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Controls on flocculation and accumulation of mud from moving suspensions: experimental insights into the roles of mineralogy, salinity, suspended sediment concentration, flow velocity and microbes

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ABSTRACT

Understanding how mud moves and deposits is essential for understanding and quantifying the dynamic nature of surface environments and their ancient counterparts. Many factors have been proposed to control the accumulation of mud, including mineralogy, salinity, suspended sediment concentration, flow velocity and microbiota. Flume studies at the IU Shale Research Lab were aimed at investigating the influence of these factors. Earlier work is summarised to provide a snapshot of the current state of understanding. A key parameter that defines mud accumulation is the critical velocity of sedimentation (CVS)—the minimum flow velocity required to keep sediment particles suspended in the water column and prevent them from settling to the bottom. CVS is influenced by a combination of particle and fluid characteristics. Most previous experimental studies have been conducted with sediment composed largely of kaolinite clay in fresh to saline water. Kaolinite, however, is not the dominant clay in natural sedimentary systems on Earth. Recent experiments with the most common clay minerals, illite and smectite, show that these minerals differ markedly in their depositional characteristics from kaolinite. CVSs for illite and smectite are significantly lower (15 and 10 cm/s, respectively) than for kaolinite (25 cm/s) in otherwise identical experiments. Unlike kaolinite, salinity does influence their CVSs. The reasons for these differences are likely related to the morphology of the different clay minerals and the distribution of their surface charges. These significant differences were not observed in earlier experiments, most likely because microbial films formed on the various clay minerals and floccules during extended experimental runs. Particle binding by microbially generated extracellular polymers appears to have counterbalanced the pronounced differences in behaviour among clay minerals observed in intentionally sterile experiments. Ubiquitous microbial coatings, typical for natural environments on Earth since the Archean, apparently make muddy suspensions behave similarly regardless of the dominant clay mineral. In abiogenic settings, however, such as planets devoid of life, mineral controls on flocculation behaviour may result in sediment distribution patterns that are unknown on Earth.

Keywords depositional parameters, flow velocity, illite, kaolinite, microbial influences, mineralogical controls, moving suspension, salinity, smectite, suspended sediment concentration.

INTRODUCTION

The details of deposition of mud-size particles (<63 microns) from moving suspensions are well documented through experimental work conducted over the past two decades (e.g. Schieber *et al.*, 2007, 2023; Schieber, 2011; Yawar & Schieber, 2017). Extensive study of sedimentary structures in ancient mudstone successions (e.g. Lazar *et al.*, 2015 and references therein) confirms the broader relevance of these experiments. Flocculation is an important process that can include both physiochemical and biological aggregation mechanisms and governs transport and deposition of mud in a wide range of depositional environments, including rivers and associated floodplains, coastal environments (e.g. estuarine and prodelta) and continental shelves (Kranck, 1973; Sternberg *et al.*, 1999; Hill *et al.*, 2001; McAnally & Mehta, 2002; Safak *et al.*, 2013; Shchepetkina *et al.*, 2018). In moving suspensions of clay to silt-size (mud) particles, van der Waals aggregation of particles, also known as flocculation, is the critical element that causes mud to give rise to sand-sized bedload floccules and to accretion of mud beds at flow velocities capable of moving sand-size quartz grains in bedload (Schieber *et al.*, 2007). The factors that influence flocculation are many and include mineralogy, salinity, suspended sediment concentration, flow velocity, fluid density and viscosity and microbiota. Their effects have been summarised by Schieber *et al.* (2023), and specifics are described in references cited therein.

In order to avoid confusion further down in the narrative, a few definitions probably bear repeating (Potter *et al.*, 1980; Lazar *et al.*, 2015). Mud and mudstone are composed largely of particles that are smaller than 62.5 μm. It is a size-based definition; no particular mineralogy is implied. Mudstones do not have to contain clays, even though they do so in many instances. Clay can mean two things: (1) particles that are smaller than 4 μm or (2) actual clay minerals (hydrous aluminium phyllosilicates). Silt is whatever is larger than 4 μm and smaller than 62.5 μm. So, when we say clay mineral, we mean just that, no size implication and when we say clay size, it means 4 μm or smaller, and silt size means everything between 4 μm and 62.5 μm.

Water-rich flocs are fragile and a long-held assumption was that they should disintegrate at the base of muddy suspensions (in the viscous sublayer, Einstein & Li, 1956; Schlichting, 1979), where shear rates are at a maximum (Mehta & Lott, 1987; McCave & Hall, 2006). The discovery that clay-mineral flocs form sturdy bedload floccules that travel across the substrata as ripples (Schieber *et al.*, 2007; Schieber & Southard, 2009; Yawar & Schieber, 2017), was unexpected and led to a paradigm shift with regard to the transport and deposition of mud in both modern and ancient mud-dominated systems (e.g. Aplin & Macquaker, 2011; Plint *et al.*, 2012; Trabucho-Alexandre *et al.*, 2012; Chang & Flemming, 2013; Lazar *et al.*, 2015; Schieber, 2016; Schieber *et al.*, 2019; Smith *et al.*, 2019; Li *et al.*, 2021).

Yet, whereas experimental work (Schieber *et al.*, 2007) and field studies (Lazar *et al.*, 2015) appear to complement each other rather nicely; there is a fundamental difference between flume experiments and the muds and mudstones whose interpretations they inform. Studies of clay-mineral behaviour in sedimentation processes, be they by civil engineers or sedimentologists (e.g. Mehta & Partheniades, 1979; Baas & Best, 2002; Schieber *et al.*, 2007), overwhelmingly use kaolinite because of its ready availability and an abundance of prior published studies, whereas most natural muds and mudstones are strongly dominated by smectite and illite. Although differences in the settling and flocculation behaviour of various clay minerals have been examined in early studies (Gibbs, 1983, 1985 and references therein) and non-linear relationships appear commonplace, to our knowledge, there are no comparable flume studies that explore how these differences relate to the behaviour of smectite and illite in sedimentation processes, especially in moving suspensions.

