Measuring suspended mud flocs in the laboratory: a comparison between two methods

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ABSTRACT: Turbid rivers and density currents carry, distribute, and deposit considerable quantities of fine muddy sediment within rivers, coastal regions, and reservoirs. The muddy sediment in these flows has the potential to flocculate, and knowing and predicting the floc size is critical for predicting mud movement. Flocs are notoriously difficult to measure. Imaging of flocs either within a turbulent suspension or in a separate settling chamber are methods widely considered to be the most accurate ways to measure floc size. The benefit of imaging flocs within the suspension is that the measurements are made within the conditions that gave rise to those particular flocs. The drawback is that it is not possible to make measurements in suspensions with concentrations $>$400 mg/L. Transferring a suspension sample to a settling chamber allows for imaging of flocs from suspensions with higher concentration. But, it also removes flocs from the environment in which they were formed, possibly leading to floc growth or breakup. In this study, we compare these two methods to determine whether or not the flocs imaged in a settling chamber are representative of the flocs found in a turbulent suspension. For the experiments, flocs are formed from kaolinite and montmorillonite clay mixed with saltwater at different concentrations and mixing conditions. The suspension is then imaged within the mixing tank, and samples from the mixing tank are imaged in a settling chamber. Results show that flocs imaged in the settling chamber tend to be slightly smaller than those imaged in the mixing chamber, though the differences are minimal if care is taken in the transfer process. Additional trends in the difference between the two methods with turbulent shear rate and concentration are discussed.

1 INTRODUCTION

Transport and deposition of the fine muddy sediment present in marine and riverine environments is strongly modulated by flocculation. Flocculation is the aggregation and breakup of these muddy sediments that yields flocs with size, shape, and density different than its original constituent particles. The process of flocculation is affected by variables such as the sediment type and concentration, salinity, and flow condition. Determining the size of the flocs is critical as it is the major factor influencing the movement and behavior of flocs in different flow conditions.

Flocs are difficult to measure due to their small size, dependence on the environment that they form in, and inherent fragility. Various experimental methods have been used to measure and characterize flocs. A method that has been extensively used in the literature is to transfer sediment suspensions to a settling column using a syringe (e.g., Gratiot and Manning, 2004), hand pipette (e.g., Manning and Dyer, 1999, Mooneyham and Strom 2018), or peristaltic pump (e.g., Jarvis et al., 2005) and obtain images from the suspension within the settling column. However, these sample delivery methods can be destructive to floc structure and yield inaccurate measurements. Imaging flocs in their own turbulent environment in which they have formed and evolved can be a relatively more accurate approach for floc size measurements. Therefore, to be as least intrusive to flocs as possible in the imaging process, efforts have been made to utilize methods that allow for capturing floc images *in situ* (e.g., Smith and Friedrichs, 2011; Keyvani and Strom, 2014; MacDonald and Mullarney, 2015; Tran and Strom, 2017; Strom and Abolfazli, 2019). A major drawback in this method is that as the suspended sediment concentration (SSC) increases, it becomes more difficult to process floc images due to the extensive overlap of flocs in the obtained images (Tran et al., 2018), and the measurements become less reliable. This may necessitate transfer and dilution of the suspension sample into a second environment, where the imaging and measurement takes place. However, it is not clear how consistent the measurements from these two methods are. The study we present in this paper is a comparison between the floc sizes obtained from *in situ* and transferred measurement methods under different suspension concentrations and shear rates. For this purpose, a series of four experiments were conducted to compare floc sizes measured using these two different methods. The remainder of this paper is organized as follows. Chapter 2 describes sample preparation procedure and the floc imaging and processing methods. Chapter 3 presents the results. Chapter 4 is discussion and conclusion.

2 MATERIALS AND METHODS

The experiments we conducted consisted of the use of two floc measurement methods. The first method was implemented in a 13 L mixing tank with dimensions of 27.5×27.5×25 cm. A paddle mixer with variable speed was used to create turbulent shear in the tank. Salt was added to the background water to achieve the salinity of 8 ppt. A mixture of 80% kaolinite and 20% montmorillonite (by dry mass) was used to mimic natural mud. Before adding the sediment to the clear water in the mixing tank, it was well mixed in a beaker and then sonicated for 15 minutes to break down any potential flocs. Once the sonicated sediment mixture was added to the tank, imaging of the suspension was commenced at a frequency of 1 Hz.

