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Sedimentation and time-of-transition techniques for measuring grain-size distributions in lagoonal flats: comparability of results

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ABSTRACT

A comparative study was performed of three instruments used to measure the grain-size distribution of thirty sediment samples from shallow lagoonal flats: the hydrometer, the Sedigraph 5000 and the CIS-1. The hydrometer and Sedigraph are based on sedimentation whilst the CIS-1 uses time of transition. The percentage of the samples accounted for by the <8 µm fraction were not affected by the technique used, but this was not the case with the clay fraction (<2 µm). Due to its relative independence from the analytical method applied, the <8 µm fraction can be used in ternary diagram classifications. This fraction also has an environmental significance in coastal lagoons in terms of hydrodynamics, organic enrichment and macrozoobenthos assemblages. The linear relationships obtained in this study may provide useful operational indications for similar studies.

Short running title: Sedimentation, time of transition: comparability

Keywords: Grain-size analysis, sortable silt, non-sortable silt, mud, time of transition

INTRODUCTION

Although common standards of analysis have not yet been established by the scientific community, grain-size and grain-size distribution are key factors in sedimentology and landscape evolution (Goossens, 2008). Sediment grain-size data are commonly used for textural classification, but the details remain unresolved for lagoon sediments. Lagoons may display a large variety of sediment patterns depending on the relative strength of waves and tides (Nichols and Boon, 1994). Grain-size distribution is certainly one of the more helpful tools for describing the environmental conditions in lagoon systems, because fine-grained material correlates strongly with pollutants. In addition, variability in chemical, physical and hydrographical parameters is always related to variation in sediment grain-size (Kjerfve, 1994). Particle dimensions therefore describe environmental conditions and provide information about processes acting on the ecosystem. They may thus be regarded as an "environmental tracer". For these reasons, it is very important to characterise the grain-size of bottom sediments. Indeed, grain-size data is essential for the modelling and management of lagoon environments.

Flemming (2000) proposed an updated version of the texture ternary diagram (Reineck and Siefert, 1980; Pejrup, 1988), which increases the range of application and the environmental sensitivity of textural sediment classification. The classification incorporates a genetic element by distinguishing between different hydrodynamic regimes. While Molinaroli *et al.* (2009a, b) showed that the <8 µm and approximately 20 µm fractions could be used to classify lagoonal sediments in terms of their hydrodynamics, Chang *et al.* (2006, 2007) showed that 8 µm is an important size-limit in the Wadden Sea sediments, delimiting the transition between cohesive flocs and aggregates and non-cohesive single grains. The debate among sedimentologists about the importance of this limit is ongoing. Moreover, the <8 µm fraction was found to be correlated with the total organic carbon and organic matter content of sediments in the Lagoon of Cabras (De Falco *et al.,* 2004; Magni *et al.,* 2008). In this homogeneously muddy system, impaired benthic assemblages were found in sediment characterised by 77% ≤8 µm, 11% OM and 3.5% TOC (Magni *et al.,* 2008).

The use of specific grain-size intervals for sediment classification, such as <2 µm (hereafter PM2) and <8 µm (hereafter PM8), requires that results derived from different analytical methods are broadly comparable. The problem of comparing grain-size analyses based on different techniques and physical principles has been discussed by several authors (Konert and Vanderbergen, 1997; McCave *et al.*, 2006; Goossens, 2008) and because of the differences in results, many comparative studies of grain-size techniques have been carried out over the last two decades (Syvitski *et al*., 1991; Shillabeer *et al.,* 1992; Duck, 1994; Bergen & Sukuda, 1995; Cramp *et al.*, 1997; Konert &

Vandenberghe, 1997; Beuselinck *et al.*, 1998; Bianchi *et al.*, 1999; Molinaroli *et al.*, 2000; McCave *et al.*, 2006; Goossens, 2008).

