Comparison of Stormwater Solids Analytical Methods for Performance Evaluation of Manufactured Treatment Devices

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Abstract: As more manufactured stormwater treatment devices enter the market, stormwater managers are searching for effective and rapid methods for evaluating device performance. Many agencies require vendors to test full-scale versions of their devices under controlled conditions. The most common parameter used to document performance is suspended solids for several reasons: (1) many pollutants attach to solids; (2) a solids simulant is relatively easy to generate; and (3) solids are comparatively easy and inexpensive to quantify. However, a controversy still exists in the profession and some regulatory agencies as to whether total suspended solids (TSS) or suspended sediment concentration (SSC), or both, should be measured. This paper focuses on the comparability of the two methods/protocols used for sample solids analysis, including lessons learned during recent evaluations of two manufactured treatment devices. Analysis of 215 sample pairs (where both TSS and SSC were measured on aliquots of the same sample) showed that statistically the TSS measured using the wide-bore pipet method and SSC results were indistinguishable from one another and from the original simulant mixture.

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Introduction

The marketplace of new manufactured stormwater treatment devices is growing rapidly. Past history of these devices has shown that the performance claims vary widely, based on the vendor’s testing. The result has been that, once these devices are installed in the field, the performance may not be at the level claimed by the vendor. Because of these discrepancies and in order to help the stormwater manager select a device based on comparable testing, several states and the federal government have developed and implemented standard testing protocols, which are managed through approved verification agencies. For example, the U.S. Environmental Protection Agency (USEPA) has NSF International’s Water Quality Protection Center (NSF Inc. 2006) acting as its verification agency as part of its environmental testing-verification (ETV) program. The verification agency/organization typically develops the required testing protocol and then selects an organization to conduct the actual testing. At the end of testing, the verification organization provides a recommendation for the anticipated performance range of a particular treatment device, or verifies the vendor’s performance claims.

Two possibilities generally exist for performing these full-scale verification activities: field installations versus laboratory testing. Because of the time required and the lack of control on the field test site, laboratory testing is often preferred by vendors and is gaining favor with some agencies for, at a minimum, interim certification. A comparison between two or more devices claiming to address a specific pollutant is much easier with controlled laboratory testing.

Many of the laboratory-testing protocols focus first on solids removal for two reasons: (1) it is the primary parameter of interest for most regulatory agencies; and (2) it is considered the easiest parameter to simulate. There are no interfering reactions to simulate and no associations between dissolved and particulate pollutants to investigate. Laboratory testing does have its limitations, however. Many of these concerns were addressed by Guo (2005) and include the ability to accurately capture and quantify the particles represented in the prescribed test mixtures. This paper focuses on the differences found in the two most common methods of solids measurement—total suspended solids (TSS) and suspended sediment concentration (SSC). The large sample sets discussed in this paper were generated using simulant solids mixtures used by two teams of researchers at Penn State Harrisburg and the University of Alabama as they verified the solids’ removal performance of two prototype and full-scale devices. In addition, these results are compared with a set of field data generated from ETV testing of one of the two treatment devices. The results of aliquots of same sample analyzed for both TSS and SSC are compared statistically.

Methodology

Much of the past research on stormwater runoff particulates has used the TSS methodology (either by EPA or by Standard Meth-
particles that may be collected in natural water, including storm-water, samples. That analysis technique is ASTM D3977-97(B) (ASTM 1997) (reapproved in 2002). A summary of the three methods is shown in Table 1.

As part of two verification projects—one for a sedimentation device and one for a filtration device, the researchers performed TSS and SSC on both influent and effluent samples, ending up with approximately 215 matched pairs. The simulant for each of these tests consisted of a solids distribution similar to that required in the NJ DEP Laboratory Testing Protocol (clay 1–2 μm, 5%; silt 2–8 μm, 15%; silt 8–50 μm, 25%; very fine sand 50–100 μm, 15%; fine sand 100–250 μm, 30%; medium sand 250–500 μm, 5%; coarse sand 500–1,000 μm, 5%). These simulants were made using sieved sand and Sil-Co-Sil 250. For the filtration device, the sample had a portion of the sand replaced by a ground fertilizer to add phosphorus to the challenge mixture. The mix water source was an artesian well in the case of the sedimentation device and tap water for the filtration device. The simulated particles were silica based to meet the NJ DEP recommendation of a specific gravity of 2.65.

