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Utility of Particle Size Distribution in Wastewater and Stormwater

A dissertation submitted in partial satisfaction of the

requirements for the degree Doctor of Philosophy

in Civil Engineering

by

Li-Cheng Chan

2010

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The dissertation of Li-Cheng Chan is approved.

Keith D. Stolzenbach

Jennifer A. Jag Richard F. Ambrose

Michael K. Stenstrom, Committee Chair

University of California, Los Angeles

2010

To my parents, Chiung-Yuan Chan and Hsueh-Ling Chan Chang

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## VITA

March 14, 1978	Born, Taipei, Taiwan
1996-2000	B. S., Agricultural Engineering National Taiwan University Taipei, Taiwan
2000-2002	Squad Leader Army service Keelung, Taiwan
2002-2003	Assistant Engineer Taipei Environmental Protection Agency Taipei, Taiwan
2003-2009	Graduate Research Assistant Department of Civil and Environmental Engineering University of California, Los Angeles

#### PUBLICATIONS AND PRESENTATIONS

- Chan, L., Li, Y., and Stenstrom, M. K. (2008) Protocol Evaluation of the Total Suspended Solids and Suspended Sediment Concentration Methods: Solid Recovery Efficiency and Application for Stormwater Analysis. *Water Environ. Res.*, 80 (9), 796–805.
- Chan, L., Li, Y., and Stenstrom, M. K. (2008) Authentic and Reliable Suspended Solids Measurement Process for Stormwater. 12<sup>th</sup> IWA DIPCON, Khon Kaen, Thailand.
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#### **ABSTRACT OF THE DISSERTATION**

#### Utility of Particle Size Distribution in Wastewater and Stormwater

by

Li-Cheng Chan Doctor of Philosophy in Civil Engineering University of California, Los Angeles, 2010 Professor Michael K. Stenstrom, Chair

This dissertation focuses on particle characteristics such as mixing properties in stormwater and in wastewater. There are two parts of the main theory. The first part is to discuss two methods of suspended solids measurement. The traditional method is called total suspended solids (TSS) and the alternate method is suspended sediment concentration (SSC). The lab work reveals that TSS method is comparable to SSC method if wide-bore pipette and proper mixing are used. The results are then used to estimate pollutant removal efficiency for stormwater best management practices. The trends show little difference between using TSS and SSC for low overflow rates, but the

difference becomes lager as the overflow rate increases, which might result in larger particles passing to the effluent. Suspended solids estimation errors involved in TSS or SSC methods may partially explain the poor performance reported in the literature of certain BMPs.

The second part is to distinguish the relationship between particle size distribution and sludge retention time. Activated sludge plants operating at high solids retention time (SRT) will have, on average, greater mean particle size in their mixed liquor suspended solids (MLSS). A simple indicator, particle average size, was developed to quantify the mean particle size of the sludge. The particle size can be integrated form 0.5  $\mu$ m up to 500  $\mu$ m. Twenty-three wastewater treatment plants were sampled. The samples were used to develop a protocol to consistently characterize particle size in mixed liquor solids and effluents. The results show that plants with long SRT have larger particle size in their aeration tanks and fewer particles in clarified supernatant.

## **Chapter 1. Motivation and Research Objectives**

## **1.1. Motivation**

Particle size is an important indicator of water quality and compliments traditional indicators such as oxygen demand, total suspended solids and other pollutants. Smaller particles, especially particles with high organic content, generally adsorb more pollutants, such as heavy metals and pathogens. Therefore particle removal is important for improving a wide range of water quality parameters.

Recent advances in particle sizing technology facilitate almost routine, automated measurements of particle size distribution (PSD). The ability to frequently and inexpensively measure PSD can broaden the understanding of treatment processes in both wastewater treatment and stormwater management. The use of PSD and how it affects or is affected by process conditions is only now being fully explored.

Particles in waters and wastewaters have traditionally been quantified by Total Suspended Solids (TSS) analysis (Standard Methods, 2000, Method 2540D), and protocols are well-known. This procedure does not provide information on particle size although only the particles larger than about 1  $\mu$ m are routinely measured. The TSS

measurement in stormwater is more difficult than in water or wastewater, because the particle size and density can be much greater, biasing the sample if it is collected from a poorly mixed location or allowed to settle in a quiescent collection container. Particles of importance in wastewater treatment usually have specific gravity (s.g., the ratio of the density of the particle to the density of water) usually have densities of approximately 1.00 to 1.02. The TSS measurement procedure in stormwater has recently been criticized because it may miss very large and dense particles (Sansalone et al., 1998). Particles in stormwater are mostly inorganic and have specific gravity of 2.5 or higher (Li et al., 2006).

An alternative method, called *Suspended Sediment Concentration* (SSC, ASTM 1999), uses a different protocol, and does not have this shortcoming. A representative sample must still be collected, but the SSC protocol requires the entire contents of the sample collection container be filtered. This avoids particle sedimentation in the collection container. Unfortunately the SSC method is not compatible with many monitoring programs, which require several constituents to be analyzed from a single sample container, such as from an automated, flow-weighted composite sampler. There are more rigors in the TSS protocol to insure better mixing while subsampling is required to avoid biasing against the larger, denser particles. If the TSS analysis can be performed

without bias, the SSC test can be avoided, except in cases where very large particles must be quantified, such as defining "bed load" or a stream (Glysson et al., 2004).

Another potential use of Particle Size Distribution (PSD) analysis is the operation of the activated sludge process for wastewater treatment. There is growing evidence (Bourgeous et al, 2003) to support the hypothesis that activated sludge plants operating at higher solids retention time (SRT) will have, on average, greater mean particle size in the biomass, and fewer small particles in their effluents. A theory to support this hypothesis is not available other than note that bioflocculation is a well known removal mechanism in the activated sludge process (Urbain et al, 1993), caused by the extracellular polymeric substances (EPS) which are noted to increase with SRT (Liao et al., 2001). To support this assumption, a large number of wastewater treatment plants were sampled and PSD was measured in the biomass as well as in the clarified effluent.

To perform PSD on both stormwater and biomass particles, a number of improvements in PSD protocol were required. These included 72-hour tests to determine the maximum permissible sample holding time before analysis, analysis to restrict the biomass PSD measurements to smaller particles and settling tests. These improvements are described in this dissertation.

## **1.2. Research Objectives**

The scope and research objectives of this dissertation are:

#### Stormwater

- Evaluate the existing TSS protocol using ideal particles, such as glass beads as well as actual stormwater particles;
- Develop and demonstrate an improved protocol in order to capture a more representative fraction of the larger, denser particles;
- Determine the potential bias that flawed TSS analysis can have on efficiency calculations of stormwater best management practices.

#### Wastewater

- Develop and demonstrate a protocol to measure mean particle size of the biosolids in the activated sludge process;
- Using the new protocol, show the relationship between SRT and mean biomass particle size, and effluent particle size and number;
- Build a database of particle size characteristic of wastewater treatment plants including plants that are not traditional activated sludge plants to allow the hypothesis of this dissertation to be extended.

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# Chapter 2. Suspended Solids Measurements in Stormwater

Total Suspended Solids (TSS) is routinely measured in water and wastewater treatment plants, and protocols are well-known. The TSS measurement in stormwater is more difficult, because the particle size and density can be much greater, biasing the sample if it is collected from a poorly mixed location or allowed to settle in a quiescent collection container. An alternative method, called *Suspended Sediment Concentration* (SSC), uses a different protocol, which analyzes the entire contents of the sample collection container. The SSC method is not compatible with many monitoring programs, which require several constituents to be analyzed from a single sample container, such as from a flow-weighted composite sample. This work addresses TSS protocol using glass beads and samples with known particle size distribution and shows that proper mixing, combined with appropriate pipettes, can largely avoid sampling error for typical sediments as large as 250 µm with specific gravity of 2.6.

## 2.1. Introduction

Total Suspended Solids (TSS) in stormwater is frequently used as a surrogate indicator of overall water quality, because TSS is often correlated with other water quality parameters, such as heavy metals, nutrients, polynuclear aromatic hydrocarbons, and chemical oxygen demand (Han et al., 2006; Schorer, 1997). The TSS is easy to measure, requiring no sophisticated instrumentation or special training. Heavy metals are often sorbed to suspended solids, and their analysis requires a metal digestion procedure (Lau and Stenstrom, 2005), requiring more time and expense for monitoring. If metals and TSS are correlated, the simpler, less expensive TSS procedure may serve as a predictive indicator or surrogate for metal concentrations (Furumai et al., 2002; Herngren et al., 2005).