In the second method, 3 mL samples from the mixing tank suspension were collected with a pipette and transferred to a 80 mL settling chamber. The settling chamber contained water with a salinity that matched that of the mixing tank. Every effort was made to transfer the suspension to the settling chamber in as little time as possible while introducing the least possible perturbation to the floc mixture. Sample delivery from the mixing tank to the settling chamber commenced 12 hours after the initiation of the mixing tank experiment to make sure that flocs in the tank were no longer changing size with time. Multiple samples were transferred to the settling chamber during each experiment. The still image capture rate in the settling chamber was also set to 1 Hz. The experiments were conducted at two different sediment concentrations (C) (i.e., $C = 100$ and 300 mg/L) and two different shear rates (G) (i.e., $G = 50$ and 95 1/s), resulting in four different experimental conditions (Table 1). Three replicates were performed for each condition. The exception to this rule was Experiment 2, in which six replicates where conducted. The camera systems used for the mixing tank and the settling chamber both provided a length to pixel ratio of 1.3 μm:1 pixel, allowing for measurement of flocs in the size range of 10-1000 μm. More details about the camera system can be found in Tran and Strom (2017). Floc images obtained from the mixing tank and the settling chamber were then processed using the procedures of Keyvani and Strom (2013) to obtain floc size distribution. Figure 1 shows a schematic of the experimental setup.

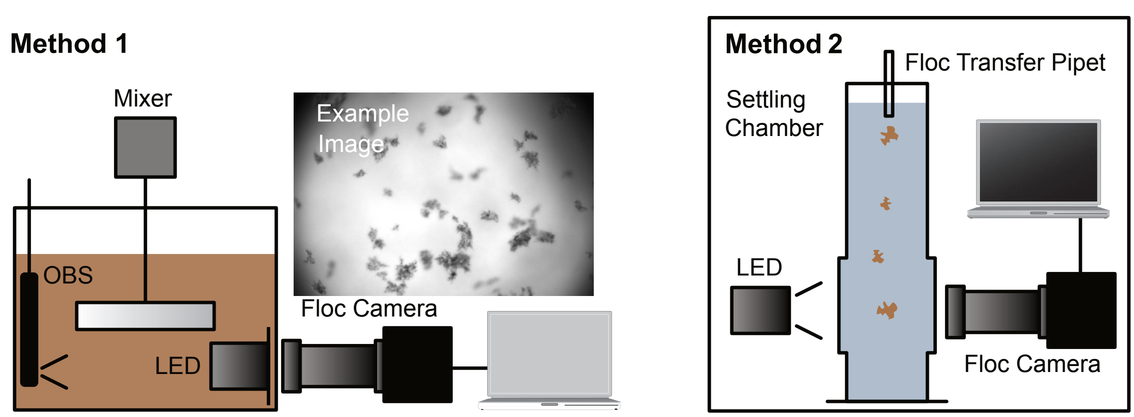

Figure 1. Experimental setup

Table 1. Description of the experiments.

Experiment	Sediment Concentration (mg/L)	Shear Rate (1/s)	Number of Replicates
1	100	50	3
2	300	50	6
3	100	95	3
4	300	95	3

3 RESULTS

Continuous collection of floc images enabled us to investigate how floc sizes evolve within the mixing tank and to produce a time series of different size distribution statistics such as the floc d_{50} (Fig. 2a). There is an evident peak in floc size around minute 5 followed by a decline down to equilibrium. The floc size reached equilibrium after about 12 hr, which is when the suspension samples were transferred from the mixing tank to the settling chamber. Figure 2b shows the floc size measured in the settling chamber in Experiment 1. In this experiment, floc size in the settling chamber was slightly smaller than those from the mixing tank (Table 2). The percentage difference between the the d_{50} measured using the two methods is

$$\Delta d_{50} = \left(\frac{d_{50mix} - d_{50set}}{d_{50mix}} \right) \times 100 \tag{1}$$

where d_{50mix} and d_{50set} are d_{50} measured in the mixing tank and settling chamber, respectively. Δd_{50} in Experiment 1 ranged between 1.0 to 3.7 in different replicates with an average of about 3, which means that on average, d_{50} was $\approx 3\%$ smaller in the settling chamber when compared to the mixing tank. Positive values for Δd_{50} in all the replicates shows smaller d_{50} in the settling chamber.

Table 2. d_{50} measured in Experiment 1 ($C = 100$ mg/L and $G = 50$ 1/s).

Replicate	Mixing Tank d_{50mix} [μm]	Settling Chamber d_{50set} [μm]	d_{50mix}-d_{50set} [μm]	Δd_{50} [%]
1	103	102	1	1.0
2	114	110	4	3.5
3	106	102	4	3.7

Experiment 2 had a larger sediment concentration compared to Experiment 1 at the same shear rate. Increasing the concentration of the sediments from 100 mg/L to 300 mg/L increased the average equilibrium d_{50} from 108 μm (Table 2) to 124 μm (Table 3). Similar to Experiment 1 ($C = 100$ mg/L and $G = 50$ 1/s), floc size measured in the settling chamber were generally consistent among the replicates. Sizes ranged from 112 to 138 μm in the mixing tank and from 104 and 120 μm in the settling chamber (Table 3). On average, d_{50} measured in the settling chamber was about 12% smaller than that measured in the mixing tank.