This paper presents the results of new multi-parameter investigations that focused on the interactions of mineralogy, salinity, suspended sediment concentration and flow velocity. In particular, flume experiments were set up to explore how illite and smectite behave under conditions that produce sediment accumulation associated with bedload transport of flocculated clays. Particular attention was paid to the critical velocity of sedimentation (CVS) and rates of transfer of mud from suspension to bedload (bedload transfer). CVS is the minimum flow velocity required to keep sediment particles suspended in the water column and prevent them from settling to the bottom. For the best possible comparability among experiments, experiment design and execution follow the protocols

Table 1. Influences on flocculation and accumulation for kaolinite, illite, smectite.

Effects & Variables	Kaolinite	Illite	Smectite	Notes
layer structure	1:1	2:1	2:1- expandable	
cation exchange capacity	low	moderate	high	
impact of increased salinity on static settling/flocculation rate	↓	↑	↑	
Critical Velocity of Sedimentation CVS	25	15	15/10	cm/sec
impact of increasing SSC on CVS	± none	± none	± none	
impact of increasing salinity on CVS	± none	± none	-5 cm/s -	(for saline)
in-flow suspended floccule size	~20 μm	~25 μm	~35 μm	salinity insensitive
impact of increased salinity on bedload floc size	± none	± none	± none	salinity boosts flocculation rate not floccule size

already developed in our earlier work with kaolinite (Schieber *et al.*, 2007, 2023). The results show clear differences between kaolinite, illite and smectite with regard to CVS and the influence of salinity (Table 1).

A better understanding of the factors that have a bearing on the critical shear stress of deposition for flocs of different mineral composition moves us closer to more quantitative interpretations of the depositional energy associated with fine-grained sediments and sedimentary rocks. Our kaolinite experiments (Schieber *et al.*, 2023) as well as the here-presented illite and smectite experiments were conducted in a way to minimise or prevent microbial activity during experiments and thus emphasised differences in flocculation behaviour due to mineralogy and inherent differences in surface charges. The results present a distinct contrast to observations from earlier experiments that were run for extended time periods (weeks to months) and in which the sedimentation behaviour of kaolinite, illite and smectite suspensions appeared somewhat similar (Schieber, 2011). This difference points to an equalising role of organic binding agents in natural systems. Although the broad importance of this issue is discussed here on the basis of presently available data, there is a clear need for a deeper understanding of the impact of organic/microbial matter on sediment transport characteristics of muddy flows.

METHODS AND EXPERIMENTAL DESIGN

The illite and smectite experiments reported here were conducted with the same equipment used for kaolinite experiments already published (Schieber *et al.*, 2023). Flume design and experimental protocols were identical for kaolinite, illite and smectite experiments and have been detailed in Schieber *et al.* (2023) (a summary of procedures is contained in Data S1). The only variable that was changed was the addition of illite or smectite (instead of kaolinite) to the flume.

Marketed as ‘argile verte’, the illite clay was sourced from Argiletz, a French purveyor of clays for cosmetic applications. X-ray diffraction analysis by Dr David Bish found the material to contain 55% 2 M1 illite, 34% quartz and calcite, 6% kaolinite and chlorite and an assemblage of minor phases. Although not a pure clay, the high illite content still ensures that experiments reflect the behaviour of illite-dominated natural suspensions. The illite feed (Fig. 1) has a grain-size distribution of 90% finer than 10 μm, 70% finer than 4 μm and 50% finer than 2 μm.

The smectite used in these experiments was sourced from Southern Clay Products Inc. in Gonzales, Texas. Marketed as ‘Bentolite’, the supplier describes it as consisting largely of Ca-montmorillonite, even though the term smectite is used in associated literature (Chipera & Bish, 2001). In addition to Ca-montmorillonite, it contains 1% to 5% fine crystalline quartz, and the term smectite will be used for the remainder of the paper. The smectite feed (Fig. 2) has a grain-size distribution of 90% finer than 25 μm, 70% finer than 7 μm and 50% finer than 3 μm.

Settling tube experiments certify the settling properties of the three clay minerals studied, with regard to salinity and suspended sediment concentrations. Largely because, depending on where a given clay is mined and processed, its