An alternative method for measuring suspended solids content is the *Suspended Sediment Concentration* procedure (SSC) (ASTM, 1999). This method differs from the TSS method principally in the way the sample is collected. In the SSC method, subsampling using a pipette or volumetric cylinder is not allowed. This direct measurement avoids potential problems of large or dense particles not being correctly sampled as a result of stratification in the sample container. Both the TSS and SSC methods require samples to be collected from representative, well-mixed locations, such as a rapidly flowing stream or a free waterfall, or from stratified flows using some type of sampler that ensures proper depth integration. The SSC method has been frequently used by researchers interested in determining the mass of sediment (bed load) that might accumulate at the mouth of a river or similar areas (Glysson et al., 2004). The TSS method has more frequently been used when analyzing pollutant concentrations.

The purpose is to investigate the utility of TSS for characterizing stormwater samples, in which the solids might have specific gravities (the ratio of solids density to the density of water) of approximately 2.6, and to show how strictly adhering to the mixing required by the protocol are necessary for obtaining representative results. To illustrate the correct methodology, silicon beads of four different sizes and embedded sediment (solids collected from the bottom of a sedimentation device) were used. Also, various editions of *Standard Methods for the Examination of Water and Wastewater* (APHA et al., 1925–2000) were reviewed to pinpoint when the mixing advisory of the TSS protocol changed. Several editions do not advise analysts to ensure mixing for heavy, large particles, and recommendations to use a wide-bore pipette are inconsistent over the editions from 1981 to 2000.

## 2.2. Methods to Exam Particles in Wastewater

#### 2.2.1. Background

Solid matter suspended in wastewater has long been quantified by a procedure called Total Suspended Solids or Total Suspended Matter, and the analytical protocol has been documented in every edition of Standard Methods (APHA et al., 1925–2000) since 1925. The primary use of this method has been to characterize the suspended material in drinking waters or wastewaters. This method uses a filter paper to separate the suspended and soluble materials. For this reason, the definition of soluble material is often arbitrarily defined as the pore size (typically 0.45 to 1.5 µm) of the filter paper used in the analysis. Particles smaller than the pore size are generally considered soluble. In drinking waters or wastewaters, suspended matter is typically organic-rich, which tends to reduce the specific gravity to relatively low values ranging from 1 to 1.1 (Tchobanoglous et al., 2003). Because of their low specific gravity, the concentration of suspended material can be measured using a subsample taken from a sample container, with little bias from sedimentation or poor mixing of solids in the container.

Suspended materials in surface waters or stormwater typically contain a relatively greater portion of minerals, resulting in much higher specific gravities compared with

that of wastewater, ranging from 1.5 (soil particles) to 2.6 (silica sand), and, in rare cases, to as much as 4.2 (garnet sand). The size of the particles of interest may be much larger, because, for example, rivers can transport particles larger than 1000 µm in diameter. especially during flooding (Glysson et al., 2004). Additionally, the need for such measurements is not always for pollution monitoring, but sometimes for quantifying sediment accumulation behind dams or in deltas. To properly estimate the concentrations of these larger, denser particles, the SSC method has been used, and the principal difference compared with the TSS method is the prohibition of subsampling. Subsampling from the original sample container using a pipette or similar device may not provide an accurate measurement of suspended solid concentration, because heavy particles settled on the bottom of the container or particles larger than the opening size of the pipette cannot be effectively collected. Therefore, subsampling that is allowed in the TSS method can produce significant error in estimating the amount of solids in surface water, such as river and stormwater runoff. For example, Gray et al. (2000) discussed the differences between the two methods and the effects of the choice of the method on sediment estimates in surface water, suggesting that studies using the TSS method are flawed, underestimating sediment concentration or mass emission rate. Glysson et al.

(2004) compared TSS and SSC data from a range of locations and concluded that there was no simple way of reconciling the two measurements.

For stormwater monitoring, the SSC method has disadvantages compared with TSS. The SSC method uses the entire sample volume, which requires a second sample to be collected if other constituents are to be analyzed. A second automatic sampler may be required, which is expensive. Moreover, investigators may be concerned that automatic samplers using tubing pumps may not be able to pump the largest, densest particles into the sample container. Another potential problem in using SSC as a replacement for TSS is that particulate pollutants are typically measured using the residue collected on the filter paper, and, if different methods are used to collect the samples being filtered, the particulate pollutant may no longer be correlated to TSS or SSC.

#### **2.2.2. Experimental Methods**

#### 2.2.2.1. Mixing Tests

To compare the SSC and TSS methods for different types, sizes, and densities of particles; mixing regimes; and pipette sizes, a series of tests were performed in 1-L glass beakers of a synthetic water sample containing known sizes and concentrations of particles. The contents of the beakers were sampled, either by taking a subsample using a pipette for the TSS method or by filtering the entire volume for the SSC method. Spherical silicon beads and embedded sediments were used to simulate suspended solids. Silicon beads were purchased, ranging from 45 to 420 µm in diameter, with a specific gravity of 2.6 (McMaster Carr, Santa Fe Springs, California). The pictures under microscope are in the Appendix. Embedded sediments were collected from a highway stormwater runoff detention basin near Los Angeles, California, and sieved into four fractions, as shown in Table 2.1. The detention basin received pavement runoff and runoff from vegetated shoulders.

In each set of experiments, 6 to 12 beakers were filled with deionized water and 500 mg of test material, such as the 45- to 90-µm silicon beads, to obtain a 500-mg/L solids concentration. One beaker was used for the SSC method, and the others were for the TSS method. Each beaker was placed on a magnetic stirrer and mixed at seven different speeds, from 200 to 1100 rpm. The mixer speed was measured using a Pocket Laser Tach 200 (Monarch Instrument, Amherst, New Hampshire). The stirring bar measured 40 mm ×10 mm. The G-factor was calculated using mixer speed and the size of the stirring bar and ranged from 81 seconds<sup>-1</sup> (200 rpm) to 1041 seconds<sup>-1</sup> (1100 rpm). The two-paddle turbulent equation parameters were used (Reynolds and Richards, 1995).

A gang-mixer (PB-700 Jartester, Phipps & Bird, Richmond, Virginia) typically used in coagulation–flocculation studies was also evaluated for mixing.

### 2.2.2.2. Total Suspended Solids and Suspended Sediment Concentration Standard Methods

The TSS method was performed according to *Standard Methods* (APHA et al., 1925–2000). The TSS discussion has been simplified since 1971, removing or reducing the discussion requiring a wide-bore or cut pipette and adequate mixing of the sample. These two points received special emphasis in the editions of *Standard Methods* published before 1971. However, all the editions of *Standard Methods* published between 1925 and 2000 suggest the use of a 100-mL pipette and a single 47-mm circle of Wathman 934-AH filter paper (1.5-µm cutoff) for sample collection and separation of suspended solids, respectively. The following three types of pipettes were used to examine the effect of pipette type on the TSS measurement:

- (1) An original or unmodified pipette,
- (2) A pipette that had been cut off in the middle of the tip contraction and fire polished, and
- (3) An open pipette that had been cut off just above the tip contraction.

The diameters of the pipette openings were 1420  $\mu$ m (original), 1840  $\mu$ m (cut), and 3950  $\mu$ m (open). The modified pipettes were calibrated to compensate for the delivery volume change by comparing with unmodified pipettes and placing a new "full mark" using tape. Generally, the change in volume for the cut and open pipettes were 0.08 and 0.65 mL, respectively.

The SSC method was performed using the ASTM procedure D3977-97 (ASTM, 1999). The procedure is similar to the TSS method, except that the entire volume of the beaker was filtered through the same type of filter paper. Solids remaining in the beaker were washed into the filter flask with distilled water. For both methods, the filter papers were carefully removed, avoiding any loss of solids, and oven dried at 103 to 105°C for 1 hour. One hour was sufficient time to ensure that the residue was dried to constant weight, changing less than 0.5 mg, and was established early in the experimental program.

The value of TSS or SSC was reported as follows:

$$SS = \frac{(W_1 - W_0)}{V}$$
(2.1)

where

$$SS =$$
 concentration of TSS or SSC (kg/m<sup>3</sup>),

V = volume of the filtered mixture (0.01 m<sup>3</sup> for TSS and 0.1 m<sup>3</sup> for SSC), and  $W_0$  and  $W_1$  = weights of the filter paper and filter paper plus filtered material (kg), respectively.

