Measuring solids concentrations in urban runoff: Methods of analysis

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ABSTRACT
Various types of solids conveyed with rainfall and snowmelt runoff into receiving waters cause numerous environmental impacts, including reduced sunlight penetration, blanketing of fish spawning substrates, and transport of pollutants contributing to aquatic pollution. For the assessment of such impacts, it is important to measure solids concentrations in both runoff and snowmelt. In this study, accuracies of three analytical methods used to measure solids were assessed: (a) A TSS (total suspended solids) method, (b) Suspended sediment method (SSC-B), and (c) a multiple filter method (MFM). For rainfall runoff samples containing 90% of particles smaller than 5 µm, the MFM measurements produced concentrations significantly higher than those obtained with SSC-B and TSS methods, at a 95% confidence level. In the case of snowmelt runoff, the SSC-B and MFM methods yielded similar concentrations, which were 10-20% higher than those measured by the TSS method, and the coefficient of variation of repeated TSS readings was up to three times higher than that of the former methods. The results indicate the importance of choosing the “best” analytical method for assessing the operational and environmental impacts of solids conveyed by urban runoff and snowmelt.

KEYWORDS
TSS, total suspended solids; SSC, suspended sediment concentration; MFM, Multiple Filter Method

INTRODUCTION
Rainwater contains particles scavenged from the atmosphere and as it is converted into runoff and flows over the catchment surface (paved streets, parking lots, and building rooftops), it collects and transports more particles with attached pollutants. In cold climate regions, the choice of snow handling practices and the use of anti-skid materials and road salts further affects the runoff quality (Reinosdotter and Viklander 2006; Reinosdotter and Viklander 2007). Thus, urban runoff may become polluted with such pollutants as heavy metals, trace organic compounds, nutrients and sediments (Hvitved-Jacobsen and Yousef, 1991; Viklander, 1997). Many studies have shown that the highest concentrations of pollutants, e.g., heavy metals and polycyclic aromatic hydrocarbons (PAHs), are preferentially associated with the smallest sediment fractions found in urban runoff and snowmelt (Lau and Stenstrom, 2005; Sansalone and Buchberger, 1997; Westerlund and Viklander, 2006).

Rainfall and snowmelt runoff, which contains high concentrations of small solids, is a major cause of suspended solids and sediment discharges into receiving waters, where such solids cause numerous impacts, including increased turbidity, reduced sunlight penetration, blanketing of fish spawning substrates, and transport of pollutants contributing to aquatic pollution (U.S.EPA, 1993).

In Sweden, municipal and commercial laboratories commonly use the conventional SS-EN 872:2005 method for determining suspended solids in wastewater and source water. An earlier comparison of analytical methods showed that the total suspended solids (TSS) method yielded noticeably lower solids concentrations compared to the suspended sediment concentration method (SSC) (Gray *et al.,* 2000 and Clark *et al.,* 2008). Also, in a study by Nordqvist *et al*. (2010) with a synthetic stormwater, the SSC-B method and the multiple filter method (MFM) yielded noticeably higher solids concentrations for large particles, compared to the TSS method. While coarse particles are important for estimating total solids loads (e.g., filling up of pond storage), small particles carrying high concentrations of contaminants are important for assessing contaminant transport and water quality implications.

The main objective of this paper is to compare the accuracy and precision of three different methods used to measure the total solids concentrations in urban runoff in different conditions reflecting seasonal variations. The methods to be compared are the newly proposed Multiple Filter Method (MFM) and the two existing methods: (a) a conventional TSS analysis method (described here as SS-EN 872:2005) and ASTM D 3977-97, 2007 Test Method B (SSC-B).

METHODS

Analytical methods

The total particle concentrations in urban runoff can be determined by the Multiple Filter Method, MFM. In this method, the whole sample is vacuum filtered through three filters with different pore sizes (25, 1.6 and 0.45 µm), with the smallest size filter being on the bottom. The filters with pore sizes 25 and 1.6 µm were used to avoid clogging the 0.45 µm filter. Following vacuum filtration, the filters are dried at 105°C ± 2° C and the mass of the residue on the filters is determined by weighing.

