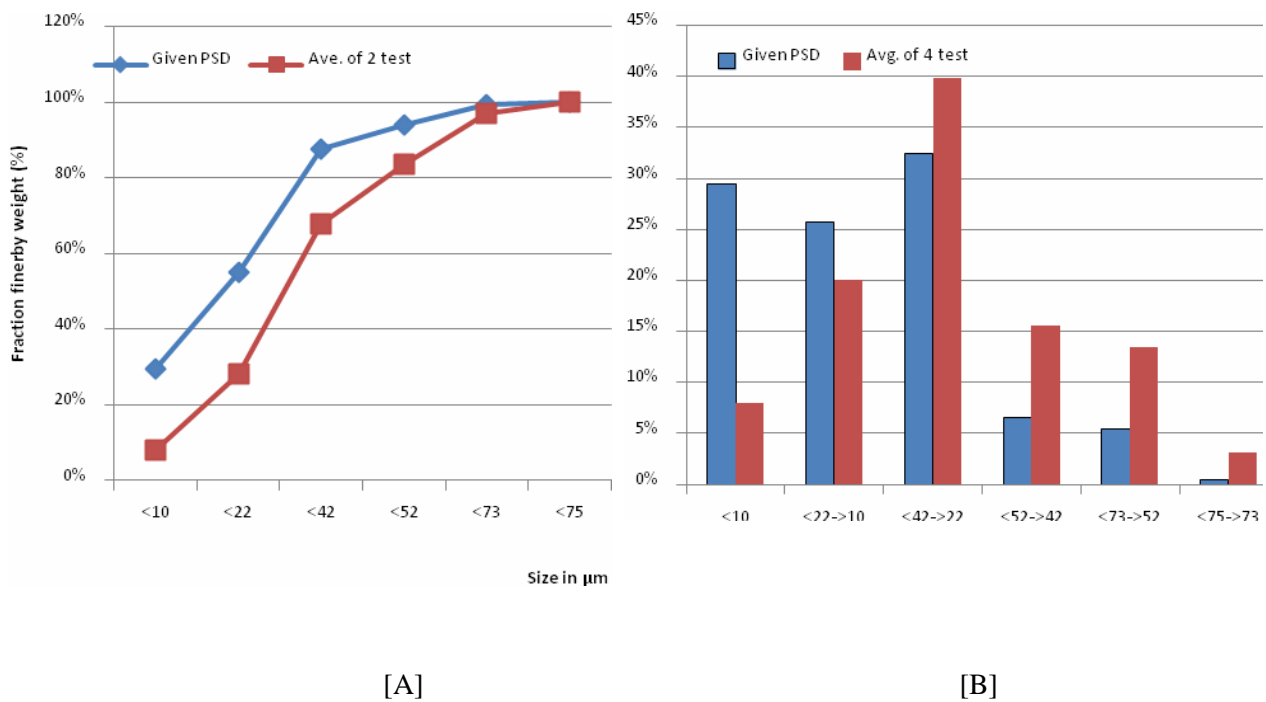


Figure 5: Comparison of given cumulative [A] and non cumulative [B] PSD of Silica with PSD obtained from settling column

Pipette method: For particles ranging between 2 to 62.5 μm , the pipette method was used. The PSD obtained from pipette method was comparable to the given PSD with the percentage difference of about 10% for particle size between 16 to 62.5 μm (Figure 6). The percentage of difference was highest in 2 to 4 μm with 70%.