For the same silty-clay sediment, laser techniques (diffraction and time-of-transition methods) tend to yield coarser grain size estimates than sedimentation techniques. Konert & Vandenberg (1997) found that the PM8 fraction measured by laser diffraction corresponded to the PM2 fraction measured by sedimentation for sediments of fluvial, aeolian and lacustrine origin. McCave *et al.* (2006) showed that the laser diffraction method increasingly overestimated the 10-63 µm fraction as the fine silt/clay content, measured by sedimentation using a Sedigraph, increased. However, they found that the differences between grain size distributions derived by laser diffraction and Sedigraph become negligible when the 10-63 µm fraction was higher than 40%. Molinaroli *et al.* (2000) found a correspondence between PM4 (<4 µm) measured by laser (time-of-transition) and PM2 measured by Sedigraph, with the differences found using the time-of-transition laser technique (Galai CIS-1) less accentuated than those found using laser diffraction (Malvern Mastersizer). Recently Goossens (2008) published a detailed comparative study analysing four sediments with ten techniques. Although the trends were generally similar, he observed differences in the results of grain size analyses conducted with different instruments. It follows from these observations that the classification of sediments in terms of sand, silt and clay ratios depends on the type of instrument used for grain size analysis (Goossens, 2008).

The aim of this study was to compare grain size data from samples collected across lagoonal flats to ascertain whether analyses performed with instruments based on laser (time-of-transition) and sedimentation techniques produced comparable results, especially for those fractions considered to be most useful in environmental sedimentology.

METHODS

Techniques and instruments

Two sedimentation methods (hydrometer and Sedigraph) and one time-of-transition laser method (CIS-1) were used. The hydrometer method (Lesikar et al., 1995) relies on the differential settling velocity of sediment grains of different sizes in a fluid with known viscosity and constant temperature. Sediment particles are dispersed with a substance such as sodium metaphosphate and then agitated. As grains of different size settle at different rates, so the specific density of the sediment-fluid mixture, measured with a hydrometer, changes and Stoke’s law can be used to calculate the grain-size distribution. The Sedigraph 5100 (Micromeritics Instrument Corporation, Norcross, GA, USA) measures the sedimentation rate by determining X-ray obscuration at different levels in a sample cell. The grain-size data are given as mass percentages. The CIS-1 (Galai Production Ltd, now owned by Ankersmid B.V., Oosterhout, the Netherlands) is based on the detection of particles by a rotating laser beam and a photodiode. The data are given as volume percentages. In this study, we employed small-volume cuvettes with magnetic stirring to suspend particles (see Molinaroli *et al.*, 2000 for analytical procedures). Four analytical replicates were carried out for each sample and each instrument, the mean grain diameter variability between replicates being <2%.

Sediment characteristics

Thirty sediment samples collected from shallow lagoonal flats in the Lagoon of Venice (LV) were analysed. Sediment samples were pre-treated with H_2O_2 (20% volume) to eliminate organic material, washed with bi-distilled water to eliminate chlorides, and then oven-dried at 40°C for 12h they subsequently were wet sieved in order to eliminate the sandy fraction (>63 µm). Finally samples were pre-treated with 6‰ Na-hexametaphosphate solution for 24 h and then sonnicated for 5 min before analysis. Sediment textures ranged between silty clay and clayey silt. The composition of the 30 sediment samples in terms of seven grain-size fractions (32-63; 16-32; 8-16; 4-8; 2-4; 1-2; <1 µm) was determined.

There are substantial differences between the sediment samples used in this study and the samples used by Goossens (2008). Unlike Goossens’ samples, lagoonal sediments generally contain significant amounts of organic matter (LV average approximately 5%) (Frangipane et al., 2009), which must be removed in order to avoid the formation of aggregate particles. There are also some differences in analytical procedures. Goossens analysed the bulk sediment without previous sieving at 63 µm. In this study the focus was on the fraction that passed through the 63 µm sieve.

RESULTS

Comparison of the three techniques

For each sediment class there were differences in the proportions determined by each method. The mean differences (± standard deviation) for each size class percentage were computed for each pair of instruments (Figure 1). Comparisons of the differences in frequency distributions as determined by hydrometer, Sedigraph and CIS-1 showed that the dissimilarities among the instruments were mainly in the 32-63 µm, 16-32 µm and <1 µm size intervals.