#### 2.2.2.3. Sediments and Sieving Methods

Sediments were collected by our laboratory from two sedimentation basins receiving highway and grassy shoulder runoff from a major freeway in the area of Los Angeles, California, having average daily traffic of approximately 200,000 vehicles. Embedded sediments were collected from site 4, and water column suspended solids entering the sedimentation basin were collected from site 5. Embedded sediments were allowed to dry to a stable, fixed weight at room temperature and humidity. These sediments were then sieved using standard Tyler sieves into the four size fractions shown in Table 2.1, and the individual size fractions were later used in settling tests. Silicon beads were purchased in four different size fractions, as shown in Table 2.1. Small portions of beads in each size range were sieved to verify the sizes. In each size fraction of beads, less than 10% of the beads, by mass, were smaller than the indicated minimum size (i.e., less than 10% of the beads in the 250 to 425  $\mu$ m sizes were smaller than 250  $\mu$ m). The silicon beads are spherical, and settling velocity should closely follow Newton's law. The embedded

sediments are not spherical, but of arbitrary shape (Sansalone et al., 1998), and should settle at lower rates than the beads, for the same mean diameter and density. Particle size distribution (PSD) was measured on samples from site 5.

	Silicon beads	Embedded Sediments
Specific Gravity	2.6	2.2 - 2.4
	45-90	<45
Size ranges	90-150	45-106
(µm)	150-250	106-250
	250-425	250-850
	200	200
	300	350
Mixing speed	450	550
(rpm)	600	700
	850	850
	1100	1100

Table 2.1. Material, size ranges, and mixing speeds

#### 2.2.2.4. Particle Size Distribution Analysis

A Nicomp Particle Sizing Systems (Santa Barbara, California) AccuSizer 780 optical particle sizer module equipped with an autodilution system and a light scattering/extinction sensor (model LE1000-2SE) was used for particle size analysis. This instrument was selected for its wide size range of detectable particles (0.5 to 500  $\mu$ m), speed (2 minutes per sample analysis), and auto dilution capability. Analysis was performed by collecting a representative sample (0.5 mL) from the well-mixed original sample using a wide-bore glass pipette and then injecting it to the AccuSizer. Between samples, the system was flushed with deionized water at least three times, which reduced
background particle concentrations to less than 3/mL. Li et al. (2005) previously described the technique to ensure that a representative sample is collected.

#### 2.2.3. Experimental Results

#### 2.2.3.1. Silicon Beads

Figure 2.1 shows the TSS recovery rate (fraction by weight of the added particles measured by the TSS analysis), as a function of mixing speed for three types of pipettes, for the silicon beads. For the smallest size fraction (45 to 90  $\mu$ m), the TSS recovery was almost 100% at 600 rpm and higher. For all of the four different size beads, the recovery rate generally increased as the mixing speed increased and reached its maximum at approximately 600 to 800 rpm and, in some cases, slightly declined after 800 rpm. The recovery rate decline at the highest mixing speed might be related to cavitation around the stirring bar or may have occurred because the momentum of moving beads was too high for the beads to change their horizontal trajectory to an upward direction into the pipette. The recovery of the 150- to 250- $\mu$ m beads was only 85% using the cut pipette at 800 to 1100 rpm and only 65% for the largest beads (250 to 420  $\mu$ m).



Figure 2.1. Silicon bead recovery during TSS analysis versus mixing speed; size fractions are as follows: upper left = 45 to 90  $\mu$  m; upper right = 90 to 150  $\mu$  m; lower left = 150 to 250  $\mu$  m; and lower right = 250 to 420  $\mu$  m.

The effect of pipette type on recovery rate was greater for the larger beads. Contrary to expectation, the open pipette had poorer recovery for the larger beads than the cut pipette. Close observation of the pipette revealed that large particles were transported into the pipette, but some portion of the particles settled out of the pipette during the brief time between the end of filling and transfer of the pipette to the filter funnel. For the cut and standard pipettes, settling also occurred, but the taper at the pipette tip reduced the settling rate, preventing the particles from exiting the pipette. To reduce the effect of the gravity settling of captured beads inside the pipette, quicker pipetting was attempted, but the improvement was insignificant. Figure 2.1 clearly demonstrates the superiority of a cut pipette and the wisdom of the explicit recommendations in the 1971 and earlier editions of *Standard Methods*. The diameter of the pipette mouth obviously has an effect, even though it is much larger than the particle diameter (i.e., 1840 µm versus 850 µm for the largest particle diameter).

One method of suspending particles while subsampling is to swirl the liquid while pouring (*Standard Methods* editions 10, 12, and 13). Li et al. (2005) was able to recover stormwater particles from highway runoff, with consistent results, from 4-L capped bottles using a repeated inverting and shaking technique. Such mixing cannot be used with open beakers, and simple swirling was able to recover only 40, 32, 25, and 18% of the silicon beads for the 45-to-90, 90-to-150, 150-to-250, and 250-to-420 µm fractions, respectively. Additionally, mixing with a gang-stirrer with 76 mm × 25 mm paddles at 50 to 300 rpm did not improve recovery compared with the results using the magnetic stirrer. Larger magnetic stirring bars (50 mm and 63 mm ×10 mm in diameter) and square containers were also evaluated, but they did not improve the recovery. Vortex occurred in circular container (pictures shown in the Appendix). Therefore, square container and baffles were added in the circular container to mitigate the influence of the vortex. Although these two methods reduced the vortex at high mixing speeds, particles settled in the stagnant zones at the corners.

#### 2.2.3.2. Embedded Sediments

The solids recovery results using embedded sediments (sediments that are recovered from a sedimentation basin or other sedimentation device) are shown in Figure 2.2 and are more consistent than those observed with silicon beads. At mixing speeds of 600 rpm or greater, the TSS method accurately measured the suspended solids concentration for particles less than 106  $\mu$ m, with all three pipette types. For particles in the 106-to-250  $\mu$ m range, the TSS method accurately measured suspended solids when the mixing speed was 700 rpm or greater, using the cut and open pipettes, but the recovery rate for the original pipette began to decrease as the mixing rate increased from 700 rpm. For the largest

fraction (250 to 850  $\mu$ m), at the maximum speed of 1100 rpm, the TSS method recovered approximately 80% of the embedded sediments, and the original pipette was inferior to the cut and open pipettes. The TSS method was more accurate for the embedded sediments than for the silicon beads, for the same mixing speeds and pipette type. This probably results because of the lower settling velocity resulting from their lower specific gravities (2.3 versus 2.6) and irregular shape.



Figure 2.2. Embedded sediment concentration versus mixing speed; size fractions are as follows: upper left = <45  $\mu$  m; upper right = 45 to 106  $\mu$  m; lower left = 106 to 250  $\mu$  m; and lower right = 250 to 850  $\mu$  m

#### 2.2.3.3. Results and Comparison

Table 2.2 summarizes the results of the experiments. The recovery of the cut-tip pipette was 13 to 18% more accurate than that of the original pipette. Moreover, mixing the sample well was essential. The experiments showed that the TSS method results approached those of the SSC method at 1100 rpm mixing speed for silicon beads and embedded sediments than are 250 µm or smaller. For particles larger than these upper limits, the TSS method underestimated the solids concentration.

	Original pipette	Cut pipette	Open pipette
Diameter (µm)	(1420 µm)	(1840 µm)	(3950 µm)
Silicon beads			
< 90 μm	100%	100%	100%
90 to 150 µm	72%	100%	89%
150 to 250 μm	68%	81%	78%
> 250 μm	38%	58%	50%
Embedded Sediment			
< 106 µm	100%	100%	100%
> 250 µm	75%	80%	90%

Table 2.2. Comparison of experiments with 1000 rpm mixing speed

#### 2.2.3.4. Mixing Time and Effect on Particle Size Distribution

For many analytical procedures, it is desirable to measure not only the TSS concentration, but also PSD. Therefore, it is important to know if the increased mixing intensity associated with improved TSS analysis will change measured PSD. To determine whether this can occur, a series of experiments was performed with embedded sediments.