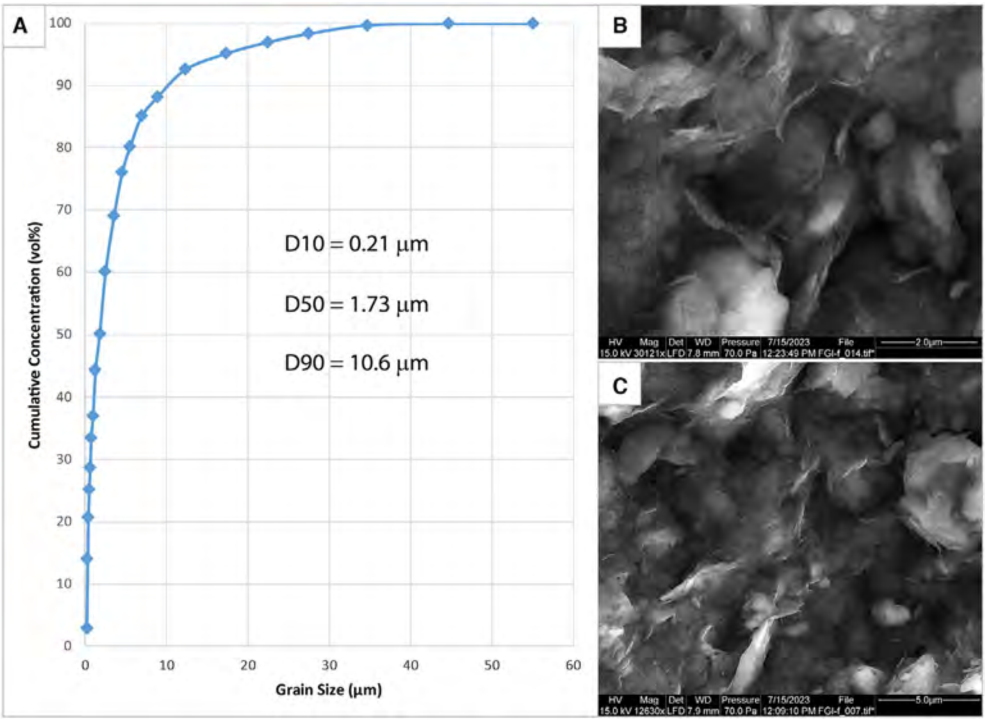


Fig. 1. (A) Grain size distribution of the illite used in experiments (determined by the Sedigraph method). Note that 90 vol% of the material is smaller than 10 µm. (B) SEM image of the dominant fine fraction, consisting of micron-sized illite flakes. (C) Overview SEM image of the illite at lower magnification, showing larger particles buried in a finer matrix.

properties may differ from the 'textbook behaviour' that tends to guide perceptions of many sedimentary geologists. In these experiments, the illite and smectite clays that were used in flume experiments were mixed with 1 litre of the same water compositions (freshwater, 2% and 3.5% salinity) that were used in flume experiments, and the settling of these mixtures over time was recorded with a time-lapse camera.

The data presented here are the result of a systematic examination of the influences of suspended sediment concentration, salinity and flow velocity (bottom shear stress) on the formation of illite and smectite bedload flocs and consequent bed accretion. These experiments complement an earlier examination of kaolinite behaviour (Schieber *et al*., 2023).

These experiments had durations of approximately 8 h (short drop) and 100 h (long drop). Flows of that duration are of interest to the sedimentologist because they compare in duration to flows associated with tidal cycles, river floods and storms, the essential mechanisms that transport mud to depocenters (Schieber, 2016). Velocity step-down experiments of week-long duration ('Long Drop', ~100 h) show the trends in sediment removal from the flow at given velocities (Fig. 3), and the shorter ('Short Drop', ~8 h) experiments were designed to explore a wide range of suspended sediment concentrations. In addition, all experiments were conducted at three salinities: tap water (~0.00005% mg/L dissolved solids), 2% salinity, and 3.5% salinity; so as to be able to compare sedimentation characteristics of freshwater, brackish and fully marine settings. Velocity was decreased in 5 cm/s steps for all our experiments (Schieber *et al*., 2007; Schieber, 2011). Suspended sediment concentrations were measured with optical backscatter sensors (OBS), and bedload transfer for a given velocity step is defined as the difference in suspended sediment concentration at the start versus the end of a given velocity step (Fig. 3).

OBSERVATIONS

Static deposition (stillwater settling)

Although this study is aimed at the controls on illite and smectite deposition from moving