The conventional determination of suspended solids, TSS-analysis (SS-EN 872:2005) is used to determine suspended solids in raw waters, wastewaters and treated wastewater effluents by filtration through a glass fibre filter. The lower limit of the determination is about 2 mg/l, but no upper limit was established. The principle of the TSS method is to use a vacuum or pressure filtration apparatus and filter the sample (a well-mixed aliquot withdrawn from the sample bottle using a graduated cylinder) through a glass fibre filter with an average pore size of 1.6 µm. The sub-sample volumes used in this study varied from 25 to 250 ml (to obtain a sufficient mass of TSS), with the largest volume used in the case of undisturbed bulk snow. Subsequently, the filter was dried at 105°C ± 2°C and the residue mass on the filter was determined by weighing.

The Standard Test Method for Determining Sediment Concentration in Water Samples, SSC-B (ASTM D 3977-97, 2007), is used for water samples from lakes, reservoirs, ponds, streams, and other water bodies. The principle of the test is to weigh the entire sample consisting of sediment and dissolved solids, and then filter the sample through a glass fibre filter with pore size of 1.5 µm using porcelain or borosilicate glass crucibles with fritted glass bases. In this study, a Whatman GF/A glass fibre filter with a pore size of 1.6 µm was used, the same pore size as used in the TSS analysis. The crucibles were then dried at 105°C and the residue mass of the sediment was determined by weighing.

Sample sets studied

The rainfall runoff samples were collected from a manhole connected to a gully pot at Södra Hamnleden in the central part of the City of Luleå. Three samples were collected during each of the three different rainfall events, and each sample was split into five subsamples of 300 ml each. For every rainfall event, 15 sub-samples were analysed with each method; thus, 135 samples were analysed in total.

In order to study the influence of the winter season, two snowmelt runoff events (snowmelt runoff 1 and 2) and one undisturbed bulk snow samples were collected. The undisturbed bulk snow sample was collected at the sites where snow had not been affected by snow handling, and the snowmelt runoff was collected from the gully pot at Södra Hamnleden. After the snow was melted, the resulting snowmelt was sieved (500 µm mesh). The choice of mesh size was guided by the earlier studies showing that most of the pollutant burdens were associated with particles smaller than 500 µm (Pitt and Amy, 1973; Woodward-Clyde, 1994; and Vaze and Chiew, 2004). Subsequently, sub-samples were withdrawn from the sample container for analysis. The sub-sample volume was chosen as 300 ml for snowmelt and 1000 ml for the bulk snow, due to low concentrations of TSS. For snowmelt runoff 1 and 2 as well as undisturbed bulk snow, 5 sub-samples were analysed by each method; in total, 45 samples were analysed.

RESULTS AND DISCUSSION

Rainfall runoff

The particle concentrations varied up to 10 times for rainfall runoff samples (see Table 1). Factors that affect the concentration and particle distributions in urban runoff include intensity and duration of rainfall, duration of dry periods between rain events and human activities. For rainfall runoff, SSC-B and TSS tend to yield lower particle concentrations compared to MFM.

Table 1. Particle concentrations in rainfall runoff samples (mg/l)

	Rainfall event 1			Rainfall event 2			Rainfall event 3 *		
Method	SSC-B	MFM	TSS	SSC-B	MFM	TSS	SSC-B	MFM	TSS
Sample 1									
Sub-sample									
1	125	113	88	217	220	180	92	99	86
2	132	141	100	198	227	183	98	102	86
3	136	139	103	202	223	178	94	99	81
4	126	132	99	228	228	184	101	105	84
5	96	139	98	195	218	175	96	98	88
Sample 2									
Sub-sample									
1	85	148	140	16	20	12	22	27	22
2	108	119	140	12	16	12	23	24	19
3	95	128	134	11	16	12	21	27	19
4	130	124	136	13	16	11	22	28	18
5	102	126	131	16	16	11	21	28	18

Sample 3									
Sub-sample									
1	34	68	58	76	85	73	67	68	61
2	59	75	57	79	84	73	59	66	58
3	40	73	53	76	82	72	64	69	60
4	58	72	57	78	86	71	60	68	57
5	59	65	53	75	83	73	67	72	56

* Particle size distribution analysis was performed using Beckman Coulter Multisizer 3. The three samples from rain event 3 were analysed, with results showing that 90% of the particles were < 5 µm.