Embedded sediments were added to a 1000-mL beaker to create a suspended solids concentration of 500 mg/L, as before. Samples were collected using the cut pipette for PSD analysis at various mixing times and speeds. Figure 2.3 shows the results for four different combinations of mixing times and speeds. The line indicated by the "+" symbols with error bars is the PSD for mixing speeds from 200 to 1100 rpm after 2 minutes of mixing. Mixing for 2 minutes had had no measurable effect on the measured PSD, and mixing as low as 200 rpm was able to keep the smaller particles (<20  $\mu$ m) in suspension. The other three graph lines show the PSD measured after 30 minutes of mixing. At 200 rpm, only the number of particles smaller than 0.6  $\mu$ m in diameter increased. The PSD changed at higher mixing speeds (350 and 800 rpm), resulting in a greater number of the smaller particles. The number of particles smaller than 10  $\mu$ m generally increased, with

the number of smallest particles (0.5 to 1  $\mu$ m) increasing 1.5 to 2 times. The pattern indicates that larger particles tend to break into finer particles during prolonged mixing. These results show that the increased mixing used to improve the accuracy of the TSS method does not change PSD, as long as the mixing time is less than 2 minutes.



Figure 2.3. Number of particles versus diameter under various mixing speeds

#### 2.2.4. Conclusions

This chapter has demonstrated that the TSS method with improved subsampling can be used in stormwater investigations when particle sizes of 250 µm or less are anticipated. It has also demonstrated that serious errors can or may have occurred because of poor subsampling. Investigators monitoring stormwater need to be mindful of mixing requirements and advise contract laboratories of the need to properly mix stormwater samples and use a wide-tip pipette. The experiments using silicon beads and embedded sediments show that a wide-tip pipette and well-mixed samples are both necessary for performing stormwater particle analysis. These mixing and pipette recommendations were described in *Standard Methods* editions from 1955 to 1971, but have been minimized after 1971. Appendix.

# Pictures of the silicon bead under microscope



# Pictures of the vortex



At mixing speed 550 RPM



At mixing speed 700 RPM



At mixing speed 900 RPM



At mixing speed 1100 RPM

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# Chapter 3. Using Total Suspended Solids and Suspended Sediment Concentration to Estimate Pollutant Removal Efficiency for Stormwater Best Management Practices

## **3.1. Introduction**

Often, the TSS or SSC concentration is used as a surrogate parameter to estimate the concentrations of the pollutants associated with the solids, such as particulate-phase metals. Also, best management practice (BMP) removal efficiencies for solid-phase pollutants are often correlated to solids removal efficiency. The following sections investigate the potential effect of poor solids recovery in the TSS procedure on solids and particulate-phase pollutants removal. Ideally, there should be no difference in observed removal rate, irrespective of the TSS or SSC method, for quantifying solids.

The recovery efficiencies shown in Figure 2.2 can be used to evaluate the utility of using TSS or SSC to estimate the removal of particulate pollutants. To determine the differences between the two methods, empirical functions were used to quantify the recovery, solid-phase concentrations, PSD, and removal efficiencies of hypothetical BMPs between the discrete observations. Continuous recovery functions, with respect to mixing speed (Figure 2.2), and literature data on particulate pollutants concentration were used to estimate recovery of suspended solids or removal efficiency of particulate pollutants in a stormwater BMP.

# **3.2. Calculating Pollutant Concentration**

#### 3.2.1. Elements of the Equation

The total concentration of a particulate-phase pollutant in a water sample can be calculated by summating the concentrations of the individual size fractions, which can be calculated as the product of particulate pollutant concentration, measured suspended solids concentration, and the recovery rate of TSS method, as follows:

$$C_p = \sum_i M_i \cdot SS_i \cdot R_i \tag{3.1}$$

where

 $C_p$  = particulate-phase pollutant concentration (µg/L);

 $M_i$  = pollutant concentration on the particles in i<sup>th</sup> size range, expressed as  $\mu g/g$ ;  $SS_i$  = solids concentration in the i<sup>th</sup> size range, measured by TSS or SSC (mg/L);  $R_i$  = fractional recovery of suspended solids using TSS or SSC for the i<sup>th</sup> size range.

The fractional recovery values will be equal to 1 in the case of SSC, where all particles are 100% recovered, regardless of size. The fractional recovery of TSS will be equal to 1 for small particles and less than 1 for large particles, as shown in Figure 2.2. Each component in Equation 3.1 can be obtained as described in the following sections.

#### **3.2.2.** Particulate-Phase Metal Concentration $(M_i)$

The concentrations of metals associated with particles have been measured by various researchers (Deletics and Orr, 2005; German and Sevensson, 2002; Lau and Stenstrom, 2005; Morquecho and Pitt, 2003; Roger et al., 1998; Sansalone et al., 1998; Zanders, 2005). Table 3.1 shows particulate copper concentrations from these references. Copper is more concentrated on smaller particles, which is a typical trend and was chosen as representative of other metals, which can be analyzed in the same fashion. The concentrations for several other metals are shown in the cited references.

Particle size (µm)	Copper concentration $(\mu g/g)$	Reference
2 to 63	530	
63 to 250	310	Deletic and Orr
250 to 500	130	(2005)
>500	50	
<75	470	
75 to 125	270	German and Svensson
125 to 250	340	(2002)
250 to 500	200	(2002)
500 to 1000	50	
<43	220	
43 to 100	230	Lau and Stnstrom
100 to 250	230	(2005)
250 to 841	240	
0.45 to 2	2894	
2 to 10	4668	
10 to 45	735	Morquecho and Pitt
45 to 106	1312	(2003)
106 to 250	2137	
>250	50	
<50	420	
50 to 100	250	Roger
100 to 200	200	et al.
200 to 500	100	(1998)
500 to 1000	50	
25 to 38	691	
38 to 45	353	
45 to 63	317	
63 to 75	326	Sansalone and Buchberger
75 to 150	312	(1997)
150 to 250	127	(1797)
250 to 425	78	
425 to 850	63	
850 to 2000	20	
0 to 32	181	
32 to 63	197	
63 to 125	212	Zanders
125 to 250	184	(2005)
250 to 500	85	
500 to 2000	47	

Table 3.1. Heavy metal concentration associated with particles

# 3.2.3. Particle Size Distribution and Suspended Solids Concentration (Ss<sub>i</sub>)

The PSD has often been used to characterize stormwater. Generally, PSD from the following three types of collection techniques have been reported:

- (1) Particles collected directly from the water column;
- (2) Particles vacuumed from street surfaces; and
- (3) Particles collected from sedimentation devices, which are typically called *embedded sediments*.

Table 3.2 shows the available literature references and divides them based on type. Sediments from sites 4 and 5 were collected by our laboratory and were used in developing Figure 3.1.

	Percent less than		Uniformity			
Solids Source	10%	60%	90%	Coefficient	Reference	
	μm	μm	μm			
	8	180	380	22.5	Li et al. (2006)	
Water	2	12	25	5.5	Morquecho and Pitt (2003)	
column	15	110	280	7.3	Kayhanian et al. (2005)	
	_20	220	700	11.0	Site 5	
	30	440	660	14.7	German and Svensson (2002)	
Vacuuming	65	340	1500	5.2	Lau and Stenstrom (2005)	
	24	210	810	8.6	Sartor and Boyd (1972)	
	30	450	650	15.0	Deletic and Orr (2005)	
	10	440	650	44.0	Roger et al. (1998)	
Embedded	70	400	1000	5.7	Sansalone and Buchberger (1997)	
Sediment	40	200	920	5.0	Shaheen (1975)	
	19	250	1000	13.2	Zanders (2005)	
	90	500	1900	5.6	Site 4	
Water						
column	11	131	346	11.5	<b>A</b> were a a	
Vacuuming	40	330	990	8.3	Average	
Embed. Sed.	43	373	1020	8.6		

Table 3.2. Solid components in water samples of different kinds of samples

The averaged parameters for the three categories are shown at the bottom three rows of Table 3.2, and the particles collected from the water column are smallest with the greatest range of sizes and highest uniformity coefficient. The water column particles are distinctly smaller than the other two groups. The embedded sediments are slightly larger than the particles from vacuuming, but they have almost the same mean uniformity coefficient.

Figure 3.1 was created using the full data sets from the sources cited in Table 3.2, to compare the PSDs of the water column, embedded sediments, and vacuumed particles from streets. Figure 3.1 demonstrates that samples collected directly from the runoff contain a greater fraction of fine particles and may contain a smaller fraction of very large particles, if a poor TSS mixing procedure was used. Particles collected by street sweeping or vacuuming have a coarser distribution, and those collected from sedimentation basins contain the coarsest particles. This probably occurs because particle collection directly from the water column can capture all particles, while embedded sediments do not contain the smaller particles, which are not removed in sedimentation basins. Vacuuming devices typically do not capture the finest particles, and fine particles may not be completely recovered from the filter. These differences in technique may explain the PSD differences among sources.