Each 500-mL influent and effluent sample from each device was split using a Dekaport cone splitter into five subsamples. An error analysis was performed for the cone splitter during a prior project, and the results showed that, over the particle size and concentration range of interest for this project, the median coefficient of variation (COV) (calculated as standard deviation/mean) between splits of the same sample was 5%, with lower COVs for higher concentration samples. One split was retained for particle size distribution analysis using the Coulter Multisizer 3, and four were used for solids analysis (TSS, TSS sieved to <250 μm, SSC, and SSC sieved to <250 μm). Because the sample bottles were filled to the brim, the actual volume split exceeded 500 mL and were approximately 115–120 mL. The TSS analyses were performed following the Standard Methods protocol 2540D using a wide-bore pipet. To reduce analytical bias, the pipet tip was not allowed to contact the bottom of the stirred sample bottle, even as the water level dropped in the sample bottle. In addition, the convex nature of the bottom of the bottles ensured that the pipet aliquot was not unduly biased by sampling those solids that did not suspend but traveled in a slurry along the bottle bottom. Additional testing (Clark and Sui 2007) by the research team of the wide-bore pipet TSS method has shown that the percentage of sample in the aliquot did not influence the solids recovery. The two subsamples were sieved to remove particles larger than 250 μm in order to quantify the mass of large particles in the original samples. These results, along with the results of the TSS/SSC sieved, are not relevant to and are not included in this paper.

<table>
<thead>
<tr>
<th>Filter nominal pore size</th>
<th>EPA TSS (160.2)</th>
<th>S.M. TSS (2540D)</th>
<th>USGS SSC (D3977-97(B))</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sample mixing</td>
<td>Not specified</td>
<td>&lt;2.0 μm</td>
<td>1.5 μm</td>
</tr>
<tr>
<td>Aliquot size</td>
<td>Not specified</td>
<td>Stir plate</td>
<td>Decant supernatant and flush bottle with DI water</td>
</tr>
<tr>
<td>Method of aliquot collection</td>
<td>Pour aliquot into graduated cylinder</td>
<td>Pipet at middepth in bottle and midway between wall and vortex</td>
<td>Entire sample</td>
</tr>
</tbody>
</table>

Table 1. Comparison of Three TSS/SSC Analytical Methods

In addition to the concerns over the ability to analyze across the variety of solids found in stormwater runoff, different organizations/agencies have slightly different methods for analyzing TSS. Two of the most common TSS methods are from the US EPA Method 160.2 (USEPA 1999) and from Standard Methods for the Analysis of Water and Wastewater, Method 2540D (APHA 1995). These solids’ characterization methods differ in two important ways: (1) the specification, or lack thereof, of the nominal pore size of the filter (or of the efficiency rating); and (2) the method used to collect the sample aliquot. Both of the TSS methods are documented to have problems capturing the larger particles found in simulated stormwater. For example, Standard Methods requires sampling middepth, yet that sampling location is likely to miss the larger particles that cannot be adequately suspended evenly in the water column. The EPA method requires a shake-and-pour aliquot selection, with the larger particles settling in the original sample container before pouring is completed. Therefore, several verification agencies have also been requiring SSC measurements. The purpose of requesting both measurements is so that comparisons to historical data can be performed.

Most historical stormwater solids concentrations were measured using one of the two TSS methodologies. The SSC analysis method was developed by the American Society for Testing and Materials (ASTM), and endorsed by the USGS, as a method better able to capture and quantify larger particles that may be collected in natural water, including stormwater, samples. That analysis technique is ASTM D3977-97(B) (ASTM 1997) (reapproved in 2002). A summary of the three methods is shown in Table 1.

As part of two verification projects—one for a sedimentation device and one for a filtration device, the researchers performed TSS and SSC on both influent and effluent samples, ending up with approximately 215 matched pairs. The simulant for each of these tests consisted of a solids distribution similar to that required in the NJ DEP Laboratory Testing Protocol (clay 1–2 μm, 5%; silt 2–8 μm, 15%; silt 8–50 μm, 25%; very fine sand 50–100 μm, 15%; fine sand 100–250 μm, 30%; medium sand 250–500 μm, 5%; coarse sand 500–1,000 μm, 5%). These simulants were made using sieved sand and Sil-Co-Sil 250. For the filtration device, the sample had a portion of the sand replaced by a ground fertilizer to add phosphorus to the challenge mixture. The mix water source was an artesian well in the case of the sedimentation device and tap water for the filtration device. The simulated particles were silica based to meet the NJ DEP recommendation of a specific gravity of 2.65.