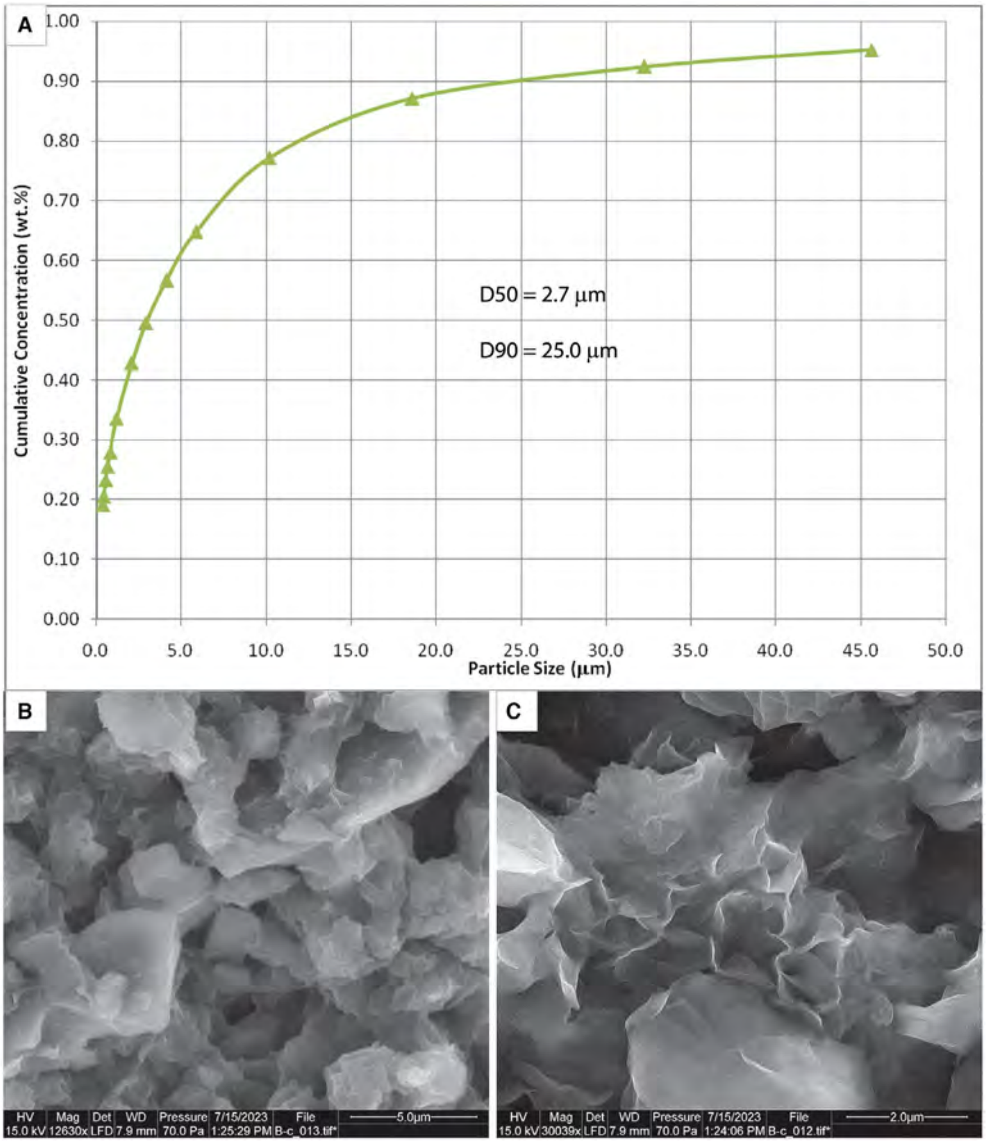


Fig. 2. (A) Grain size distribution of the smectite used in experiments (determined by the pipette method). Note that 78 wt% of the material is smaller than 10 μm. (B) Overview SEM image of the smectite at lower magnification, showing aggregated larger particles. (C) SEM image of the smectite at higher magnification, showing characteristic honeycomb texture.

suspensions, evaluating their behaviour in a dynamic setting benefits from knowing how the experimental materials behave under static conditions (gravity settling from still suspensions). It is also important to do this for better comparison of our results to comparable future studies by other investigators, because there can be substantial differences between clays produced from different mines/localities and stratigraphic intervals (David Bish, pers. comm.), even though they may all be classified as illites or smectites. In addition, depending on chemical and heat treatments, commercially available clays may deviate significantly from natural samples (David Bish, pers. comm.). Static settling characterisation of a clay gives guidance with regard to potential behaviour in flume experiments vis-à-vis suspended sediment concentration (SSC) and salinity. For the earlier kaolinite experiments (Schieber *et al.*, 2023), settling behaviour was examined at 1, 10 and 20 g/L and in freshwater and 3.5% salinity water, and the

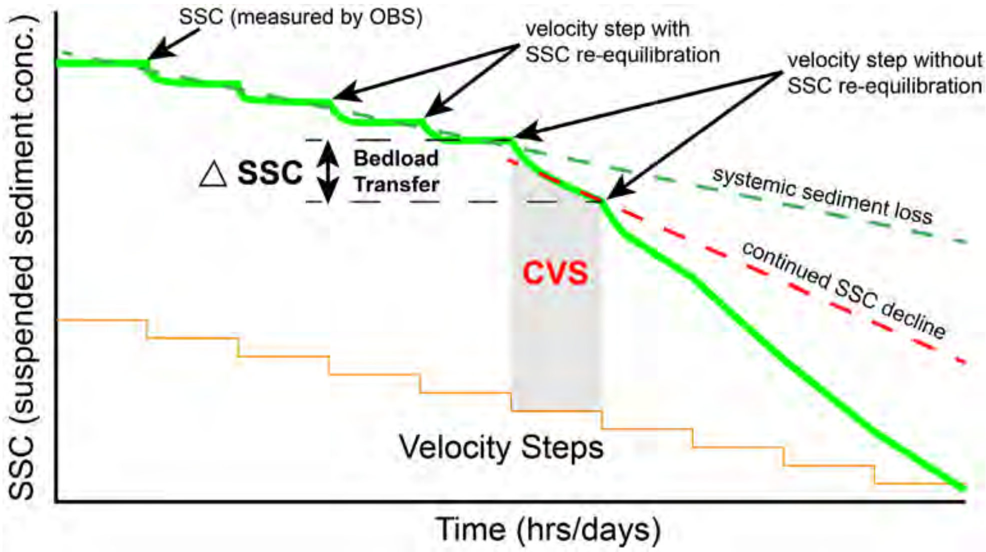


Fig. 3. Schematic depiction of velocity step-down experiments. Orange step line shows decreasing flow velocity (usually in 5 cm/s increments, starting at 50 cm/s) over time. Green line shows suspended sediment concentration (SSC) over time, responding to velocity reduction. At velocities above critical velocity of sedimentation (CVS), each velocity step causes some coarser silt to transfer to bedload, and SSC re-equilibrates at a lower level. The first velocity step without re-equilibration (grey shading) marks the CVS and onset of continuous bedload transfer of flocculated material (dashed red line). Systemic sediment loss (dashed green line) is a combination of coarser particles becoming bedload as turbulence decreases and sediment loss in the reduced turbulence 'corners' of the flume. Bedload transfer (Δ SSC) significantly increases once CVS is reached.

same parameters were used for the illite and smectite experiments (Fig. 4).

Kaolinite, illite and smectite show distinct differences in settling behaviour with regard to salinity and SSC, although the received wisdom is that clay flocculation generally increases as salinity increases (e.g. Potter *et al.*, 1980). At low sediment concentrations (a few grams per litre), kaolinite settling (Schieber *et al.*, 2023) is initially largely unaffected by salinity, though after several hours of settling, saline matrices have cleared more effectively. At high kaolinite concentrations (10 g/L or more), however, freshwater settling initially outpaces settling in saline waters and roughly equals saline settling over longer time periods (Schieber *et al.*, 2023).

In illite suspensions, in contrast (Fig. 4), salinity-enhanced settling becomes evident as little as 30 min into the experiment at all sediment concentrations, with the effect being increasingly conspicuous at higher sediment concentrations. Nonetheless, in illite/freshwater suspensions, visible interfaces between suspension and supernatant still develop over time.

In smectite suspensions the freshwater versus saline behaviours show the strongest contrast (Fig. 4). Saltwater settling distinctly outpaces freshwater settling after as little as 30 min, and most freshwater suspended sediment is still in suspension after several hours, although the silt-size fraction forms a thin deposit at the bottom (yellow arrows in Fig. 3). The saline settled smectite does not show this feature, presumably because of silt-size grains being trapped in larger floccules. Overall, kaolinite, illite and smectite illustrate a depositional continuum from least saline influenced (kaolinite), to most saline influenced (smectite), with illite occupying an intermediate position. This general relationship also finds expression in the nearshore to offshore distribution of major clay species that have been observed in modern and ancient sediments by Parham (1966), Edzwald & O'Melia (1975) and Gibbs (1977).