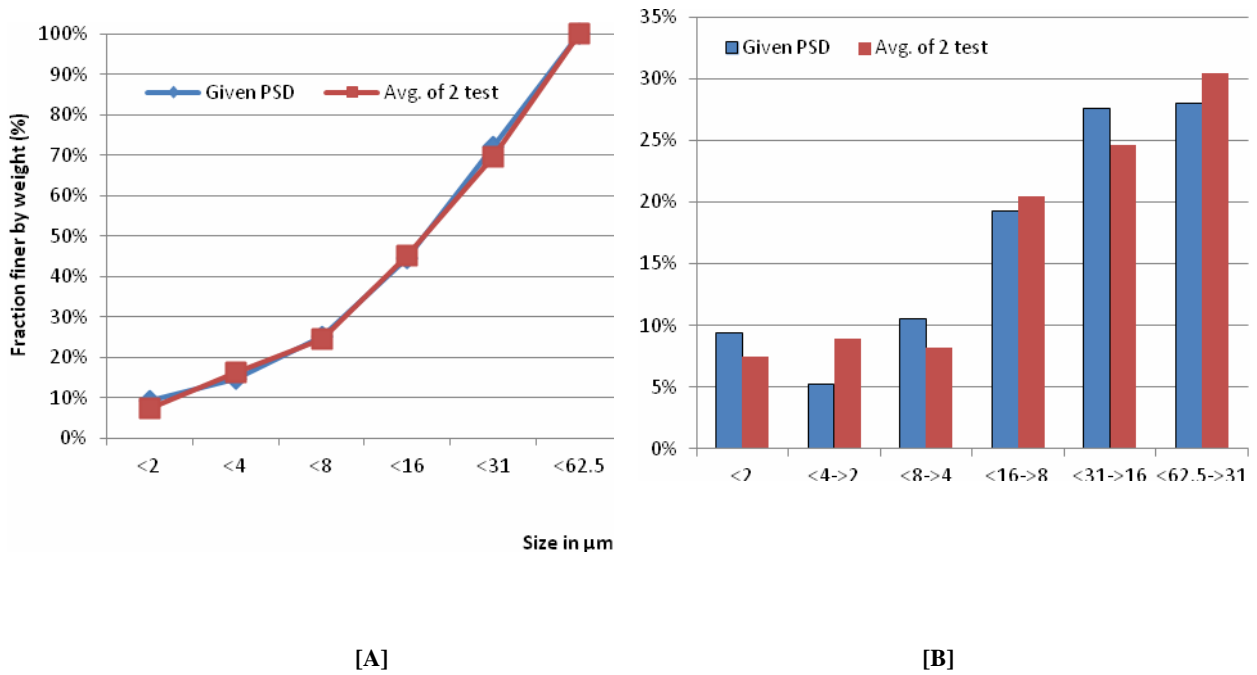


Figure 6: Comparison of given cumulative [A] and non cumulative [B] PSD of Silica with PSD obtained from pipette test

Particle size analysis by the settling technique depends on knowing the relationship between fall velocity and size. For the particles of same density, it is understood that large particles falls faster than smaller ones. However, the shape affects its settling velocity. The effect of shape on size analysis by settling techniques has been discussed by McNown & Malaika (1950), Janje (1966), Komar & Reimers (1978) and other. As Stokes Law is based on the assumption of spherical particles further testing on the silica particle shape were carried out.

The view from the Electronic microscope showed that the particles $< 75\mu\text{m}$ analysed under microscope were not spherical in shape (Figure 7 and 8). This could be one of the reasons for variation in the estimated and given PSD.

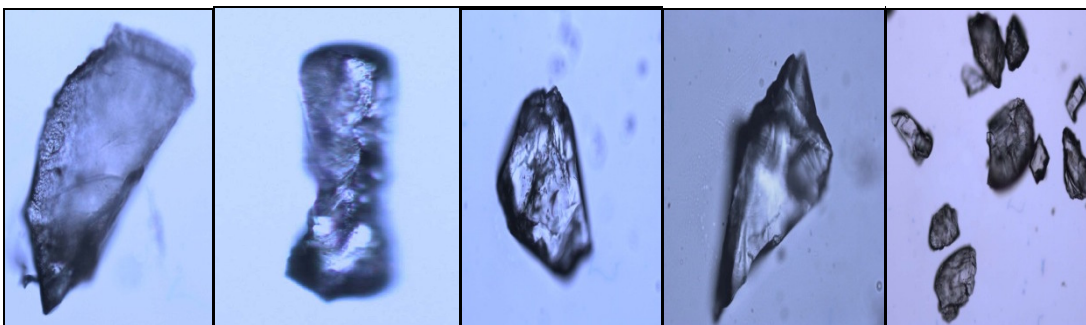


Figure 7: Images of Particles between $75\mu\text{m}$ to $63\mu\text{m}$ under the microscope.

