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Micromechanical determination of the tensile strength of flocculated artificial marine cohesive sediment

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ABSTRACT

Strength characteristics of the flocculated aggregates (flocs) of clay minerals in an aqueous marine environment are important in many modeling applications, such as penetration of heavy objects in the cohesive seafloor, hydrodynamic transport of coastal and seafloor sediments, dredging, and remote sensing among many. Measuring floc strength accurately has been a difficult task due to the fragile, amorphous, and transient nature of the flocs. Here, we describe a micromechanical technique that was used to measure the tensile strength of soft flocculated aggregates produced from simulated marine clay sediments. In this study, artificial flocs similar to the ones that are found on and near the seabed, were prepared using Na-montmorillonite, guar gum, and sea salt. Using the micromechanical technique the average tensile strength of the flocs was found to be 667 ± 189 Pa. The range of tensile strength values is consistent with the nature of the flocs and the measurement technique.

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1. Introduction

Organic flocculants and inorganic clay particles, transported by large river systems, are discharged into lagoons and estuaries. Coagulation and flocculation of this organic and inorganic matter may begin in the fresh water but intensifies significantly as these materials mix with saline seawater. Flocculated materials, referred to as "flocs", can be seen most often in suspension near the sediment-water interface, ([1–3]), or deposited on the seafloor, representing the most recent addition to the consolidating soft sediment layer. Change in the physico-chemical properties of these aggregates is imminent with increasing salinity in the estuary as it connects with the open sea. A decrease in the distance between particles occurs due to a decrease in the double layer repulsive forces in the saline water, resulting in coagulation and flocculation. The size of the flocs depends also on the mixing velocity [4]. Flocs created under low shear stress, as is the case in low turbidity and slow flow velocities, tend to be large and porous, while the flocs created under high turbulent shear tend to be smaller and denser [5].

Quantifying the floc strength is of interest to many applications such as determining the amount of impact and subsequent burial of heavy objects in cohesive sediments for marine engineering (*e.g.* [6]), in efficient removal of aggregated particles in wastewater during treatment for environmental engineering (*e.g.* [7]), in modeling cohesive sediment resuspension (*e.g.* [8]), and even in determining the functions of biofilms (*e.g.* [9]). These approaches need an understanding as well as direct experimental evidence of the principles of individual particle (or a representative floc or aggregate) interaction and breakage. In general, the strength of flocs depends on the inter-particle bonding characteristics. Flocs break if the stress applied is larger than this inter-particle bond strength. In particular, the floc strength is an important factor in sediment transport in the nearshore regions as the floc settling and accumulation depend on the size and the density of the flocs ([5], [10]).

Modeling floc dynamics is of particular interest for cohesive sediment resuspension and transport in rivers, estuaries, and coastal regions. Models for floc dynamics are composed of aggregation and disaggregation terms that attempt to balance the population (*e.g.* [11]). While aggregation of flocs may be modeled mathematically with reasonable accuracy, models for disaggregation (or breakup) tend to be more empirical. Early models assume constant fractal dimension and constant floc yield strength (*e.g.* [12]). Later fractal dimension was allowed to vary as a function of floc size (*e.g.*

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[13,14]). However, these models are not able to predict the temporal evolution of floc size observed in laboratory experiments. When floc yield strength is allowed to vary with floc size and the fractal dimension using a theoretically derived formulation, significant improvement is gained in predicting the temporal evolution of floc size observed in laboratory experiments [15] and field [8].

Quantifying floc strength has been a very difficult task due to their fragile structure and the natural variability in size and morphology. It has often been limited to indirect measurements that infer the strength properties of flocs based on floc disaggregation rates under some form of imposed turbulent flow or impinging jet conditions in an experimental tank [16]. While these types of studies provide valuable statistical results for validating model results, they do not provide direct measurements of strength of individual flocs as a function of the floc size and fractal dimension, needed for calibrating flocculation models for the disaggregation of cohesive sediments.

In our study, a micromechanical manipulation technique was adopted for direct measurements of the floc strength. The study was conducted on flocs created in the laboratory under carefully controlled conditions, representing those found in a natural marine depositional environment. The micromechanical technique, which was first introduced for quantifying flocs prepared from precipitated calcium carbonate as primary particles [17], involves measuring the inter-particle forces within individual flocs. Subsequently, a similar micromechanical technique was also used to quantify the tensile strength of various soft particles such as biofilms [18] and bull sperm [19]. These methods are useful in that they provide evidence of floc strength by direct force application and direct measurements of the results of this application. The force that is needed to break an individual floc is often measured in these studies using a micro cantilever (micropipette) that is observed with the aid of a microscope. In the micromechanical technique [17], opposite ends of the floc are held by two micro pipettes by applying a small amount of suction. While one pipette, referred to as the pulling pipette, is used for pulling the floc, the other pipette, referred to as the force measuring pipette (often with a much smaller diameter and more flexible), is used to measure the force via optical microscope monitoring. During the test, the pulling pipette is moved away from the force measuring pipette, while keeping the tip of the force measuring pipette free to flex until the floc fracture. The deflection of the force measuring pipette is recorded for force calculations, based on some numerical simulations of glass cantilever bending, under assumptions of well-known geometry and glass properties [17].