To compare different methods, the average of five sub-samples for each individual method was divided by the average of the sub-sample concentrations for all the three methods (15 samples in total, see Fig. 1). Figure 1 shows that the MFM method yielded higher particle concentrations than methods SSC-B and TSS (except for rainfall 1:2). This could be explained by the high concentrations of small particles in the samples and the fact that the MFM method measured even the smallest particles (<1.6 µm), since the smallest filter pore size was 0.45 µm, compared to 1.6 µm in the case of both TSS and SSC-B methods, which had the smallest filter pore size of 1.6 µm. Nordqvist *et al.* have shown that TSS underestimated the actual concentrations because of the presence of large particles, which are not picked up when withdrawing sample aliquots for conducting the analysis.

The TSS analysis tended to yield a lower coefficient of variation (CV) than SSC-B and MFM (Figure 1), probably because the large and heavy particles, which influence the solids concentrations more than the small particles, were not included in the TSS analysis. The MFM analysis tends to yield lower CVs than SSC-B, which may be due to the fact that a fraction of small particles will be transported through the filter with a large pore size.

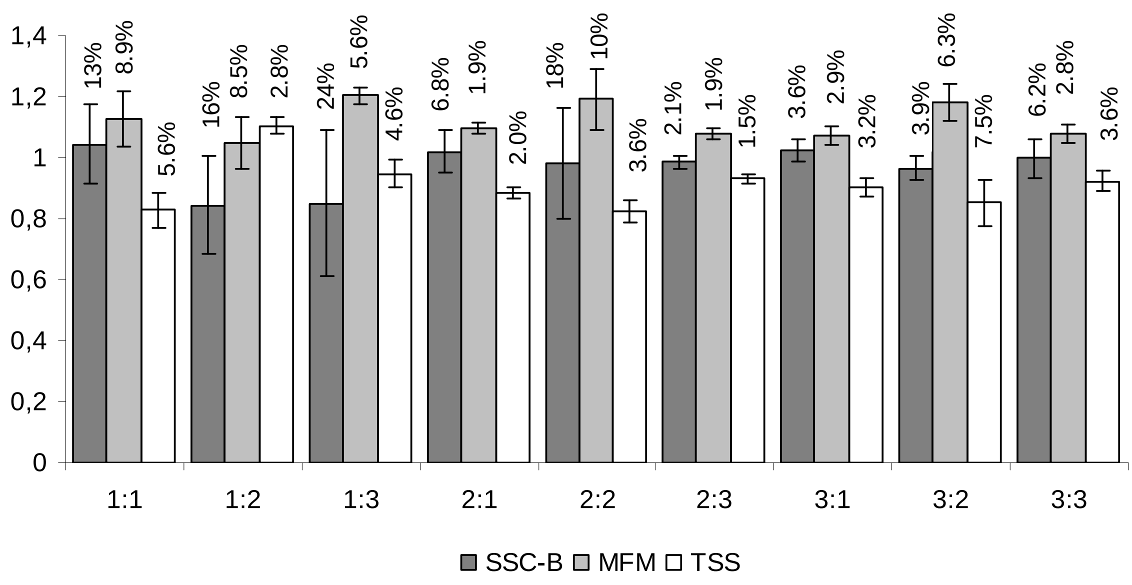

Figure 1. The average of five sub-samples for each individual method divided by the average of the sub-samples for all the methods (15 samples in total). CV (%) was calculated for each individual method, for rain events 1-3 (rain event : sample).

For all the samples, a statistical analysis was performed using a one-way ANOVA test to determine if a significant difference existed between the methods for different samples. The ANOVA test gave a p-value of less than 0.05, i.e. the particle concentrations for MFM were significantly higher than the SSC-B and TSS concentrations, at a 95% confidence level. The ANOVA test yielded no significant differences between the SSC-B and TSS results.

The results further showed that MFM yielded the highest particle concentrations and the ANOVA test implied that the particle concentrations produced by MFM were significantly higher compared to the SSC-B and TSS concentrations. In the case of rainfall runoff, small particles (0.45-1.6 µm) influence the results. Also, a visual observation supported the notion of the influence of 0.45-1.6 µm particles, since the filtrate from MFM was more transparent than that from the SSC-B and TSS methods.