Measured percentages of the <1 µm grain size fraction were, on average, higher when measured with the sedimentation techniques rather than the laser method. The devices have different lower limits for grain-size detection, with the CIS-1 measuring particles down to a limit of 0.5 µm but the sedimentation techniques measuring finer particles than that. Differences were also found between the two sedimentation techniques: the Sedigraph yielded higher percentages in the <1 µm class (approximately 8% more) than the hydrometer. Comparison of coarser classes shows that the CIS-1 yielded lower values for the 32-63 µm fraction than the hydrometer (approximately 20% less) and Sedigraph (approximately 7% less) (Figure 1). The hydrometer considerably overestimated the coarse-silt fraction (32-63 µm) by approximately 14%, compared to the Sedigraph. The opposite trend was found for the 16-32 µm class. The CIS-1 values were approximately 16% higher than those by the hydrometer method and approximately 6% higher than those given by the Sedigraph. The values given by the Sedigraph were approximately 9% higher than those given by the hydrometer. In general, the 32 µm boundary is a critical point, beyond which the instruments yield the most widely varying results.

PM2 and PM8 fractions

The PM2 and PM8 are descriptors for the transition from cohesive flocs and aggregates to non-cohesive single mineral grains. Two-way comparison plots (based on the entire data set of 30 samples) for PM2 and PM8 as measured with the three devices are shown in Figures 2 and 3 respectively. Since there is uncertainty in both the dependent and independent variables, a type-II regression model was adopted. which minimises the perpendicular distance between the data points and the model line.

With the PM2 data, the correspondence was poor. In contrast, good relationships were found for the PM8 fraction, allowing PM8 data to be transformed from values measured using one instrument to equivalent estimates as if measured by another instrument. The linear relationships between the data obtained by Sedigraph, CIS-1 and hydrometer for the PM8 fractions were:

% CIS-1 = 0.74 • % hydrometer + 14.73 ($r=0.90$; $p<0.001$)

% Sedigraph = 1.09 • % hydrometer + 0.18 ($r=0.94$; $p<0.001$)

% CIS-1 = 0.63 • % Sedigraph + 17.0 ($r=0.89$; $p<0.001$)

DISCUSSION

The comparison of grain size data from the different instruments used in this study shows that the biggest differences are in estimation of the clay fraction (PM2), thus confirming for lagoonal sediments, the findings of previous investigations for sediments from other environments (McCave *et al.*, 2006; Beuselinck *et al.*, 1998; Konert and Vandenberghe, 1997). As here, Beuselinck *et al.* (1998) and Konert and Vandenberghe (1997) showed that the PM2 estimates obtained from laser diffraction were generally less than those measured using sedimentation techniques. This discrepancy was attributed to the effects of clay mineralogy (Beuselinck *et al.*, 1998). Our results showed that the analysis of PM2 in lagoonal samples using different instruments did not produce comparable results, thus confirming that the problem with comparing grain size data obtained with different devices seems to be mainly related to the finest fractions. In contrast, the analysis of PM8 in the lagoonal sediments indicated good relationships between the three instruments.

The proportions of PM2 and PM8 as detected with the three techniques were compared with the results obtained by Goossens (2008). The Atterberg cylinder method (tested by Goossens) and hydrometer techniques are based on the same principle, so we consider the two techniques to be similar, just as the CIS-100 tested by Goossens is considered to be similar to the CIS-1. Goossens (2008) compared four sediment samples (G-A, G-B, G-C, G-D) characterised by decreasing median grain diameter (35 µm, 30 µm, 12 µm, 9 µm respectively) and increasing clay content (3%, 8%, 10%, and 15%). In order to compare our results with those of Goossens (see table 1 and Fig. 8 in Goossens, 2008), we grouped our samples into four classes with increasing PM2 and PM8 content, using the mean percentage values (±SD) of each class for comparison (Figure 4).