Figure 3.1. PSD of three groups among all the references

# 3.2.4. Recovery of Total Suspended Solids or Suspended Sediment Concentration $(R_i)$

The recovery of particles using the TSS method can be calculated from Figure 2.1 for silicon beads or Figure 2.2 for embedded sediments. An empirical, smooth function to facilitate calculations was created to describe the particle recovery at 1100 rpm with the cut pipette. The embedded sediments data were selected for these examples. The continuous recovery function was applied to the range of measured particles shown in Table 3.2.

### **3.2.5.** Removal Efficiency $(\eta_i)$

To compare the effect of PSD on removal efficiency, particle removal efficiency  $(\eta)$  was calculated from the particle settling velocity and overflow rate. Particle settling velocity was calculated based on Newton's Law at 20°C for spherical particles with a specific gravity of 2.6. The overflow rate (surface area divided by flowrate) ranged from 0.01 to 3000 m/h.

$$\eta = \frac{V_p}{V_o}$$
(3.2)

where

 $V_p$  = particle settling velocity (m/s or m/h; 1m/h = 1/3600 m/sec), and

 $V_{\rm o}$  = overflow rate (m/s or m/h)

As Figure 3.2 shows,  $\eta$  decreases with increasing overflow rates. The lowest overflow rates shown in this figure are not realistic, because micro-mixing and non-ideal conditions prevent the smallest particles from being removed. Particles with diameters smaller than 5  $\mu$ m are rarely removed in sedimentation devices (Tchobanoglous et al., 2003) because of this effect. The functions in Figure 3.2 can be used in Equation 3.1, which is the fractional removal of different particle sizes at a given overflow rate.



Figure 3.2. Relationship between particle removal efficiency (PRE) and particle diameter

#### 3.2.6. Overall Removal Efficiency (E)

The overall particle or particulate-phase metal removal efficiency (E) is defined by Equation 3.3.

$$E = \frac{C_{inf} - C_{eff}}{C_{inf}}$$
(3.2)

The observed influent concentration (Equation 3.4) must be expressed as the product of influent solids, particulate phase metal concentration, and recovery, which is unity for SSC and a function less than unity for TSS. The observed effluent concentration (Equation 3.5) is calculated the same way, except that the particulate removal vector is added.

$$C_{inf} = \sum_{i=1}^{n} \left( M_i \cdot SS_i \cdot R_i \right)$$
(3.4)

$$C_{eff} = \sum_{i=1}^{n} \left( M_i \cdot SS_i \cdot R_i \cdot [1 - Re_i] \right)$$
(3.5)

where

$$C_{inf}$$
 = observed influent concentration (kg/m<sup>3</sup>; 1mg/L=10<sup>-3</sup> kg/m<sup>3</sup>), and  $C_{eff}$  = observed effluent concentration (kg/m<sup>3</sup>).

The Equations above can be used to calculate the removal efficiency for suspended solids or particulate-phase metal pollutants. For demonstration purposes, six distinct size groups were selected. Morquecho and Pitt's (2003) size ranges and copper concentrations were selected from Lau and Stenstrom's (2005) data. Linear interpolation between particle sizes in Lau and Stenstrom's data was used to match Morquecho and Pitt's particle size ranges.

Table 3.3 shows an example calculation for copper and TSS recovery using the cut pipette and 1100 rpm mixing speed for embedded sediments form site 5. An influent concentration of 250 mg/L TSS or SSC size settling basin with an overflow rate of 100 m/h were assumed.

Efficiency (*E*) can be calculated using either TSS or SSC, depending on the data availability. When TSS measurements are used, suspended solids concentration can be obtained using the recovery function in Figure 2.2. When the SSC measurements are used to calculated *E*, the recovery rates for all size ranges are set to unity. If the metal concentrations ( $M_i$ ) are set to unity, then Equations 3.4 and 3.5 calculate the TSS or SSC concentration removal efficiency, depending on the value of the recovery vector.

Particle	Copper	Solid	Fraction	Fraction	Influent	Effluent
diameter	concentration	fraction	recovered	removed	concentration	concentration
(µm)	(µg/g)			_	(µg/L)	(µg/L)
20	220	0.010	0.98	0.013	0.54	0.53
70	230	0.098	0.93	0.153	5.3	4.4
175	230	0.29	0.84	0.959	13.9	0.58
550	240	0.34	0.58	1.000	11.8	0
1500	0	0.16	0.22	1.000	0	0
2200	0	0.10	0.11	1.000	0	0
Sum		1.000			31.5	5.6

Table 3.3. Copper removal efficiency using TSS influent and effluent data of Lau and Stenstrom (2005)

Table 3.4 shows the overall removal efficiency for particles and particulate-phase copper in an ideal sedimentation tank using TSS or SSC as analytical methods. Table 3.4 is based on the favorable recovery function for the cut pipette and 1100 rpm mixing speed. The particulate-phase copper concentrations from all seven literature references shown in Table 3.4 were evaluated. Three overflow rates were simulated. The largest difference in removal efficiency between TSS and SSC was 16.6% at 1000 m/h of overflow rate for German and Svensson (2002). The trends shown in Table 3.4 show little difference between using SSC and TSS for low overflow rates (i.e., high removal efficiency), but the difference becomes larger as the overflow rate increases, which allows the larger particles to pass to the effluent, and TSS is unable to quantitatively recover the large particles, overestimating removal efficiency.

If the recovery function for a standard pipette and 450 rpm mixing speed are used for the same calculation as shown in Table 3.4, experimental biases will become larger. This experimental condition might work well for sampling light, flocculent solids associated with wastewater treatment, but it provides only 28.4% recovery for the largest embedded sediments and only 1.6% for the largest silicon beads. For these conditions, the differences in observed suspended solids concentration, using Lau and Stenstrom's (2005) data for demonstration, are 84 mg/L for poor mixing and 147 mg/L for good mixing. For the same condition, copper removal efficiency of 73.1% is predicted, which is quite different than the removal efficiency predicted with the higher recovery vector (56.2%).

		Particle removal efficiency		Copper removal efficiency (%)			
Pof		(%) whe	n using		when using		
	Overflow	TSS inf.	SSC inf.	TSS inf.	SSC inf.	SSC inf.	
	rate (m/h)	TSS eff.	SSC eff.	TSS eff.	SSC eff.	TSS eff.	
	5	21.5	21.7	9.9	10.0	10.9	
1	100	1.4	1.4	0.6	0.7	1.7	
	1000	0.1	0.1	0.1	0.1	1.2	
	5	98.7	99.3	98.7	99.0	99.1	
2	100	83.4	89.5	82.3	86.1	87.1	
Í	1000	43.9	61.5	39.9	48.6	56.2	
	5	93.3	95.6	85.1	87.9	88.2	
3	100	78.3	85.5	58.1	65.4	66.9	
	1000	50.0	62.2	21.1	28.4	37.7	
	5	100	100	100	100	100	
4	100	70.0	80.0	41.2	48.1	52.0	
	1000	42.1	57.3	16.1	23.2	31.6	
	5	91.7	94.6	77.9	81.8	82.4	
5	100	75.7	83.8	42.7	52.3	54.4	
	1000	50.6	62.9	20.2	29.6	36.4	
6	5	93.1	93.3	88.8	90.3	90.3	
U U	100	67.6	68.5	45.0	50.5	50.7	
	1000	38.3	39.5	9.9	14.1	15.0	
7	5	94.6	96.5	86.0	88.4	88.7	
/	100	79.1	85.9	50.4	58.0	60.1	
	1000	51.0	62.9	18.9	26.6	34.8	

Table 3.4. Preference weights of SWP water categories during each month of year 2007

Reference: (1) Morquecho and Pitt (2003), (2) Lau and Stenstrom (2005), (3) German and Svesson (2002), (4) Sansalone and Buchberger (1997), (5) Roger et al. (1998), (6) Zanders (2005), and (7) Deletic and Orr (2005).

\* inf = influent; eff = effluent.