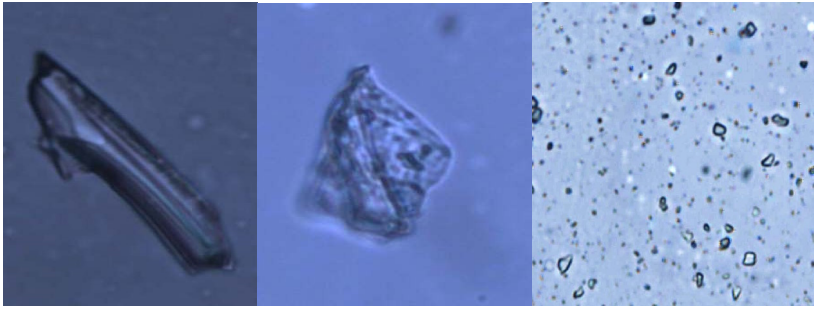
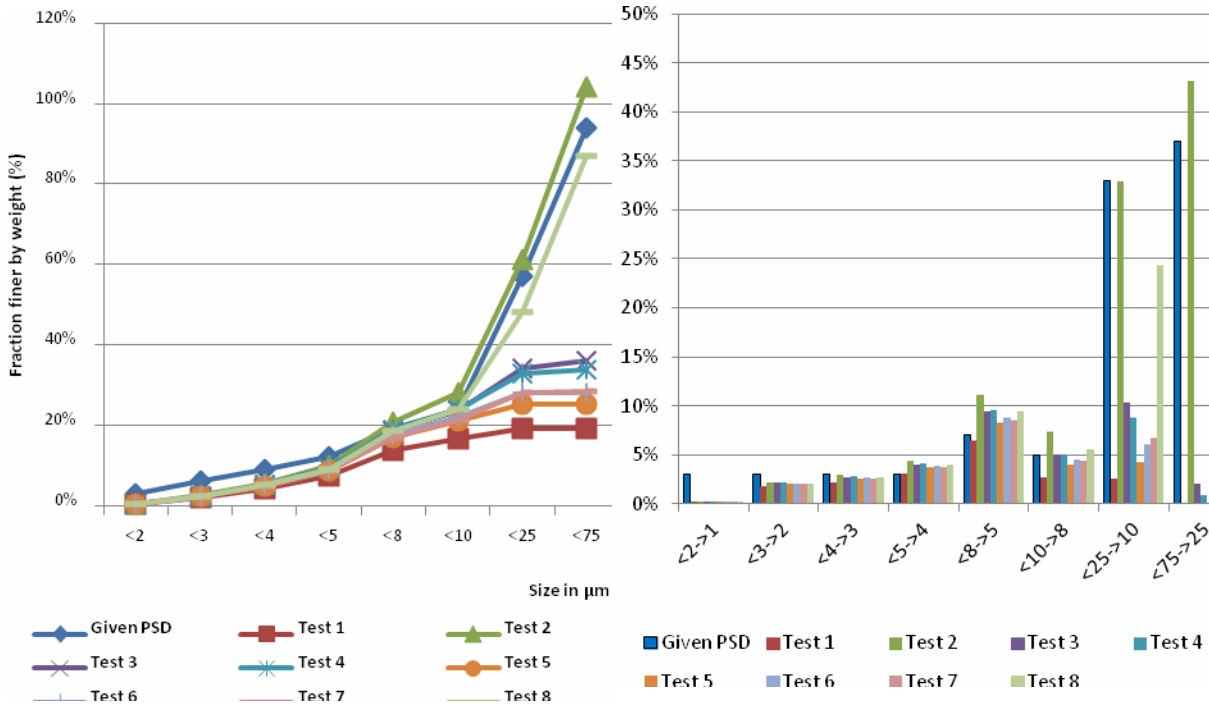