Examination of Goossen’s data shows (Fig. 4A, B) that the CIS-100 underestimated the clay fraction (PM2) by between 1 and 10% compared to the Atterberg technique. In contrast, the CIS-100 overestimated the fine silt fraction (2-8 µm), especially in the silty samples (G-C and G-D), with values that were 10-20% higher on average than the Atterberg values. The PM2 fraction determined by Sedigraph was overestimated by 2-14% and 3-22% with respect to the Atterberg and CIS-100 respectively. The Sedigraph overestimated the PM8 fraction by 8-17% with respect to the Atterberg, while the Sedigraph data differed by between -13% and 16% with respect to the CIS-100, depending on the silt content.

For the data from this study (Fig. 4C, D), there was poor correspondence between the Sedigraph, CIS-1 and hydrometer for the PM2 fraction (Fig. 4C), while good correspondence was found for PM8 (Fig. 4D). The PM8 fraction measured by Sedigraph was overestimated with respect to the hydrometer by just 1-5%. In contrast, the Sedigraph data differ by between -5% and 8% with respect to the CIS-100 depending on the silt content. The results of this study are therefore in agreement with Goossens, according to whom there is no optimum technique for measuring the grain-size distribution of loamy sediments. The choice of technique thus depends on several factors such as type and quantity of sediment, speed of measurement, complexity of the measurement protocol, data processing and reproducibility of the results.

The comparability of PM8 data obtained by the sedimentation and CIS-1 methods suggests that the latter can be used for the determination of the sortable/non-sortable ratio (*sensu* McCave *et al.*, 2006), which is considered a proxy for palaeo-current speed. Whereas laser diffraction systems tend to overestimate the sortable/non-sortable ratio, especially for sediments with high clay content (McCave *et al.*, 2006), the CIS-1 laser system, based on time transition, does not.

The PM2 and PM8 intervals have specific sedimentological significance. The 2 µm boundary is used for sediment classification (clay/silt limit) in ternary diagrams (Flemming, 2000). The PM8 fraction is known as the non-sortable silt fraction, consisting of both single particles and an aggregated or flocculated fraction, whereas the 8–63 µm fraction consists of sortable (non-aggregated) silt particles (McCave *et al.*, 1995; Chang *et al.* 2006, 2007; Molinaroli *et al.*, 2009a, b). Furthermore, the results for PM2 and PM8 indicate that the appropriateness of a textural classification based on the sand/silt/clay ratio with boundaries at 63 µm and 2 µm (Flemming, 2000) depends on the instruments used for the analysis. In contrast, the <8 µm fraction is comparable across instrument readings and our analysis suggests that a simple linear relationship can be used to convert one data set to another. In this case, ternary diagrams might more usefully utilise < 8 µm as a classification boundary.

CONCLUSIONS

This study compared grain size data for 30 sediment samples taken from shallow lagoonal flats obtained with instruments based on laser (time-of-transition) and sedimentation techniques. The main results are:

1. The size of the PM8 (<8 µm) fraction was comparable regardless of the three techniques used, while this was not the case for the clay fraction (PM2= <2 µm).
2. The PM8 grain size data obtained from the three devices may be converted into a comparable form by using simple linear relationships.
3. The <8 µm fraction is suitable for ternary diagram classification due to its relative independence from the analytical method used. This limit has also an environmental significance in coastal lagoons in terms of hydrodynamics, organic enrichment and macrozoobenthos assemblages.
4. The linear relationships obtained in this study may provide useful operational indications for similar studies in coastal lagoons.

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FIGURE CAPTIONS

Fig. 1. Mean differences (n = 30 samples) between the frequencies of seven clay – silt grain size fractions measured using two sedimentation methods (hydrometer and Sedigraph) and a laser time-of-transition method (CIS-1).. Standard deviations are indicated by bars.

Fig. 2. Two-way comparison plots, based on the entire data set of 30 samples for the PM2 fraction as measured with the three devices. The equation y=x shown in the graph represents the theoretically perfect reproducibility of analyses using different methods. Given the poor correspondence between methods, the relative models were not calculated.

Fig. 3 Two-way comparison plots, based on the entire data set of 30 samples for the PM8 fraction as measured with the three devices. The equation y=x shown in the graph represents the theoretically perfect reproducibility of analyses using different methods. The good correspondence between methods enabled models to be calculated in all three cases (see text for the relative equations).