Suspended solids estimation errors involved in TSS or SSC methods may partially explain the poor performance reported in the literature of certain BMPs. For example, the utility of street sweeping is widely debated (Kang and Stenstrom, 2008) and may be explained, in part, by poor technique in measuring the larger particles in runoff from swept and unswept control studies. Also, undetectable differences between influent and effluent quality in hydrodynamic separators were reported by Geosyntec Consultants (2006) in the International BMP database. Laboratory studies (Woodward-Clyde Consultants, 1998) found that such devices can remove 95% of sand particles (specific gravity 2.6) greater than 425 µm, 78% of particles between 250 and 425 µm, 47% of particles between 150 and 250 µm, and 20% of particles between 75 and 150 µm. The lack of significant removals reported in street sweeping studies or in the database could be easily explained if poor TSS sampling techniques were used in the database studies. Subsampling for TSS at 400 rpm would not detect the most significant removal ranges of the sweepers or separators, which falsely suggests that they do not affect water quality.

### **3.3. Conclusions**

For BMP monitoring of devices that remove particles larger than 250  $\mu$ m, which include most sand filters, sedimentation basins, and vortex separators, the TSS subsampling method should be able to accurately sample effluent suspended solids. The SSC method should be used when larger particles are expected or when the entire sample can be used for SSC without penalty (i.e., when no chemical analyses are being performed, because there is no need for a subsample or additional sample).

This chapter has also shown that attempts to correlate previously collected TSS and SSC data will not be possible in general, unless the PSD and recovery of different size particles are known or can be inferred from experimental conditions.

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# Chapter 4. PARTICLE SIZE DISTRIBUTION AND SOLIDS RETENTION TIME

# 4.1. Introduction

Particle size in wastewater is an important indicator for traditional water quality parameters such as oxygen and total suspended solids but other pollutants as well. Smaller particles, especially particles with high organic content, generally adsorb more pollutants, such as heavy metals, polynuclear aromatic hydrocarbons (PAHs), and chlorinated pesticides, for example, DDT (Schorer 1997; Han et al. 2006; Bentzen and Larsen 2009; Liu et al. 2009). There is growing evidence to support the belief that activated sludge plants (ASPs) operating at higher solids retention time (SRT) will have, on average, greater mean particle size (Bourgeous et al, 2003). This study describes the distribution and mean size of particles in the 0.5 µm to 50 µm, 100 µm, and 200 µm ranges, which generally have higher concentrations of adsorbed pollutants, and are more difficult to remove in secondary clarifiers. The larger particles, 200 µm to 500 µm, are not considered in this study since they generally settle very well, and they can also be influenced by flocculation or aggregation in clarifiers, which is difficult to reproduce in a

laboratory or even representatively sample and analyze. The conventional ASPs operating at shorter SRT (1 to 2 days) are traditionally used in many wastewater treatment applications, and are considered the lowest cost method of secondary treatment. Operation at longer SRT has recently shown benefits, such as to more stable operation (with selectors), improved effluent quality with respect to emerging contaminants (Bolong et al., 2009; Petrovic et al., 2003), and improved oxygen transfer efficiency (Rosso et al., 2008).

Particle Size Distribution (PSD) analysis may serve as a useful tool to characterize the "healthiness" of activated sludge. Biomass particle size ranges from 0.5  $\mu$ m (single cell) to 1 mm (Tchobanoglous et al., 2003); and a healthier or more desirable sludge should consist of lower fraction of very small particles.

Two hypotheses will be evaluated in this chapter. The first is that larger particle size is associated with longer SRT. The second is that fewer effluent particles are associated with larger biomass particle size. To evaluate these hypotheses, wastewater treatment plants (WWTPs) close to UCLA or visited during the research period will be sampled and analyzed for PSD. A protocol will be developed to insure repeatable analyses. Also, the impact of activated sludge process modifications, such as step-feed or contact stabilization, will be noted.

# 4.2. Materials

#### 4.2.1. PSD Instrument

PSD was performed by using a Nicomp Particle Sizing Systems (Santa Barbara, CA) AccuSizer 780 optical particle sizer module equipped with an autodilution system and a light scattering/extinction sensor (Model: LE400-0.5SE). The instrument can detect particle size as small as 0.5  $\mu$ m in the diameter and counts particles in 0.007 to 7  $\mu$ m intervals. 0.5-mL sample solution is taken by the standard pipette and then injected in the dilution chamber. The PSD result is available after 90 seconds, if the sample does not require autodilution. Longer analysis time may be necessary if the sample concentration exceeds 12000 #/mL since the machine must dilute the sample to less than 12000 #/ml. Between each PSD test, three auto-flush cycles are used to ensure the cleanness of the dilution chamber and the sensor, which takes about 6 minutes to complete. The total time to process one sample is approximately 10 minutes.

#### 4.2.2. Particle Average Size Calculation

The median particle size was calculated using the first and second moments, in a fashion similar to the way retention time is calculated from a tracer study (Levenspiel 1967), as follows:

Total Particles = 
$$\int_{0.5}^{M} Nds$$
 (4.1)

First Moment = 
$$\int_{0.5}^{M} S \cdot N ds$$
 (4.2)

$$PAS = \frac{\int_{0.5}^{M} SNds}{\int_{0.5}^{M} Nds}$$
(4.3)

where M is the upper limit of particle size; N is particle count; S is particle size ( $\mu$ m); PAS is particle average size.

PAS is the mean or centroid of PSD and independent of the total number of particles or mixed liquor suspended solids (MLSS) concentration, which is an important property because the various sampled treatment plants all have different MLSS concentration. Storage time between sampling and analysis is controlled to six hours or less, except for samples collected at treatment plants that are more than one days travel from UCLA. A sensitivity analysis was performed to determine the impacts of storage time. For the initial samples storage time less than 24 hours had minor impact on the particle size distribution. A growing database of the PSDs and treatment plants operating parameters is being created. Twenty-three plants have been sampled and reported in this dissertation up to date. A sensitivity analysis of the cut-off point for PAS calculation was performed and reveals that it is not a particularly sensitive parameter, which is helpful. This results because the PAS is weighted by particle number and not particle mass. Fewer particles exist above 200 µm which minimizes its impact on the PAS calculation. Additionally, particles larger than 200 µm are probably more related to the amount and type of flocculation that occurs just before counting. Also the particle counter itself may deflocculate large particles.

# 4.3. Experimental Methods

Samples taken from WWTPs were diluted by one tenth to one fiftieth depending on the concentration estimation, in order to make the PSD testing faster and more precise. Two series of PSD tests were performed. The first was to determine the effect of holding time on PSD. The second was to develop an experimental method to simulate sedimentation that occurs in a clarifier.

To determine the effect of sample storage on PSD, samples were collected from ten different wastewater treatment plants. The samples were collected in 500 mL bottles from the effluent end of the aeration tank. Bottles were only partially filled to trap air in the bottle. Generally two samples were collected from each plant and analyzed in parallel to estimate variability. Next, samples were analyzed at the time of arrival in the laboratory and periodically over the next 72 hours. The objective was to determine if and when changes in PSD occurred. Li et al. (2005) noted that PSD in fresh stormwater samples changed (particle size increased) after 6 hours.

Samples for this test analyzed as soon as they arrived in the laboratory, which was no more than 4 hours after collection. After initial PSD analysis, they were allowed to sit on a laboratory bench at room temperature. The samples were retested after 3, 6, 12, 24, 48, and 72 hours. The samples were mixed just before PSD analysis in the sample bottle by gentle shaking and inverting. This technique was adapted from Li et al. (2005). A standard 10-mL pipette having a 1.42 mm internal tip diameter was inserted to mid depth of the sample bottle (approximately 3 inches under the surface) to collect the samples. These series of 72-hour experiments were performed for ten WWTPs.

The procedure for the analyses to estimate clarification was similar. Samples (not the same samples used in the 72-hour tests, but mostly from the same treatment plants) were immediately analyzed for PSD after they arrived in the laboratory. After the first PSD test, the samples were allowed to settle for 2 hours. PSD analysis was performed after on the supernatant (the clarified upper layer of the sample) after 0.5, 1, and 2 hours. The sample was collected with a 10 mL standard pipette having a 1.42 mm internal tip diameter. The

sample for PSD analysis was collected 2 inches under the surface or the middle of the clear zone. Care was taken to insure that the supernatant and sludge blanket in the sample bottle were not disturbed. This procedure was performed on every sample except those collected very early in the study, before the 72-hour tests were being completed.

A database of test results was created consisting of sampling date, sampling time, storage time, PAS, operation type, SRT, PSD, WWTPs' webpage, and other tables and plots among all WWTPs. There are not only WWTPs in Los Angeles, but plants in other cities or countries. The database can be expanded to include all water pollutants, pictures taken from the water samples and information related to the WWTPs.