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Results

Analysis of Entire Data Set

The 215 paired influent and effluent TSS and SSC samples were compared through a scatterplot analysis shown in Fig. 1. A linear regression of these data, assuming that the intercept is zero, showed that the SSC measurements were predicted to be 1.4% less than the TSS measurements from the same sample (SSC = 0.986 × TSS). The slope coefficient was significant with \( p \ll 0.001 \). However, the 95% confidence interval on the slope coefficient was 0.935–1.04, indicating that the slope coefficient, while statistically significant, could not truly be distinguished from 1.0. The direction of this statistically insignificant result would be needed for the differences in the data sets to be confirmed statistically.

As noted in the method discussions, subsampling techniques can greatly influence the results (Fig. 3 shows the same data as above, but broken down by analyst). Analysts 1–3 and Analyst 5 were trained in the methods thoroughly by previous analysts prior to commencing sample work, including on the difficulties of obtaining a “representative” subsample for TSS analysis. Analyst 4 was given the TSS method (using Standard Methods protocol) and a set of samples. Analyst 4 therefore followed the Standard Methods recommendation of sampling midway between the vortex and the wall at middepth, while the others were within 1 cm of the bottom of the bottle and midpoint between the wall and the vortex. For Analyst 4, TSS measurements were approximately one half those of the corresponding SSC measurements. Sampling at the middepth location was insufficient for retrieving a representative fraction of the larger particles which tended to slide along the bottom of the beaker or bottle. Unlike the treated sanitary wastewater that this method was designed to analyze, the

Influence of Subsampling Techniques

The regression equations for these analyses show that the relationship between the SSC and the MB calculation is SSC = 1.02 × MB, whereas for TSS, it is TSS = 0.72 × MB. Both slope coefficients are statistically significant with \( p \ll 0.001 \). An analysis of variance (ANOVA) between the three groups of samples (TSS, SSC, MB) indicates that, for the number of samples in each group, none of the groups could be distinguished statistically from the other \( (p > 0.01 \) for all combinations). Therefore, while it appears in Fig. 2 that the SSC and the MB are the best replicates of each other, the TSS data cannot be statistically distinguished from them. From a reporting standpoint, the entire dataset could be combined and used to document performance, as opposed to separate reporting for TSS and SSC required by the agencies. Given the variability in the data set, substantially more samples would be needed for the differences in the data sets to be confirmed statistically.
The regression equations and the 95% confidence intervals on the slope coefficient are shown in Table 2. All slope coefficient values were statistically significant at the $p < 0.001$ level. $R^2$ values ranged from 0.66 to 0.95. Most of the confidence intervals contained the slope coefficient of 1.0, indicating that for the various analysts, TSS and SSC measurements for this data set were statistically indistinguishable from one another. Only Analyst 3’s confidence intervals for the slope coefficient did not contain 1.00, thus statistically indicating a slight bias.

According to Gray et al. (2000), an analysis of paired TSS and SSC samples showed that TSS results were typically between 25 and 34% less than the paired SSC sample. According to this testing, with the exception of Analyst 4, SSC results were between $-10$ and $+10\%$ of the TSS values. For Analyst 3 (the only analyst whose 95% confidence interval on the slope coefficient does not contain 1.00), the SSC bias was approximately $+10\%$ of the TSS values. TSS values were not consistently negatively biased, unlike the results found by Gray et al. (2000). Additionally, the results shown as Analyst 4 demonstrate the importance of subsampling near the bottom of a container when using the TSS method if the goal is to have the TSS results reflect all solids in the water and not just those that are well mixed in the water column. TSS subsampling near the top or middle of sample bottles that have a substantial fraction of larger particles will lead to the biases similar to or larger than those documented by Gray et al. (2000).

**Influence of “Large” Particles**

The second notable result from Gray et al. (2000) was that the size of the difference between the SSC and TSS result was greatest in samples where the sand fraction (>62 μm) equaled/exceeded 25% of the sediment. Their results showed that TSS was relatively similar to the SSC results when most of the particles are finer than 62 μm. To determine the effect of a substantial fraction of particles being sand sized (>250 μm for these tests) on the TSS and SSC results, the data in Fig. 3 were reanalyzed using only the influent samples, since it was shown that the effluent samples had very few, if any, particles >250 μm (less than 10% of the effluent samples had solids >250 μm). The influent-only sample results are shown in Fig. 4 and Table 2. According to the testing, with the exception of Analyst 4 (see description above of effect of in-bottle sampling location on results), SSC results were between $-13$ and $+10\%$ of the TSS values. TSS values were not consistently negatively biased in the influent-only analysis. The regressions of this data show that the

![Fig. 3](image1.png)  
**Fig. 3.** SSC versus TSS for 215 full-scale verification tests

![Fig. 4](image2.png)  
**Fig. 4.** SSC versus TSS for 112 full-scale verification tests using influent samples