Fig. 4. Comparison of results from Goossens (2008) (A, B) measured by the three techniques and the results of the present study (C, D) for the PM2 and PM8 grain-size fractions. G-A, G-B, G-C and G-D = the four samples analysed by Goossens; 1, 2, 3, 4 = the groups of samples in this study (The number of samples for each group are 7, 9, 7 and 7, respectively). Standard deviations are indicated by bars.

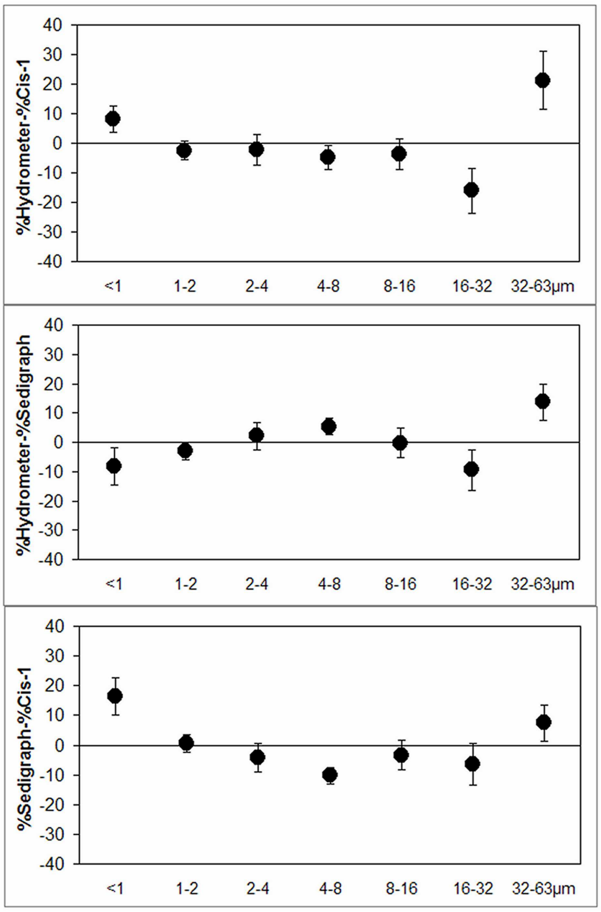
%Hydrometer-%Cis-1
%Hydrometer-%Sedigraph
%Sedigraph-%Cis-1
40
30
20
10
0
-10
-20
-30
-40
<1
1-2
2-4
4-8
8-16
16-32
32-63µm

FIG.1

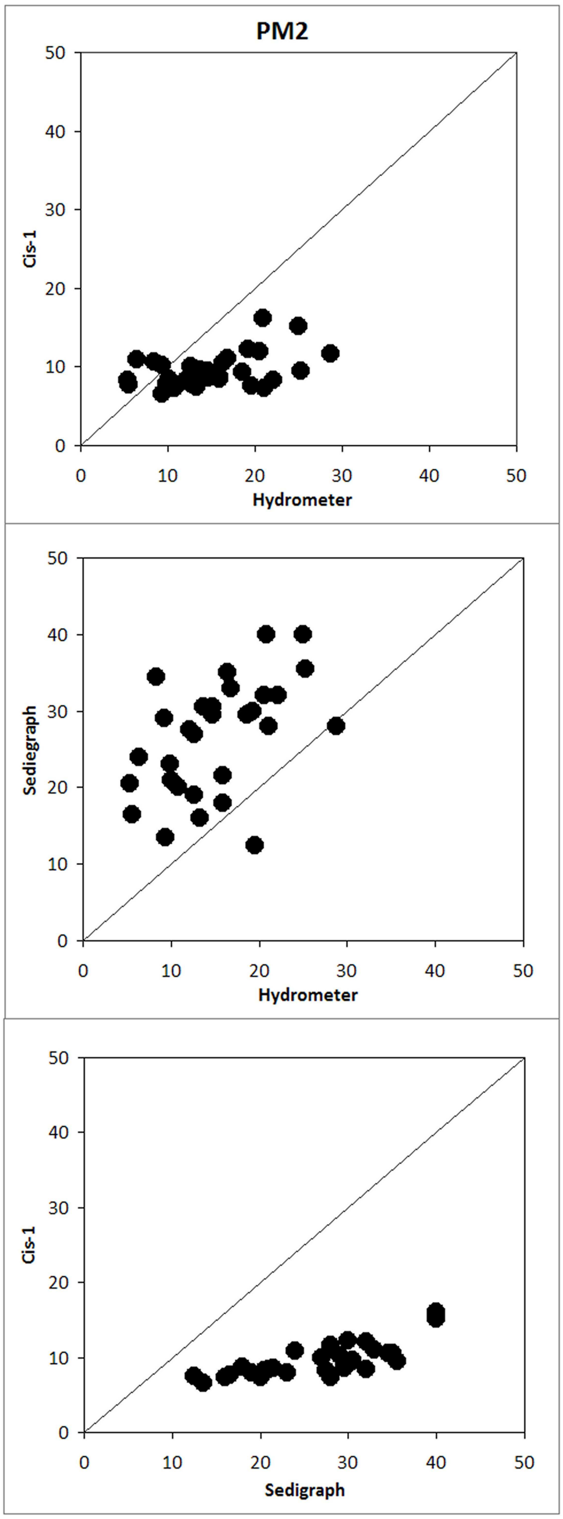
PM2
Cis-1
50
40
30
20
10
0
0
10
20
30
40
50
Hydrometer
Sediegraph
Hydrometer
Cis-1
Sedigraph