In our current study, we attempted to replicate the technique of Yeung and Pelton [17]; however, we found that the larger floc sizes used in our tests, as well as the fragile and porous nature of the flocs composed of natural clay-organic components, did not allow for the application of suction, sufficient to hold the floc with the force measuring pipette. Unfortunately, the force measuring pipette needs to be thin and flexible enough to result in sufficient (for optical displacement resolution) flexure under imposed load, until the floc breakage. While utilization of larger pipettes (with higher suction) could assist in such floc attachment, it would also result in much less flexible pipette. Likewise, we necessarily avoided application of excessive suction to minimize the likelihood of affecting the internal floc stress distribution close to the expected rupture plane and also to prevent portions of the floc from being detached into the pipette. Several preliminary force-displacement calibration trials (as further discussed below) showed that the resolution of the force measurement would decline unacceptably low in such circumstances. Consequently, we have devised an alternative method of piercing the floc with the force measuring pipette to yield reliable and satisfactory results.

2. Materials and methods

2.1. Materials

Flocs were created using Na-Montmorillonite (SWy-2) obtained from the Clay Mineral Society (Chantilly, VA). Na-montmorillonite is a 2:1 phyllosilicate. The atomic structure of this type of clay mineral is composed of an octahedral (O) sheet sandwiched between two tetrahedral (T) sheets. These structures are called TOT layers. In order to form clay particles, several of these TOT layers stack with an interlayer space between them. For dry clays, this interlayer distance is about 1 nm [20]. Normally, the clay layers are negatively charged due to the isomorphous substitution. The negative charges of the layers are balanced by the cations that occur in the interlayer. The interlayer cations in Na-montmorillonite clay are represented by sodium. The cation exchange capacity and the surface area of Na-montmorillonite are 91.5 meq per 100 g and 751.8 $m^2\,g^{-1}$ respectively [20]. Mineralogical and chemical analysis of Na-montmorillonite clay may be found in several baseline studies ([21–23]).

2.2. Floc creation

Artificial flocs were prepared using Na-montmorillonitte as the clay mineral, guar gum as a representative marine organic matter, and sea salt. First, the flocs were prepared as a suspension. During the preparation of the floc suspension, the concentration of the clay (Na-montmorillonite) was taken as 3 g L^{-1}. The concentration of the organic matter (OM) was taken as 3% relative to the concentration of the clay mineral, which yielded a measured overall concentration of OM as 0.09 g L^{-1}. The salinity of the suspension prepared was 35 practical salinity units (PSU). All of these selections were based on ranges naturally occurring in marine environment (*e.g.* [24]). Preliminary microscopic imaging studies (unpublished data) also indicated that clay and guar gum tended to flocculate effectively at these ratios leaving little unflocculated clay or guar gum in the suspension. After the mixing of the ingredients, the suspension was sheared for 25 min under constant shear rate condition of 50 s^{-1}, corresponding to a linear velocity of 2.1 cm s^{-1}, in a concentric cylindrical measuring system CC40 (Brookfield r/s + Rheometer Operating Instructions Manual No. M08-219) having a gap of 1.06 mm. The mixing velocity referred to here is the rotational velocity of the double-gap rheometric system used in floc formation, which may also be converted to a linear flow velocity in a typical depositional environment. The mixing procedure approximates the natural formation processes under typical river discharge velocities. Further details on the floccular suspension preparation, mixing methods, as well as the rheological properties are given in [25]. After preparation, the flocs were then used in micromechanical experiments within 5–6 h. Flocs that aged for more than 5–6 h were discarded. This qualification avoids any time-delayed processes of floc deposition onto the bottom of the preparation container, where accumulation and the beginning of the initial stages of the consolidation processes may begin. This maximum time period, after the end of mixing and before floc extraction for micro-manipulation testing, was determined based on several trials and microscope observations of the state of the resulting suspensions. While it would be important to study the changing structure and properties of the flocs during this transition from suspension to consolidated sediment, this was outside the scope of the current project.

2.3. Micromechanical technique: Pipette preparation

Preparation of suitable pipettes is one of the most important tasks in floc manipulation work. The micro-pipettes used in this