Dynamic deposition (from moving suspension)

Dynamic deposition: in-flow floccule sizes

In initial experiments, observation of floccules was focused on those that travelled over the flume bottom as bedload, because they were

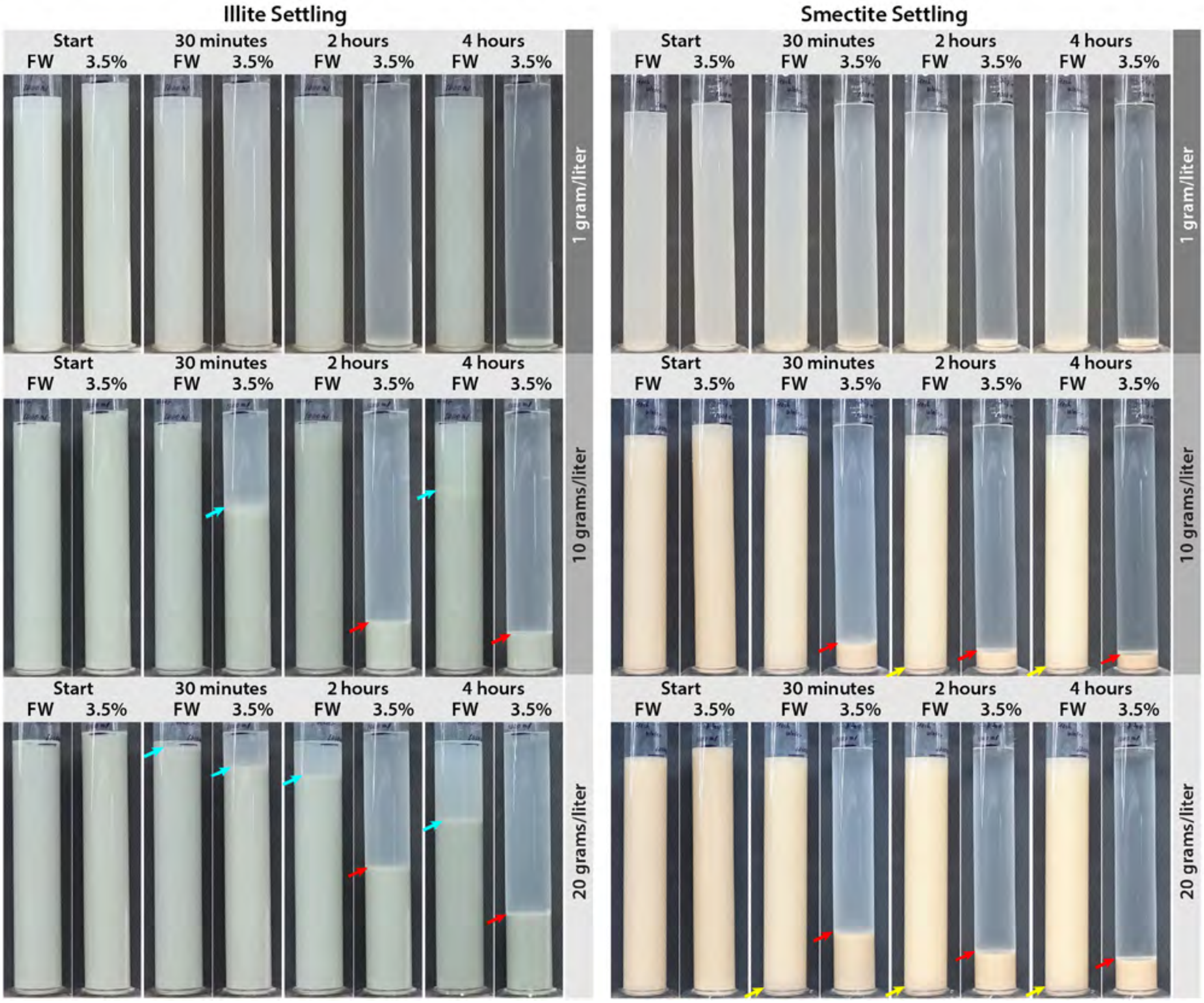


Fig. 4. Settling behaviour of the illite and smectite clay used in the described flume experiments. Turquoise arrows mark diffuse interfaces in freshwater suspensions of illite, yellow arrows mark basal silt deposits in freshwater suspensions of smectite, and red arrows mark sharp interfaces in saline suspensions of both illite and smectite.

comparatively easy to observe and record. A floc camera system on loan from Virginia Tech was used to acquire the sizing of in-flow floccules. Recording in-flow flocs from the start of each experiment showed an abundance of in-flow floccules even at high flow velocities (Fig. 5). Although the floccule data are a bit 'noisy', one has to keep in mind that automated floccule measurement is still a technique under development, and that the data nonetheless provide useful information.

Regardless of salinity, in-flow floccules fall into the 20 to 40 μm size range (Fig. 5). Even though the in-flow turbulence gradually decreases as flow velocity is reduced, floccule size largely does not reflect this decrease. This observation suggests that across the explored velocity range (10 to 50 cm/s; negligible in-flow floccules at 5 cm/s), turbulent shear is sufficient to limit in-flow floccules to a maximum size of some 10 μm. There also appear to exist systematic differences in in-flow floccule size with regard to the clay minerals involved. In the preceding kaolinite experiments (Schieber *et al.*, 2023), in-flow floccule sizes hovered around 20 μm, whereas illite in-flow floccules register slightly larger (~25 μm), and smectite in-flow floccules are the largest (more fluffy?) at around 30 to 40 μm.