FIG.2

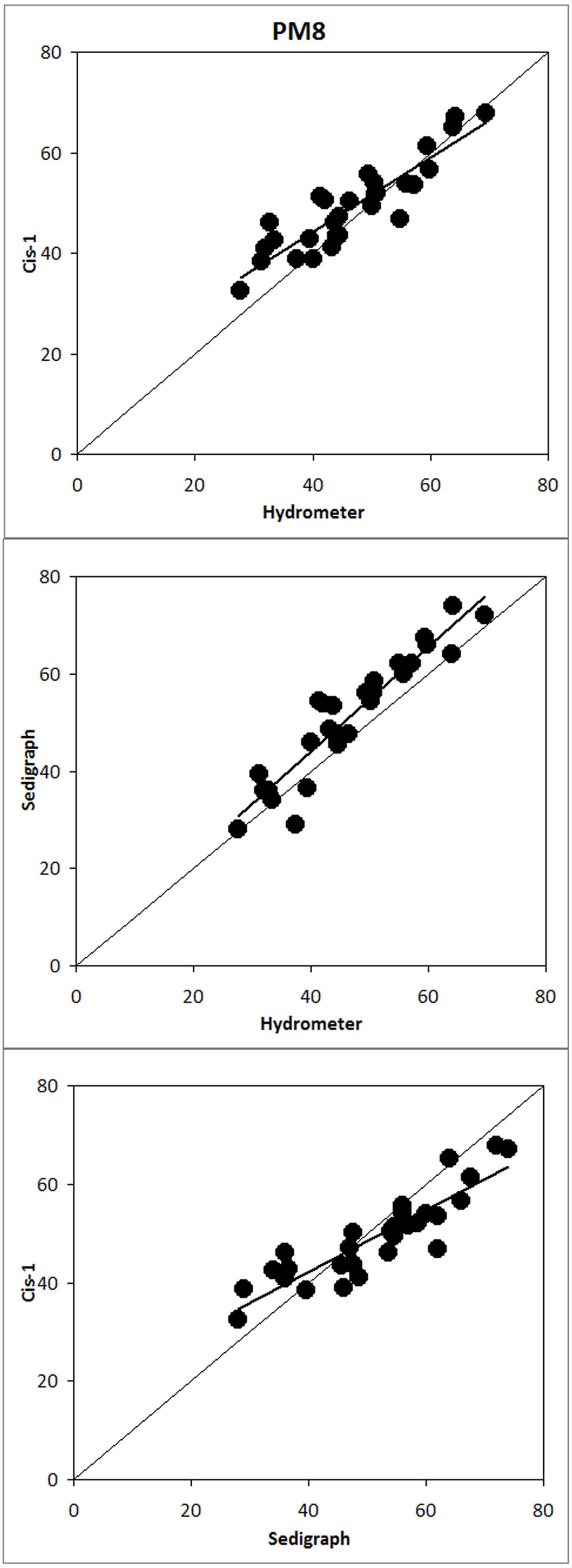
PM8
Cis-1
Hydrometer
Sedigraph
Hydrometer
Cis-1
Sedigraph
0
20
40
60
80