For rainfall runoff, with high concentrations of small particles, the results showed that MFM yielded the highest particle concentrations, except for sample 1:2. TSS produced the lowest CV and significantly lower particle concentrations compared to MFM. However, in this case, the low CV does not reveal if the method measured the desired output; i.e., all the particles in the sample. Instead, the low CV could be explained by the relatively uniform particle distributions in the samples. For the TSS methods, the analysis was performed on sub-samples withdrawn from a large sample and a visual observation showed that the largest particles tended to slide along the bottom of the large sample container and, therefore, were not included in the sub-samples analyzed. An earlier study by Nordqvist *et al.* (2010) showed that the TSS method underestimates particle concentrations for samples containing particles larger than 63 µm (0.063 mm). These larger particles influence the variance more than smaller particles; hence, the low value of CV in the TSS method.

Snowmelt runoff

The results for snowmelt runoff 1, snowmelt runoff 2 and undisturbed bulk snow in Table 2 show that snowmelt runoff samples contained solids concentrations about 15-80 times higher than those in undisturbed bulk snow. For snowmelt runoff 1, the particle concentrations were 2.5-3 times higher than for snowmelt runoff 2. The TSS method yielded a larger spread between the min and max values for snowmelt sub-samples compared to the SSC-B and MFM methods. The SSC-B and MFM produced somewhat diverging results for one of the undisturbed snow sub-samples, Nos. 2 and 5, respectively.

Table 2. Solids concentrations (mg/l) in snowmelt runoff events 1 and 2, and undisturbed bulk snow samples

	Snowmelt runoff 1			Snowmelt runoff 2			Undisturbed bulk snow		
	SSC-B	MFM	TSS	SSC-B	MFM	TSS	SSC-B	MFM	TSS
Sub-sample									
1	1083	1148	1158	377	367	303	19	18	18
2	1528	1246	923	381	328	284	22	19	19
3	1475	1289	924	378	345	284	19	18	17
4	1428	1130	982	377	346	291	18	18	17
5	1214	1373	1512	396	368	349	19	24	18

Figure 2 shows that the CV of the TSS method readings was considerably higher (1.5-3 times) for snowmelt runoff compared to that of the SSC-B and MFM methods. Compared to the other methods, TSS results were influenced more by large particles and indicated the largest spread among the measured snowmelt sub-samples (see Table 2). For undisturbed bulk snow samples, CV was higher for the SSC-B and MFM methods, which could be explained by the diverging sub-samples in each sample set. The higher concentrations in these two sub-samples could be explained by the presence of one or more large particles. However, if the diverging sub-samples were disregarded, these methods resulted in the same or better CV values compared to the TSS method (the corresponding CVs were 1.8 and 3.6%, for SSC-B and MFM, respectively).

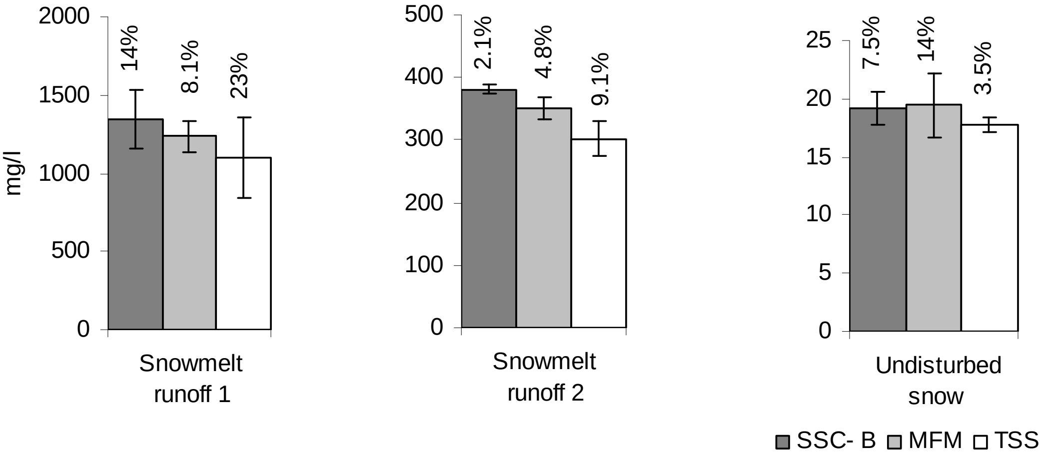

Figure 2. Measured mean solids concentrations (mg/l) and CVs (%)