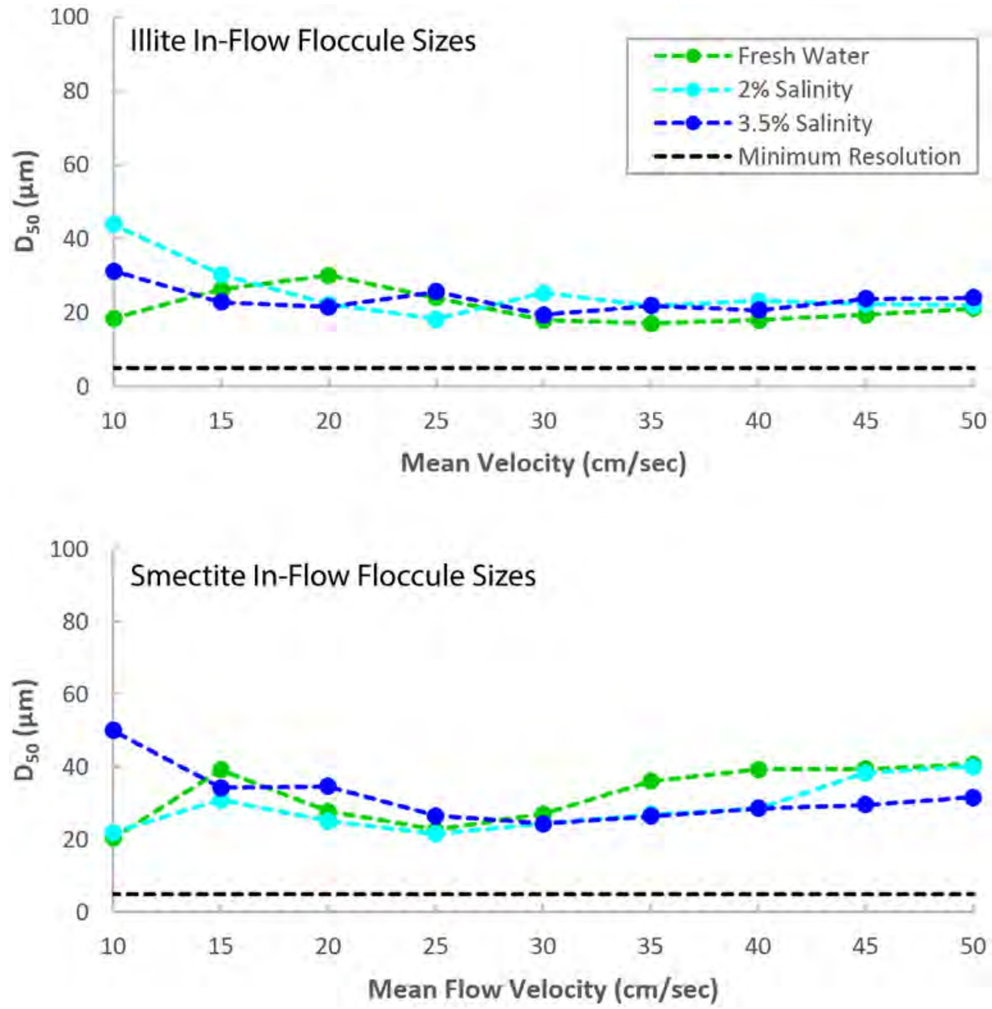


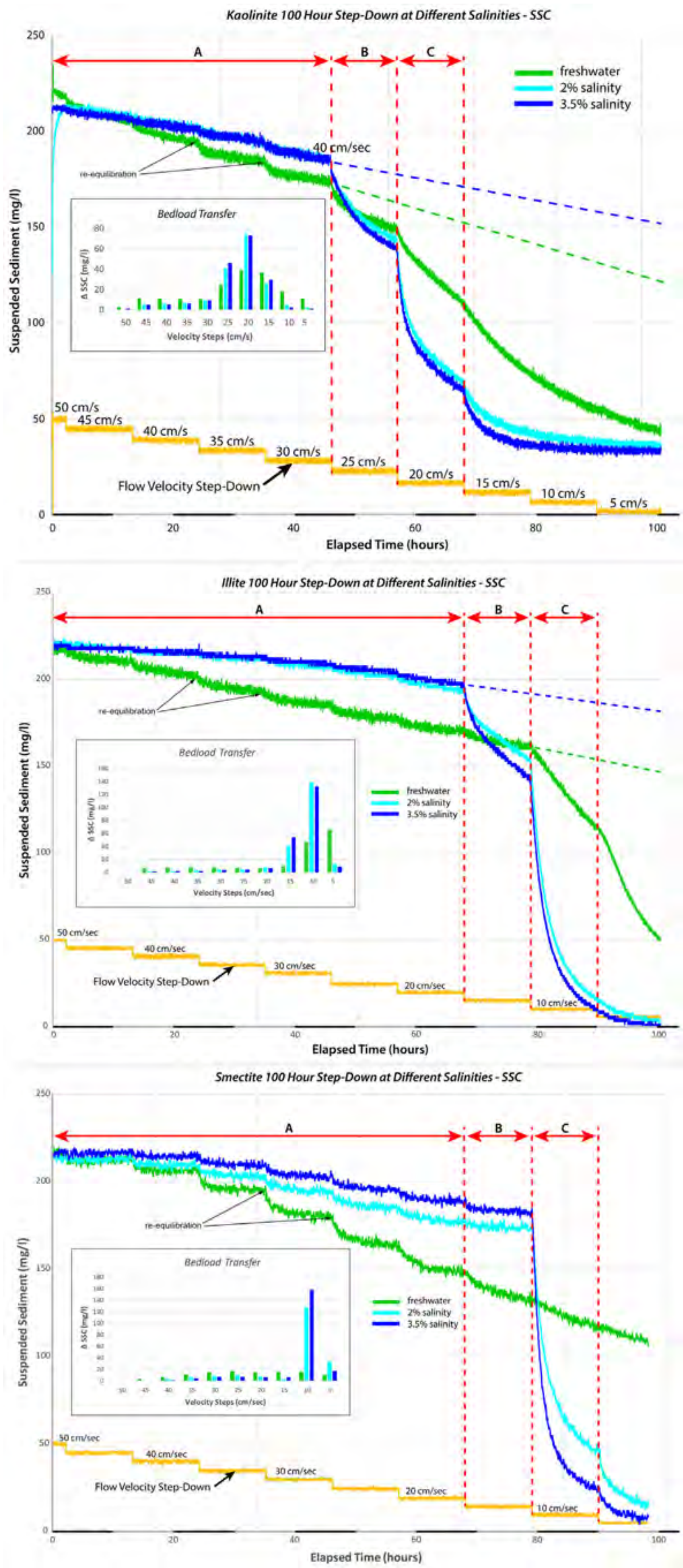
Fig. 5. In-flow floccule sizes for illite and smectite at three different salinities.