Figure 8: Images of Particles smaller than 4 μm

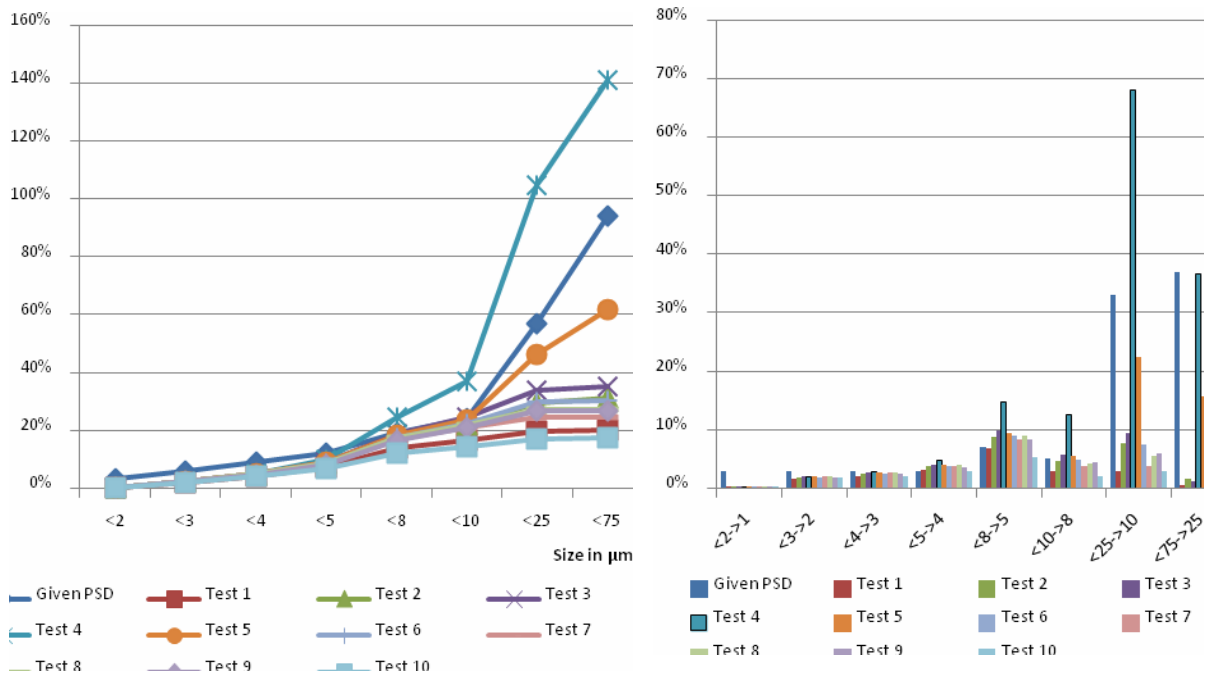
The comparative analysis highlights substantial differences between the sieving (especially dry sieving) and settling column results with the given PSD. A better match between particle distributions was achieved when the pipette test was used. As this technique covers particle sizes as small as 2 μm and appears to confirm the given PSD, the given PSD was adopted and used as the benchmark to develop the PAMAS particle counting protocol.