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**Table 2.** Regression Equations for Influent+Effluent and Influent-Only Sample Sets

<table>
<thead>
<tr>
<th>Analyst</th>
<th>Influent+effluent equation (SS=a*TSS)</th>
<th>Influent-only equation (SS=a*TSS)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>SSC=0.904*TSS ($R^2=0.953$, CI: 0.757, 1.05)</td>
<td>SSC=0.896*TSS ($R^2=0.927$, CI: 0.690, 1.10)</td>
</tr>
<tr>
<td>2</td>
<td>SSC=1.004*TSS ($R^2=0.715$, CI: 0.857, 1.15)</td>
<td>SSC=1.003*TSS ($R^2=0.707$, CI: 0.797, 1.21)</td>
</tr>
<tr>
<td>3</td>
<td>SSC=1.104*TSS ($R^2=0.953$, CI: 1.04, 1.17)</td>
<td>SSC=1.104*TSS ($R^2=0.952$, CI: 1.01, 1.19)</td>
</tr>
<tr>
<td>4</td>
<td>SSC=1.902*TSS ($R^2=0.661$, CI: 0.998, 2.08)</td>
<td>SSC=2.015*TSS ($R^2=0.669$, CI: 0.385, 3.64)</td>
</tr>
<tr>
<td>5</td>
<td>SSC=0.994*TSS ($R^2=0.870$, CI: 0.828, 1.16)</td>
<td>SSC=0.878*TSS ($R^2=0.865$, CI: 0.647, 1.11)</td>
</tr>
<tr>
<td>Field</td>
<td>SSC=0.935*TSS ($R^2=0.954$, CI: 0.838, 1.03)</td>
<td>SSC=0.898*TSS ($R^2=0.943$, CI: 0.742, 1.05)</td>
</tr>
<tr>
<td>All analysts</td>
<td>SSC=0.986*TSS ($R^2=0.872$, CI: 0.935, 1.04)</td>
<td>SSC=0.960*TSS ($R^2=0.848$, CI: 0.884, 1.04)</td>
</tr>
</tbody>
</table>
Conclusions

As more manufactured treatment devices enter the marketplace, stormwater managers need to document that the device performs as anticipated. For this reason, many verification agencies have incorporated a laboratory testing component into their approved protocols for documenting device performance. However, questions remain about setting up a representative laboratory program that can adequately represent field conditions and about how to accurately measure the solids concentration in the device’s influent and effluent.

Stormwater researchers and managers have been debating as to whether to use TSS or SSC to quantify the solids concentration in runoff samples and the ability of the treatment device to remove these solids. Other researchers, including those of the USGS, found that TSS measurements were consistently lower than paired SSC measurements by up to 25–34%. This research has outlined the inconsistencies of the three traditional solids analysis methods (such as the filter pore size—which, if specified, differs between methods and between researchers—and the method of obtaining subsamples for filtering) and the differences obtained between the two methods for this laboratory. The results have shown that, for the same analytical filters, the bias between TSS (Standard Methods 2540D) and SSC is not as great as seen by the USGS for the particle-size distribution of the challenge solution samples, and the differences are not statistically significant. However, substantial differences can exist between analysts, if training methods are not consistent. This is particularly crucial when performing TSS analysis, which uses a small subsample of the original sample. If the aliquots are not collected at a location in the mixing sample where a representative fraction of the larger solids in the mixture can be collected by the pipette, SSC results will be measurably greater than the TSS results for the same sample.

This research also showed that the mass balance method of calculating influent solids concentration (where “we know what we added”)—the simplest and most reliable—can be well represented in both the TSS and SSC measurements. Therefore, there is an argument to be made that mass balance calculations are comparable to the more time-consuming laboratory analyses and could be substituted for the analytical efforts on the influent side. This research shows that they are statistically identical. However, if it is desired to have an analytical result that most “resembles” the entire mass of solids added, then the SSC method is recommended since it provides the “closest” results to the known solids concentration. If the goal is to measure the solids that are well mixed in the water column, then the TSS method may be most appropriate.

This research has shown that many questions still exist in how to document the performance of manufactured treatment devices, especially when developing and implementing full-scale laboratory testing protocols. Therefore, it is incumbent upon stormwater managers who are interpreting the results of verification testing to determine the methods of sample analysis used and match the analytical methodology with the goals of the program. Differences in each of these between vendor verification testing can impact on the results. The purpose of standardized laboratory testing has been to ensure that the comparison is “apples to apples” but the lack of attention to the details of testing and analysis has left the profession examining devices using a “red apples to green apples” approach.
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References