Dynamic deposition: long-duration-flow ('long drop') experiments

In 'Long Drop' experiments, 150 g of clay was added to 650 litres of water and flow velocities were gradually reduced over a 100 h time interval. Although the sediment addition computes to a nominal SSC of 230 mg/L, because the coarse silt portion is largely in bedload at 50 cm/s, actual initial SSC values are in the 210 to 230 mg/L range (Fig. 6).

A general observation that can be made in Fig. 6 for all clays is noted that in region 'A', the velocity reduction steps result in slight successive drops and re-equilibration of SSC (see also Fig. 3), and overall SSC decline seems to follow a linear trend (the dashed blue and green lines in Fig. 6). Given that region 'A' is in terms of flow velocity above the CVS (Schieber *et al.*, 2007), the decline of SSC probably has two components. The main component is

Fig. 6. 'Long drop' curves for kaolinite (Schieber *et al.*, 2023), illite and smectite in freshwater, brackish water (2% salinity) and seawater (3.5% salinity). In each diagram, the curves are demarcated with red double arrows 'A', 'B' and 'C'. Region 'A' is characterised by a succession of re-equilibration steps (Schieber, 2011), and ends where the next velocity step does not re-equilibrate, and SSC declines continuously for at least one salinity level. Region 'B', marks the first non-equilibrating step down, and region 'C' marks the following, typically steeper SSC decline.


Kaolinite 100 Hour Step-Down at Different Salinities - SSC
A
B
C
freshwater
2% salinity
3.5% salinity
40 cm/sec
re-equilibration
Bedload Transfer
Δ SSC (mg/l)
Velocity Steps (cm/s)
Suspended Sediment (mg/l)
50 cm/s
45 cm/s
40 cm/s
35 cm/s
30 cm/s
25 cm/s
20 cm/s
15 cm/s
10 cm/s
5 cm/s
Flow Velocity Step-Down
Elapsed Time (hours)
Illite 100 Hour Step-Down at Different Salinities - SSC
A
B
C
re-equilibration
Bedload Transfer
Velocity Steps (cm/sec)
freshwater
2% salinity
3.5% salinity
Suspended Sediment (mg/l)
50 cm/sec
40 cm/sec
30 cm/sec
20 cm/sec
10 cm/sec
Flow Velocity Step-Down
Elapsed Time (hours)
Smectite 100 Hour Step-Down at Different Salinities - SSC
A
B
C
re-equilibration
Bedload Transfer
Velocity Steps (cm/sec)
freshwater
2% salinity
3.5% salinity
Suspended Sediment (mg/l)
50 cm/sec
40 cm/sec
30 cm/sec
20 cm/sec
10 cm/sec
Flow Velocity Step-Down
Elapsed Time (hours)

plausibly systemic sediment loss in the flume itself (see Schieber *et al.*, 2023 for details), whereas the second component, small SSC down-steps (re-equilibration) that coincide with velocity steps (Fig. 6), is associated with silt-size particles that transfer from suspension to bedload as flow turbulence decreases (Schieber *et al.*, 2023). The SSC trendlines for freshwater experiments (dashed green lines in Fig. 6) are steeper than for saline experiments, and the comparatively larger degree of sediment loss reflects the density (buoyancy) difference between freshwater and saline fluids (Schieber *et al.*, 2023).

The most important and new observation to be made in Fig. 6 is that CVS differ for each clay tested. For kaolinite, the first non-equilibrating velocity step (= CVS) is at 25 cm/s for all salinities; for illite, it is at 15 cm/s for all salinities, and for smectite, it is at 15 cm/s for freshwater, and at 10 cm/s for saltwater. CVS appears to depend on what clay mineral dominates the flow, plausibly due to the shape and size of clay particles and by extension surface charge distribution and likely floc strength.

The 15 cm/s step (region 'B') for illite shows a steady though gentle decline in the freshwater experiment, and significant drops for the saline flows. The system is below the CVS for all three salinities, but one may surmise that the freshwater CVS for illite is just above 10 cm/s, whereas for the saline flows, it is likely closer to 15 cm/s. Once region 'C' (Fig. 6) is reached, the illite drop curves steepen considerably for freshwater flows, and dramatically for saline flows.

Although smectite in freshwater reaches non-equilibration and continuous decline when 15 cm/s is reached, the decline curve does not steepen appreciably for the 10 and 5 cm/s velocity steps. Thus, even though the CVS for freshwater flows is in the 15 cm/s bracket, sediment loss once reaching that mark does not mark a significant break in flow behaviour. In contrast, when smectite in saline flows reaches non-equilibration at 10 cm/s. SSC drops so dramatically that the flow clears up (becomes transparent) noticeably within a few hours.

A drop in SSC readings implies the transfer of suspended sediment to bedload, and thus the drop curves for the various clays result in specific bedload transfer behaviour. Whereas for kaolinite, most sediment enters the bedload realm between 25 cm/s and 15 cm/s, this region is in the 15 cm/s and 10 cm/s velocity range for illite, and largely occurs during the 10 cm/s velocity step for smectite (Fig. 6).