3.2 Protocol for using PAMAS S4031 particle counter

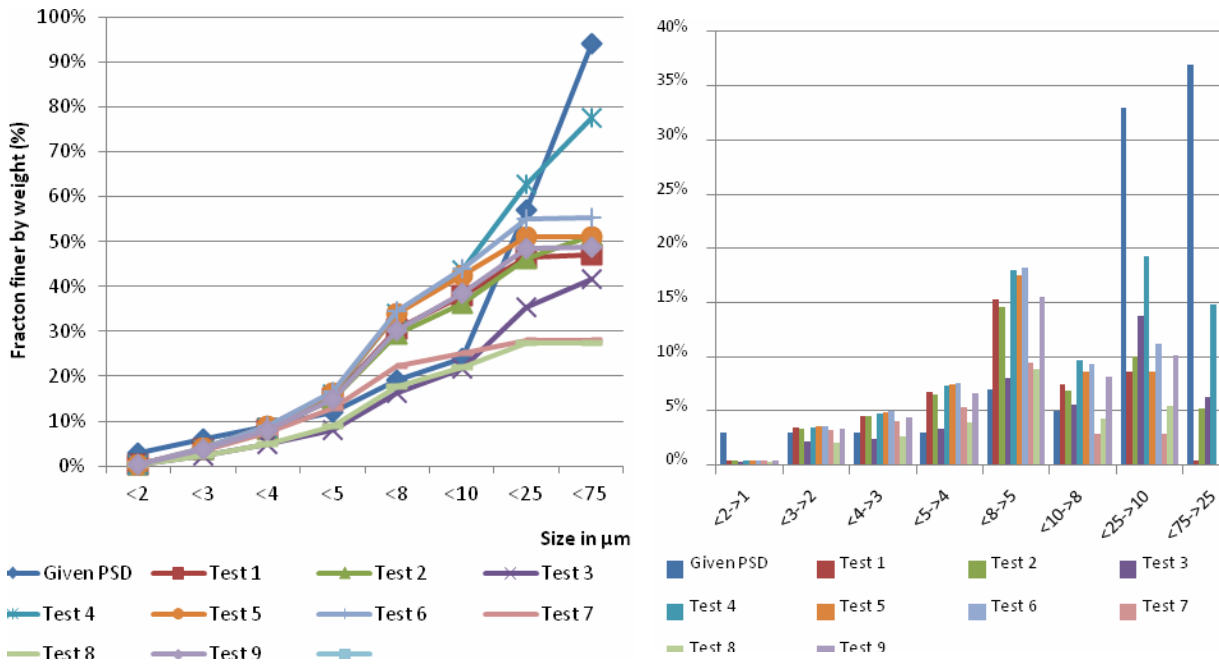
Results of 31 tests for silica solution (particles < 75 μm) at 10, 20, 40, 80 ppm are shown in figure 9. To make the comparisons clearer, the PSD data based on particle counts are presented as a percentage of the given PSD. This illustrates how much the count-based PSDs are above or below relative to the given PSD. Percentages of particle count were 75% or higher for particles < 10 μm - > 3 μm . Percentage of particle counted decreased as the concentration of silica increased from 40 to 80 ppm and also as particle size increased. However, in some measurements, especially at higher size range the percentage of particle count was more than 100%. This resulted in part because there were fewer particles to count in this size range.