FIG. 3

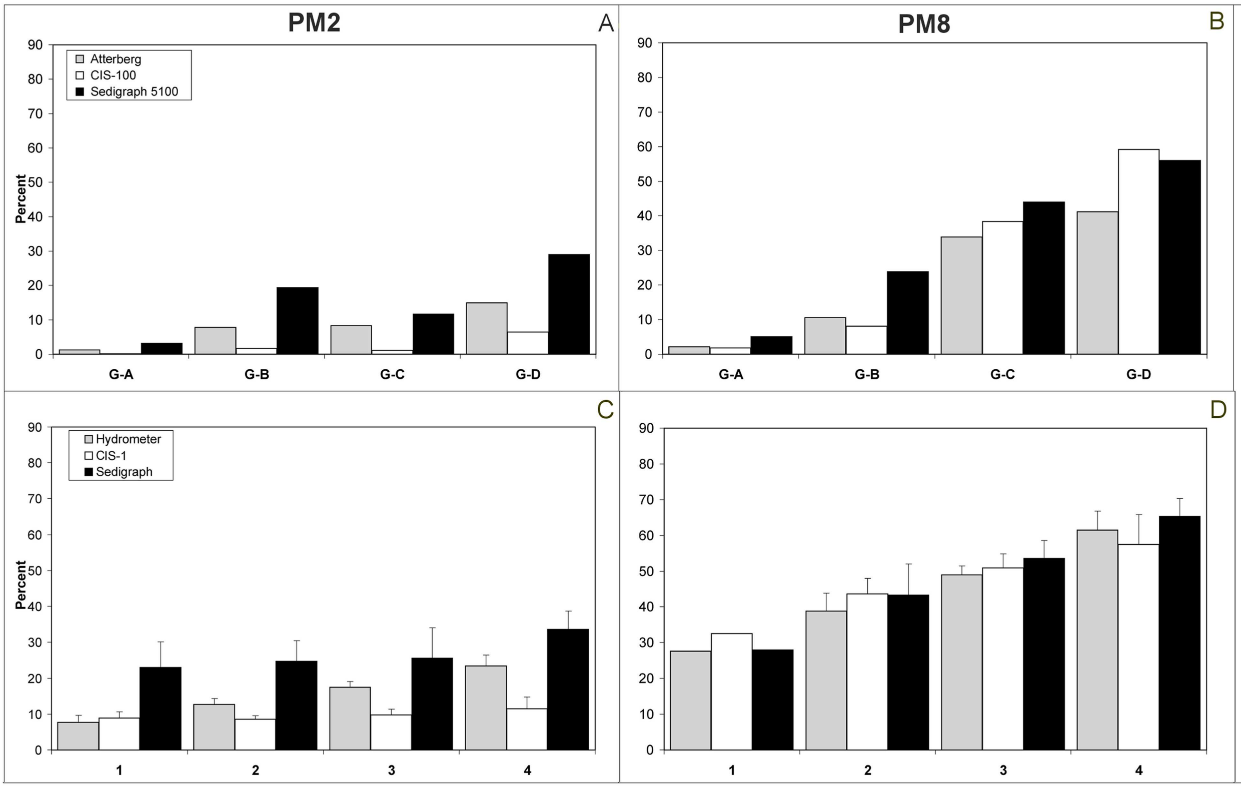
PM2
A
Atterberg
CIS-100
Sedigraph 5100
Percent
90
80
70
60
50
40
30
20
10
0
G-A
G-B
G-C
G-D
PM8
B
C
Hydrometer
CIS-1
Sedigraph
1
2
3
4
D

FIG. 4