# 4.4. Results and Discussion

#### 4.4.1. PSD Results

Figure 4.1 shows an example of PSD plots for the MLSS from Sacramento Regional Plant. Two samples were collected from the plant and each was analyzed twice, which creates four results for a single WWTP. Figure 4.1 shows the four PSD plots and it is obvious that the results are virtually the same. The difference in PAS is 4.2% or less for the four analyses. The largest difference for any single particle size is less than 10%. Similar results were obtained for integrations to 100 ad 200 µm. The result shows that

precision of the PSD analysis is quite high. Based on these results it was decided to collect and analyze two duplicate samples each time a treatment plant was sampled. PSD graphs for the other 22 WWTPs can be found in the Appendix.

The analysis shows a large number of particles in the very small range, 0.5  $\mu$ m and smaller. Sizes smaller than 0.5  $\mu$ m are of importance but are beyond the capabilities of the Accusizer 780. Particles smaller than 0.5  $\mu$ m are best analyzed using laser light scattering, as opposed to discrete counting, as performed by the Accusizer 780.



Figure 4.1. PSD plots for the MLSS tanks in Sacramento Regional wastewater treatment plants

In order to establish a cut-off point for the integration to calculate PAS (equation 4.3) samples from four treatment plants were analyzed and integrated over different size ranges. Integrations were performed from 0.5  $\mu$ m to 25  $\mu$ m, 0.5  $\mu$ m to 50  $\mu$ m, 0.5  $\mu$ m to 75  $\mu$ m etc., and up to 500  $\mu$ m, and are shown in Figure 4.2. Duplicate samples were collected and analyzed from three of the plants. Only one sample was available from the Orange County Plant.

It was hoped that a plateau of PAS would be observed as the limits of integration are increased. Figure 4.2 shows that this happened but the upper limit is somewhat different for the four plants. The PAS for the Orange County and Bellingham plants stabilized at approximately 100  $\mu$ m, while the PSD for the Tillman and Simi Valley plants did not stabilize until approximately 200  $\mu$ m. This early result confirms one of the primary hypotheses of the study. The Tillman and Simi Valley plants operated at longer SRTs (8 and 12 days) while the other two plants operate at SRTs below 2 days. The results of these analyses show that there are nearly zero particles above 100  $\mu$ m in the low-SRT MLSS; longer SRT plants have a greater abundance of larger particles. Differences are observable for upper integration limits above 50  $\mu$ m. Based on these results it was decided to calculate PAS for multiple upper limits of 50, 100 and 200  $\mu$ m.



Figure 4.2. The trends of PAS for different integration limits

#### 4.4.2. Impact of holding time on PAS

The 72-hour tests show that particles may break up, flocculate, or re-suspend after storage. Figure 4.3 shows the ratio of PAS at any given time to PAS at time zero. A ratio of 1.0 shows no change.

A number of the plants showed almost no change in PAS over time. For example, Inland Empire RP1 and RP4 show very little change – less than 20 and 10% even after 72 hours. Another plant, San Jose Creek 1 (SJC 1) showed a 50% change after just three hours. Most of the plants showed less than +20% or -10% after three hours holding. Generally, large changes can occur after 6 hours holding time.

There are also some interesting trends. San Jose Creek Water Reclamation Plant (SJC WRP) SJC 1, SJC 2 and SJC 3 all showed large changes. This is the only plant in the database that uses the step-anoxic process. In this process there is contact between primary effluent and MLSS at several points along the aeration tank, including a point close to the effluent of the tank. Therefore, primary effluent solids from this plant have had less time to interact with the MLSS and may for that reason still be rapidly changing. After 6 hours the measured PASs are very similar to the original PASs but this may just be a random artifact. Samples from ten plants were used in this experiment, and the results show that 7 plants have less than 10% difference of PAS after 12-hours; 6 plants

have less than 20% difference of PAS after 24-hours, and only 3 plants have less than 20% difference after 72-hours. If the results of SJC WRPs are separated from the analysis, there is a modest trend towards smaller particles. Deflocculation of the MLSS to smaller particles can be supported by theory, in that biomass samples that remain in an endogenous condition for extended periods loose their ability to bioflocculate (Urbain et al. 1993).

The graph and analysis shows that it is desirable to minimize the time between sample collection and analysis. Generally in this study it was possible to minimize holding time to less than 4 hours. The situation for SJC WRP biomass warrants further investigation.



Figure 4.3. PAS ratio integrated from 0.5 to 50  $\mu$  m for 72 hours among 10 WWTPs.

#### 4.4.3. Particle Average Size Result

Table 4.1 shows the information about WWTPs and reveals operation at shorter SRT leads to smaller PAS with the range of particle diameter of 0.5 µm to 50 µm. These results are shown graphically in the following sections. Three high purity oxygen activated sludge process (HPO-AS) has been sampled, which are all operated at low SRT condition. One Trickling Filter – Solids Contact Plant was sampled. The San Jose Creek plant is actually three independently operating plants are the same location and uses a step-anoxic type of process (Reardon et al, 2003), which is similar to the classical step-feed process (Tchobanoglous et al, 2003). The rest are either conventional, low-SRT plants or longer SRT plants removing nitrogen using the Modified Ludzack-Ettinger (MLE) process. This process is the most commonly used for plants that need to remove nitrogen, down to limits approximately 10 mg N/L total nitrogen. Table 4.2 compares the PAS for the same set of plants for different integration end points.

WWT plants	Date	Storage time (hrs)	WWT Type	SRT (days)	Average size
Michelson	7/8/2009	4	MLE	<u>(uujs)</u> 8.5	21.75
Orange county	9/17/2009	4	Conv. P.F.	1	11.02
Tillman	10/06/2009	3	MLE	12	21.58
Simi Valley	10/07/2009	3	MLE	8	13.11
Bellingham	10/13/2009	28	HPO-AS	2	10.06
Simi Valley	11/10/2009	6	MLE	8	12.24
San Jose City	12/5/2009	31	MLE	8	13.42
Sac. Regional	12/5/2009	11	HPO	1.5	5.71
Annacis Island	12/17/2009	20	TF-solid	2.5	7.36
Inland Empire RP1	1/13/2010	6	MLE	8	19.64
Inland Empire RP4	1/13/2010	6	MLE	8	23.80
San Jose Creek 1	1/13/2010	5	step-anoxic	12	6.96
San Jose Creek 2	1/13/2010	6	step-anoxic	12	11.29
San Jose Creek 3	1/13/2010	6	step-anoxic	12	7.73
Whittier Narrows	1/13/2010	6	MLE	12	9.46
Michelson	1/26/2010	23	MLE	8.5	10.14
SOCWA Costal	1/27/2010	6	MLE	3.5	7.17
SOCWA Regional	1/27/2010	4	MLE	4	15.55
SOCWA 3A	1/27/2010	4	MLE	2	6.34
JWPCP	2/11/2010	3	HPO-ANA	1.5	14.37
Valencia	2/22/2010	4	MLE	30	27.75
Saugus	2/22/2010	4	MLE	30	27.93
Simi Valley	2/22/2010	3	MLE	10	16.07

Table 4.1. The relationship between SRT and PAS for particle size less than 50  $\,\mu$  m

WWT plants	Date	WWTP	SRT	Integrated	Integrated	Integrated
		Туре	(day)	to 50 µm	to 100 µm	to 200 µm
Michelson	7/8/2009	MLE	8.5	21.75	47.68	66.57
Orange county	9/17/2009	Conv. P.F.	1	11.02	15.40	18.12
Tillman	10/06/2009	MLE	12	21.58	42.26	52.33
Simi Valley	10/07/2009	MLE	8	13.11	29.46	51.72
Bellingham	10/13/2009	HPO-AS	2	10.06	12.57	13.59
Simi Valley	11/10/2009	MLE	8	12.24	31.36	49.04
San Jose City	12/5/2009	MLE	8	13.42	33.22	52.14
Sac. Regional	12/5/2009	HPO	1.5	5.71	6.15	7.66
Annacis Island	12/17/2009	TF-solid	2.5	7.36	7.77	8.54
Inland Empire RP1	1/13/2010	MLE	8	19.64	36.87	48.73
Inland Empire RP4	1/13/2010	MLE	8	23.80	26.46	28.65
San Jose Creek 1	1/13/2010	step- anoxic	12	6.96	21.31	60.42
San Jose Creek 2	1/13/2010	step- anoxic	12	11.29	31.69	57.16
San Jose Creek 3	1/13/2010	step- anoxic	12	7.73	24.64	62.52
Whittier Narrows	1/13/2010	MLE	12	9.46	20.69	61.45
Michelson	1/26/2010	MLE	8.5	10.14	27.43	61.36
SOCWA Costal	1/27/2010	MLE	3.5	7.17	12.87	14.84
SOCWA Regional	1/27/2010	MLE	4	15.55	25.51	30.22
SOCWA 3A	1/27/2010	MLE	2	6.34	11.29	15.44
JWPCP	2/11/2010	HPO- ANA	1.5	14.37	14.89	15.11
Valencia	2/22/2010	MLE	30	27.75	36.11	37.82
Saugus	2/22/2010	MLE	30	27.93	43.44	60.65
Simi Valley	2/22/2010	MLE	10	16.07	29.88	37.47

Table 4.2. The comparison of PAS integrated to 50, 100, and 200  $\mu$  m.