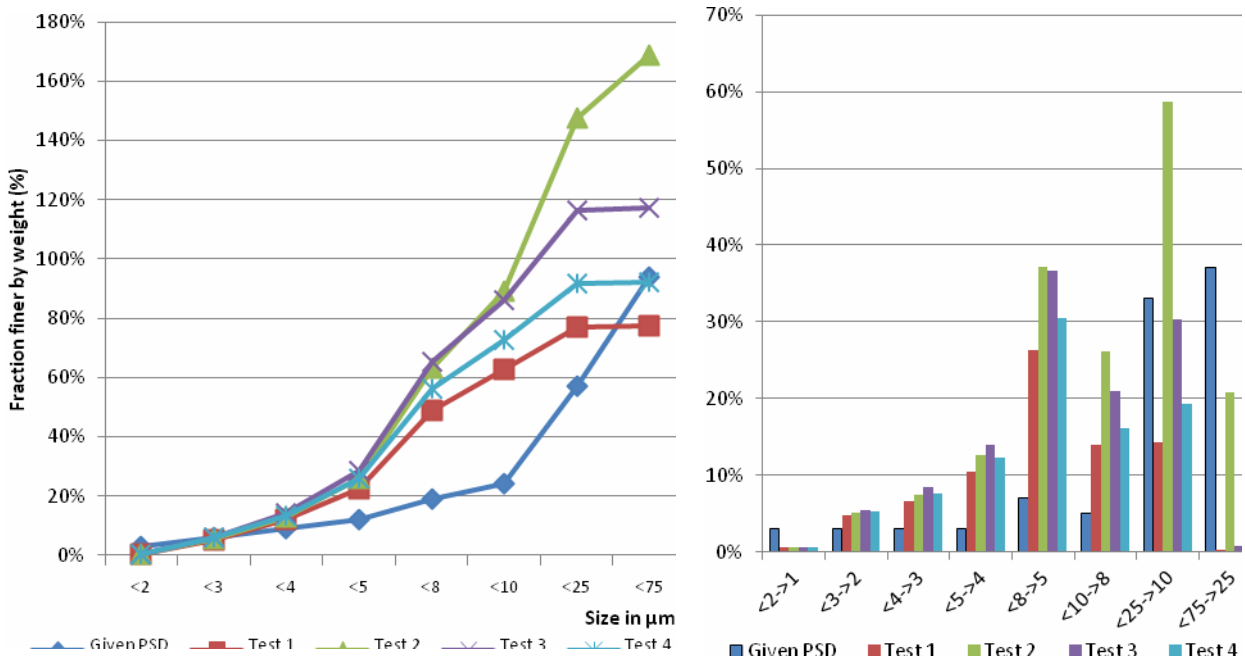
10 ppm



20 ppm



40 ppm



80 ppm

[A]

[B]

Figure 9: Comparison of given cumulative [A] and non cumulative [B] PSD of Silica at 10 ppm , 20 ppm, 40 ppm, 80 ppm at different size ranges between 1 μm to 75 μm with PSD obtained from PAMASS4031 particle counter

Two out of nine tests carried at 10ppm, given and calculated mass were found to be comparable and recovered solid concentration were 10 and 9 mg/L respectively. Similarly, at 20 ppm 2 tests out of 10 were within 50 % error in terms of percentage of mass recovered. Test were also carried for 40, 80, 400 ppm, recovered mass were ranged from round 40 to as low as 15 %. It was concluded that the instrument is potentially able to accurately measure the particle count when the solid concentration is around 10-20ppm but for a limited particle size range of 3 to 10 μm .

Due to the inconsistency in the PSD results, it was decided to remove the (relatively) large particles as far as physically possible and measure only fine particles. Initially a nylon filter of 25 μm pore size was used. However, using a Nylon filter of such a small pore size was time consuming and possibility introduced an error due to cake formation on top of filter. So the smallest sieve of practical size was trialled, which was 45 μm .

Particle count after filtration through 45 μm sieve showed a slight improvement in the particle count at lower size range for 1 - 3 μm especially for low concentrated sample i.e. 10 ppm compare to higher concentrated sample (Figure 10). At 10 ppm the percentage of particle count for 1-2 μ and 2-3 μm were around 75%. For larger particles, the count-based PSD represented only a fraction of the given PSD, as was the case when a <75 μm silica sample was used (Figure 9). However, a significant improvement in the overall consistency of the count-based PSD is evident.