An increase in shear rate from $G = 50$ to 95 1/s led to a reduction in the equilibrium floc size in both the $C = 100$ mg/L and 300 mg/L experiments due to increased floc breakup (Experiments 3 and 4 compared to Experiments 1 and 2). The average equilibrium d_{50} across the replicates decreased from 108 μm (Table 2) to 73 μm (Table 4) at 100 mg/L and from 124 μm (Table 3) to 82 μm (Table 5) at 300 mg/L when the shear rate in the mixing chamber was increased from 50 1/s to 95 1/s. At the shear rate of 95 1/s, the highest difference between d_{50} measured in the mixing tank and settling chamber was at the concentration of 100 mg/L (Table 4), where the average measured d_{50} was about 5% less than that in the mixing tank. The average measured d_{50}

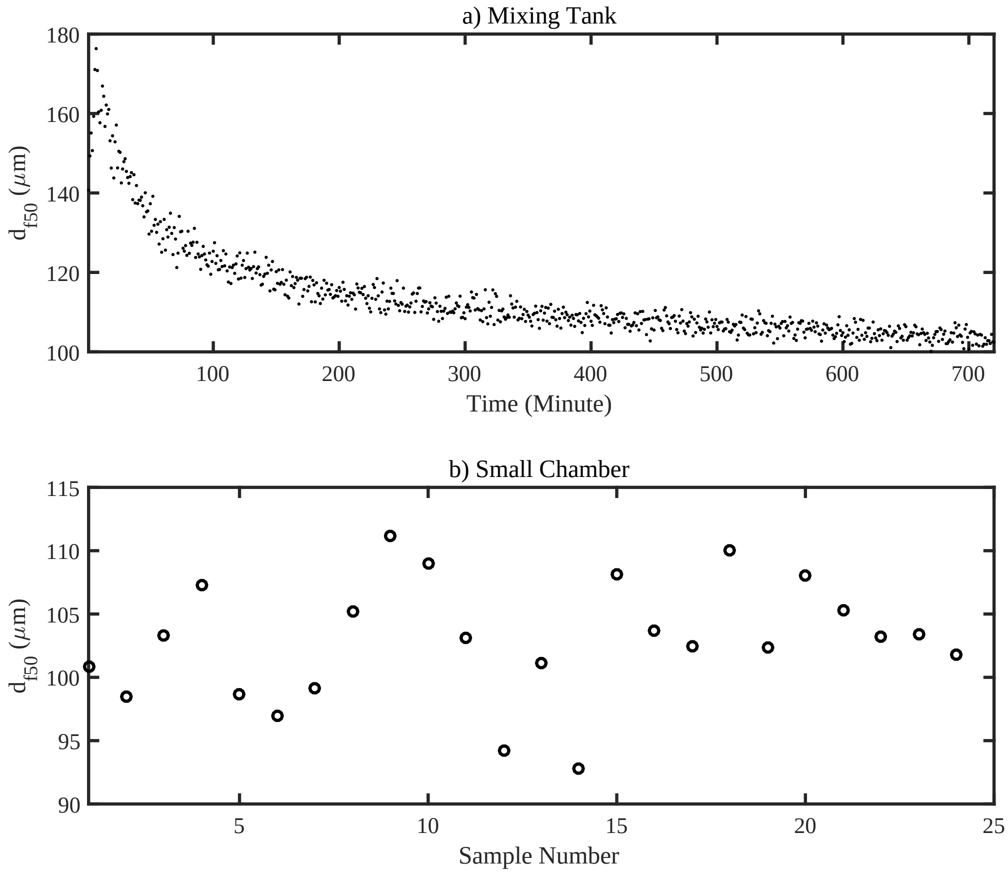

Figure 2. Time series of d_{50} from Experiment 1 as measured in a) the mixing tank (method 1) and b) the settling chamber (method 2).

Table 3. d_{50} measured in Experiment 2 ($C = 300$ mg/L and $G = 50$ 1/s).

Replicate	Mixing Tank d_{50mix} [μm]	Settling Chamber d_{50set} [μm]	d_{50mix}-d_{50set} [μm]	Δd_{50} [%]
1	122	105	17	13.9
2	119	113	6	13.4
3	132	116	16	12.1
4	121	105	16	13.2
5	112	104	8	7.1
6	138	120	18	13.0

at the concentration of 300 mg/L was interestingly the same as that measured in mixing chamber (Table 5), the differences did exist for individual replicates.