#### 4.4.4. SRT and Particle Average Size

Figure 4.4 shows plants with larger SRT have high PAS by integrated from 0.5  $\mu$ m to 50, 100, and 200  $\mu$ m. There is a clear trend of increasing particle size with increasing SRT, using any of the three integration ranges. There seems to be two clusters of sizes with smaller particles, less than 20  $\mu$ m PAS in the SRT ranges from 1 to 4 days and greater than 20  $\mu$ m for SRTs above 8 days. The actual sizes depend on integration range and the trends are probably most distinct using 100  $\mu$ m as an upper integration limit. The very long SRT plants (Saugus and Valencia WRP) have large PAS but the increase in size is not as great as the size increase from 1-4 to 8-12 days SRT.

The "gap" in SRTs between 4 and 8 days is to be expected. Plants that do not need to remove nitrogen must operate at low SRT, typically under 4 days for warm climates such as in southern California. For reliable nitrogen removal, the SRT should be well above the minimum SRT for nitrification. Therefore, most plants that need to remove ammonia must be operated at 8 days SRT or higher.

There are several exceptional cases, for example, San Jose Creek Water Reclamation Plant (SJC WRP), Whittier Narrows Water Reclamation Plant (WN WRP), and Orange County Water Reclamation Plant (OC WRP). Theses cases are against the assumption that plants with high SRT have higher PASS. The reason for that is these plants use step-feed or step-anoxic processes in aeration tanks. Step-feed process introduces primary effluent into the MLSS at multiple positions, and some are closer to the effluent. PAS might be lower due to this special treatment. Both SJC and WN WRP have low PAS with high SRT of 12 days. OC WRP with 1-day SRT has this special treatment and PAS is a little bit higher than those plants with 2-day or 2.5-day SRT.

In Figure 4.4, MLE, tricking-filter solid contact, conventional plug flow, and HPO WWTPs are all included. Typically, HPO plants operate at low SRT and the PAS in HPO plants is lower; MLE and conventional plants must be operated at longer SRT and PAS is higher. SRT is a control variable that can be manipulated by plant management, and depends upon the season or the weather. Generally, SRT is higher in the winter time or with lower water temperature.



Figure 4.4 PAS of all the WWTPs by integrated to 50, 100, and 200  $\,\mu$  m

#### 4.4.5. 2-Hour Test for the Supernatant

The second hypothesis of this research is to show that larger particle size of the MLSS is associated with fewer effluent particles (higher quality effluent). If this were not true, the PAS of the MLSS would be relatively unimportant. Lower PAS in MLSS should result in poorer sedimentation and more resuspension in the event of poor clarifier performance.

The two-hour test described previously was used to simulate clarifier performance. The number of particles in the supernatant of quiescent samples was measured at 0.5, 1 and 2 hours, roughly corresponding to the retention time of clarifiers used in secondary treatment. The results from the three time periods were virtually identical and the 0.5 hour (30 minute) period was chosen for comparison.

Figure 4.5 shows the number of particles in the clarified effluent versus MLSS PAS. WWTPs with smaller MLSS PAS have more particles in the supernatant than those with high PAS. This trend occurs irrespective of the cut-off point for integration. Cut-off points of 100 and 200 µm are shown in Figure 4.6 and 4.7, respectively.

The results indicate that low SRT WWTPs generally contain more particles and potentially more adsorbed pollutants than high SRT WWTPs.



Figure 4.5. Particle numbers of supernatant from 0.5 to 50  $\mu$  m after 30-min settling. (MLE= Modified Ludzack-Ettinger; HPO= high purity oxygen; No label= conventional; ANA= anaerobic/aerobic selector; SRT= solids retention time)



Figure 4.6. Particle numbers of supernatant from 0.5 to 100  $\mu$  m after 30-min settling. (MLE= Modified Ludzack-Ettinger; HPO= high purity oxygen; No label= conventional; ANA= anaerobic/aerobic selector; SRT= solids retention time)



Figure 4.7. Particle numbers of supernatant from 0.5 to 200  $\mu$  m after 30-min settling. (MLE= Modified Ludzack-Ettinger; HPO= high purity oxygen; No label= conventional; ANA= anaerobic/aerobic selector; SRT= solids retention time)

### 4.4.6. Impact of Types of WWTPs with Same SRT

The results of this work begin to show some additional differences in PSD and process type. There is a clear trend of larger PAS with increasing SRT. Certain process configurations, such as MLE are only defined for longer SRT (above the minimum SRT for nitrification). Other configurations may operate at both high and low SRT.

The database includes only one HPO treatment plant which shows very high effluent particle number. Generally HPO plants are thought to have more turbid effluents. The step-anoxic plant had unusually small PAS. Further work will be needed to support these observations.

# 4.5. Conclusion

Prior to this work, suspended matter from wastewater treatment plants was defined by cruder techniques such as total suspended solids, volatile suspended solids or turbidity. This work has shown that newer techniques for particle size analysis can be used to characterize activated sludge plant operation and effluent quality. The differences in operation can now be quantified and used to justify modified and improved plant operational strategy. The following specific conclusions are made:

- 1. A series of 72-hour tests showed that particle size can change with sample storage. Generally the time between sample collection and analysis should be minimized. Large changes in particle size were observed with some samples even after 6 hours holding time. Samples from 10 plants showed that 7 plants have less than 20% difference of PAS in a 12-hour period; 6 plants have less than 20% difference of PAS in 24-hour period; only 3 plants have less than 20% difference of PAS in 72-hour period. Samples from 9 of the 10 plants had smaller particle size after 72-hours, suggesting that deflocculation is important.
- It is possible to describe the PSD of MLSS samples using a single parameter which is calculated from an integration of the PSD from 0.5 to 50, 100, or 200 μm. All three integration end points can be used to distinguish differences in

PSD and SRT. Integration to 200  $\mu m$  was more useful in differentiating longer SRT treatment plants.

3. An easily observable trend was noted between average MLSS particle size and the number of effluent particles. This result is the most important observation and is consistent with sedimentation theory.

# Appendix.

Pictures of the water sample and supernatant sample




















































Particle Diameter (um)

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## **Chapter 5. Conclusion**

This dissertation focuses on particle characteristics such as mixing properties in stormwater and in wastewater. Two parts of the main theory have been verified. The first part is that traditional method of suspended solids measurement, TSS, is comparable to SSC method if a wide-bore pipette and proper mixing are used. It has also demonstrated that serious errors can or may have occurred because of poor subsampling. These results were used to estimate pollutant removal efficiency for stormwater best management practices. The trends show little difference between using TSS and SSC for low overflow rates, but the difference becomes lager as the overflow rate increases, which might result in larger particles passing to the effluent. Suspended solids estimation errors involved in TSS or SSC methods may partially explain the poor performance reported in the literature of certain BMPs. TSS subsampling method should be able to accurately sample effluent suspended solids. The SSC method should be used only when larger particles are expected or when the entire sample can be used for SSC without penalty.

The results have also shown that attempts to correlate previously collected TSS and SSC data will not be possible in general, unless the PSD and recovery of different size particles are known or can be inferred from experimental conditions.

The second part is to distinguish the relationship between particle size distribution and sludge retention time. Activated sludge plants operating at high SRT have, on average, greater mean particle size in MLSS tanks. A simple indicator, PAS, is developed to calculate the mean particle size of the sludge. The particle size can be integrated form 0.5  $\mu$ m up to 500  $\mu$ m. Twenty-three wastewater treatment plants were sampled and the results show that plants with long SRT have larger particles in their aeration tanks. In some special cases, plants with long SRT may have small particles because these plants adapt step-feed or step-anoxic process to operate the aeration tanks.