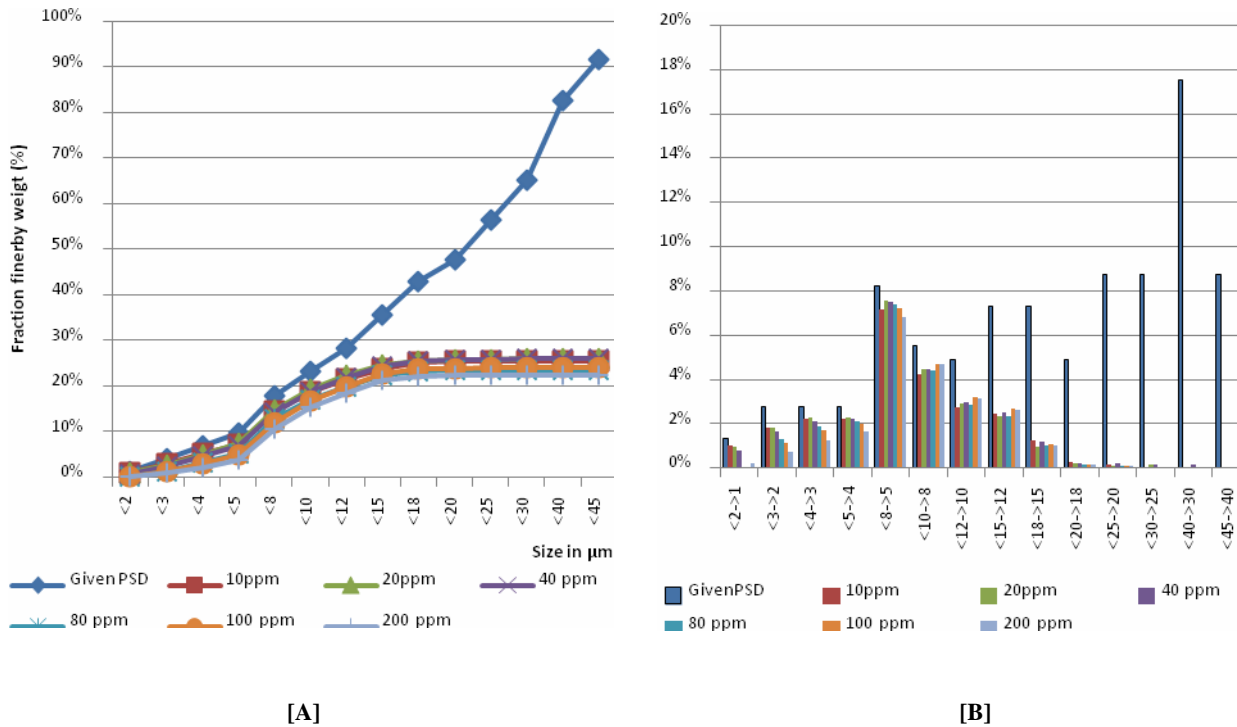


Figure 10: Comparison of given cumulative [A] and non cumulative [B] PSD of Silica at 10 ppm , 20 ppm, 40 ppm, 80 ppm, 100ppm, 200ppm at different size ranges between 1 µm to 45 µm with PSD obtained from PAMASS4031 particle

Another advantage of using a screened sample is that the sample will have fine particles smaller than 45 µm this will indeed make it easy to make the suspension homogeneous when stirred. So instead of using a single analysis, multiple analyses can be carried out. The advantage of multiple analyses for a sample is that the instrument can be rinsed with the sample before it is analysed. Since the sample is kept homogeneous using a stirrer, a portion of sample can be used to rinse the instrument. Pre-run time is set to 60 seconds in order to rinse the instrument with sample to be measured. This can save time and make analysis easy and quick, as the particle counter does not need to be low in particle count depending on the quality of sample to be analysed which otherwise could take 30 minutes.

Filtration through 45 µm filter, improved the reading for particles in the lower size range. However, not much change was found in larger particles, the percentage of particle count decreased as particle size increased. It is likely that these larger particles settled out before being measured. Another possibility could be that the pump flow rate through the instrument during measurement of 10mL/min is not enough to mobilise the larger particles.

4. CONCLUSIONS

- The advantage of using PAMAS particle counter over traditional methods of suspended solids analysis includes the ability to rapidly analyse samples and assist in decision making for selection of treatment system. The protocol developed is easy and quick to use. It takes around 5-12 minutes to measure a sample.
- Sample can be tested directly without pre-treatment. However, the sample needs to be screened through a 45 μm sieve before analysis. The instrument is not suitable for highly concentrated samples. Samples need to be diluted before analysed. Dilution required could reach as high as 50 times depending upon the sample concentration.
- Percentages of particle count for particles larger than 10 μm were below 75%, this is attributed to the larger particles settling in the inlet pipe before being analysed as they were visible during the analysis. The instrument has a fixed flow rate of 10 mL/min, so an instrument with a higher flow could perform better. The instrument has a designed optimum range of performance between 1-30 μm within an overall measurement range of between 1-100 μm . However, its most effective measurement range was very narrow range of 3-10 μm .
- Due to irregular shape of particles, actual particle count in each size range can be very different than the one obtained from the calculation and particle counter as both methods is based on assumption of spherical shape particles. The results of one method cannot be considered superior over another, however when a method provides consistent and repeatable results, it can be used to measure the performance of a system. It is just the matter of providing detailed information on the methods and instrument used for counting. As various researchers have found, the particle count from different instrument and setups could vary greatly depending upon its sizing techniques.
- Providing a uniform method is used, the PAMAS instrument indicated highly reproducible results. It could provide an easy and fast alternative method for stormwater suspended solids analysis especially where a large number of tests are required.

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