Table 4. d_{50} measured in Experiment 3 ($C = 100$ mg/L and $G = 95$ 1/s).

Replicate	Mixing Tank d_{50mix} [μm]	Settling Chamber d_{50set} [μm]	d_{50mix}-d_{50set} [μm]	Δd_{50} [%]
1	76	77	-1	-1.3
2	69	66	3	4.3
3	74	66	8	10.8

Table 5. d_{50} measured in Experiment 4 ($C = 300$ mg/L and $G = 95$ 1/s).

Replicate	Mixing Tank d_{50mix} [μm]	Settling Chamber d_{50set} [μm]	d_{50mix}-d_{50set} [μm]	Δd_{50} [%]
1	79	80	-1	-1.2
2	80	87	-7	-8.7
3	88	80	8	9.1

4 DISCUSSION AND CONCLUSION

We conducted four experiments with multiple replicates to compare the d_{50} measurements in the larger mixing tank and the smaller settling chamber. The d_{50} averaged over the replicates was reasonably consistent between the two methods in each experiment. Results generally indicate that transferring the sediment suspension from the mixing tank to the settling chamber results in smaller d_{50}, likely due to the breakup of some flocs. If we consider the average of the replicates as an indication of how the two measurement methods compare, Experiment 4 with the suspended sediment concentration of 300 mg/L and shear rate of 95 1/s yielded the best agreement (Table 5). This could possibly be due to higher floc aggregation rate in the settling chamber, allowing flocs that had broken up some in the transfer process to grow. In this experiment, the relatively high floc concentration facilitates floc collisions that results in the aggregation of flocs. This is obvious in the first and second replicates of the experiment in which the d_{50} measured in the settling chamber were in fact greater than that measured in the mixing tank. The third replicate, however, indicates that d_{50} was smaller in the settling chamber. The variability among the replicates was consistent between mixing tank and settling chamber (9 and 7 μm, respectively). Experiment 1 showed the next best consistency between the floc sizes measured using the two methods with the average d_{50} in the settling chamber being only about 3% smaller than that in the mixing tank (Table 2) with 11 and 8 μm variability within replicates in mixing tank and settling chamber, respectively. This suggests that the flocs were not large enough to undergo substantial breakup when they were being transferred. This conclusion is somewhat consistent with the results of Experiment 2. This experiment had the largest equilibrium d_{50} in the mixing tank and showed the greatest difference between the measurements conducted in the two chambers (Table 3). In this experiment, the d_{50} measured in the settling chamber was in average 12% smaller than that in the mixing tank, suggesting a more significant breakup rate when the relatively large flocs were being transferred. The greatest variability among replicates was also in this experiment (26 and 16 μm in mixing tank and settling chamber, respectively). In Experiment 3, due to the relatively low sediment concentration and high shear rate, flocs had the smallest equilibrium d_{50} (Table 4) and yet they underwent some degree of breakup with no significant aggregation in the settling chamber. In this experiment, d_{50} measured in the settling chamber was about 5% smaller than that in the mixing tank. Small floc size was in line with the small variability among replicates (7 and 11 μm in mixing tank and settling chamber, respectively).

Although the d_{50} measured in the settling chamber was generally smaller than that in the mixing tank, it does not mean that the transfer methods cannot be used for floc size quantification. The maximum average difference in floc size between the two methods in the experiments was about 12%. This suggests that in cases that floc suspension cannot be imaged within the mixing tank due to high sediment concentration, transferring floc samples to a second environment can be considered a viable option. However, such measurements might be made more accurate by adding in a correction factor that depends on the sediment concentration and flow properties. It also should be noted that we initially observed significant differences between the measurements obtained from the two methods. That is, it was only after refining the sample delivery method and the image processing procedure that the floc size measurements in the settling chamber were close to those in the mixing tank. For the sample delivery procedure, great care was taken not to disturb the flocs. For the image processing procedure, once the images were obtained from the dilution chamber, they were manually inspected, and the images that did not contain any flocs or those with out-of-focus flocs were deleted.

Knowing the size distribution of flocs is important in accurately modelling floc transport and deposition in riverine and marine environments. This study was a step towards understanding the sensitivity of floc size quantification between two different measurements techniques that are widely used in the literature. We found that d_{50} measured *in situ* in the mixing tank can be different and was generally larger than that measured in a smaller settling chamber. We also found that it is possible for the differences between the methods to be a function of suspension concentration and shear rate. Future research can be focused on comparison between floc sizes obtained using other floc measurement techniques and the potential effect of sediment composition.

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