To determine if a significant difference existed between the methods for measuring particles in snowmelt runoff and undisturbed bulk snow, a statistical analysis was performed using a one-way ANOVA test. The ANOVA test yielded a p-value less than 0.05 for sample snowmelt runoff 2, meaning that the SSC-B and MFM methods yielded significantly higher particle concentrations compared to the TSS method, at a 95% confidence level. For snowmelt runoff 1 and undisturbed bulk snow samples, the ANOVA test showed no significant differences between the methods.

In the case of rainfall runoff, the MFM method with the smallest filter pore size of 0.45 µm resulted in significantly higher particle concentrations than the SSC-B and TSS methods. Evidently, for rainfall runoff where most of the particles are small, the particle size will influence the results and the MFM method will produce the most accurate concentrations. In environmental studies, small particles are of interest because of their impact on surface water quality by increasing turbidity, reducing sunlight penetration and changing habitat for aquatic life (EPA, 1993). Small particles are also of interest in contaminant transport, because they contain the highest contaminant concentrations (Sansalone and Buchberger, 1997).

The SSC-B, MFM and TSS methods yielded similar concentrations for snowmelt runoff and undisturbed bulk snow containing particles < 500 µm, except in the case of snowmelt runoff 2, where TSS yielded significantly lower concentrations. In the earlier studies by Gray *et al.* (2000), Clark *et al.* (2008) and Nordqvist *et al.* (2010), the TSS method produced noticeably lower concentrations compared to the SSC-B method for samples containing large particles. However, the spread between the min and max values for sub-samples in this study was higher for the TSS method than for the SSC-B and MFM methods, implying large measurement uncertainties for this method.

When comparing particle concentrations for snowmelt runoff and rainfall runoff, it becomes apparent that snowmelt runoff contained solids concentrations 10-90 times higher than those in rainfall runoff. An earlier study conducted by Westerlund (2007) also showed that the highest TSS concentrations were found for snowmelt events. The results showed eight times higher concentrations and five times more particles (4-120 µm) during snowmelt than in rainfall runoff events. This could be explained by the larger amount of available material (solids) accumulated on the catchment surface during the winter period. Furthermore, it appears that, in the study area, snowmelt transports larger particles than rainfall runoff events, and consequently, the measurement of concentrations of both small and large particles is of great importance under such circumstances.

In summary, the results presented show the importance of choosing the right analytical method for determining the mass of solids in both rainfall and snowmelt runoff. Depending on the study objectives and the sampled water characteristics, choosing the correct analytical method is crucial. From the point of view of drainage operation and management, the TSS method will underestimate the total mass of solids transported and possibly deposited in catch basins, sewer pipes, and stormwater management ponds. The settleable solids, which are not captured by the TSS method, can settle in sewer pipes, stormwater ponds and on the bottom of receiving water bodies and hinder drainage system operation and contribute to pollutant accumulations in receiving waters. However for assessing environmental impacts of drainage effluents, including increased turbidity, reduced sunlight penetration, transport of potentially harmful chemicals (heavy metals, toxic trace organics and nutrients), and the risk of acute toxicity, it is important to focus on and measure fine solids (TSS). The analysis presented in this study showed the importance of using the right analytical method to obtain the results meeting the study objectives.

CONCLUSIONS

A study of solids in urban rainfall-induced runoff and in snowmelt showed that rain runoff contained higher concentrations of small particles, and under such conditions, the MFM method produced significantly higher solids concentrations compared to those produced by the SSC-B and TSS methods, at a 95% confidence level. This finding was explained by the influence of small particles (0.45-1.6 µm) captured only by the MFM methods, but not by the SSC-B and TSS methods, with the two latter methods producing comparable results without any statistically significant differences. All the three methods, SSC-B, MFM and TSS, yielded similar results for measuring solids in snowmelt runoff and in undisturbed bulk snow. However, the TSS method concentrations were characterized by a considerably higher CV in the case of snowmelt runoff, implying large measurement uncertainties of the method in this particular application.

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