Dynamic deposition: short-duration flow ('short drop') experiments

In 'Short Drop' experiments (8.5 h duration), sediment loading of the flume was step-wise increased (50, 100, 150, 300 and 600 g) to explore the influence of SSC (nominally 77, 154, 231, 462 and 923 mg/L) on the CVS. The experiments were conducted in freshwater, as well as at 2% and 3.5% salinity (Figs 7–10). Although these experiments were in duration less than 1/10 of the 'Long Drop' experiments, the saline drop curves show essential features (non-equilibration, continuous decline, etc.) at the same velocity marks (Figs 8 and 10) as observed in the 'Long Drop' experiments (Fig. 6). Freshwater runs do not show as much differentiation (Figs 7 and 9), presumably because more time is needed for re-equilibration steps to manifest. Whereas in 'Long Drops', CVS for illite and smectite is detectable at 15 cm/s (Fig. 6), the freshwater decline curves for 'Short Drops' are largely sloping lines without significant breaks. Overall, the 'Short Drops' indicate that the flow behaviour of illite and smectite is consistent across the time spans of most natural flow events, and is insensitive to changes in SSC. Only freshwater and fully saline (3.5% salinity) experiments are shown; the 2% salinity experiments look very similar to the 3.5% salinity experiments and can be found in Data S2.

Bedload floccules, sediment accumulation and bed formation

In an earlier study of kaolinite behaviour (Schieber *et al.*, 2023), bedload floccule sizes were measured manually for all experiments because attempts at automating the process via image processing software were not successful. That study illustrated clearly the correspondence of bedload floccule formation and distinct slope breaks in SSC decline curves (Fig. 6). Because manual measurement of floccule sizes is an exceedingly tedious task (mindblisteringly dull, really), in the current study, the SSC decline curves were used instead to determine the onset of bedload floccule formation, bedload transfer and CVS for each experiment. Close-up cameras were set up for routine recording of particle transport across the flume bottom, and representative images of particle/aggregate sizes for the 20 to 5 cm/s velocity steps are shown in Fig. 11 (a set of images that covers the full 50 to 5 cm/s

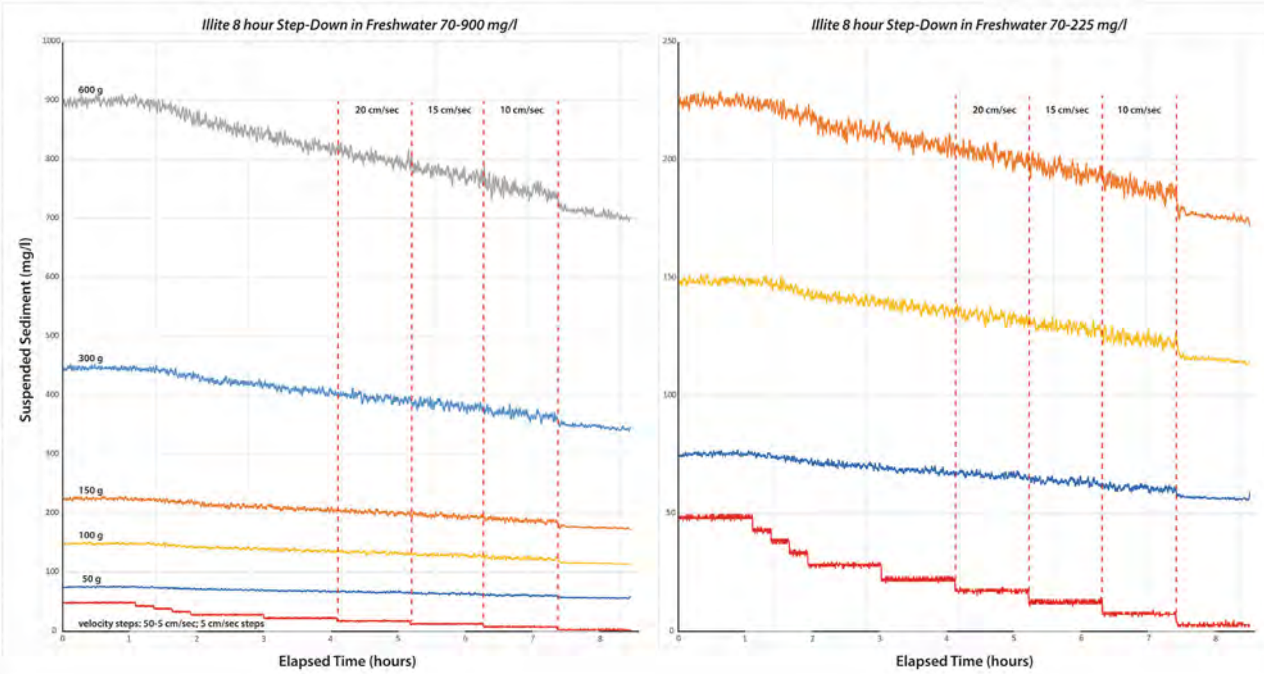


Fig. 7. Illite 'Short Drop' experiments for freshwater (left), and at right, more detailed plots of the 77 through 231 mg/L experiments. Unlike in the 'Long Drop' experiment (Fig. 6), there is no clearly developed drop of SSC associated with the 10 cm/s velocity step. There is a drop at the 5 cm/s step, and the reduced scatter of the SSC curves suggests that at this point, the internal turbulence is low enough to allow faster settling of the remaining sediment below the level of the OBS sensor (at flow half height). This effect manifests in a modified form in the saline experiments as well (Fig. 8).

velocity range is available in Data S3). The onset of coarser particles (sand-size aggregates/floccules) coincides with the SSC changes observed in the experiments, and the amount of bottom sediment mirrors the steepness of the SSC drops (Fig. 6). There is good correspondence between directly observed changes in particle sizes and bed coverage and the SSC evolution of the velocity step-down experiments.

As seen in Fig. 11, at 20 cm/s neither illite nor smectite show anything but silt-size particles at the flume bottom (20 to 40 μm) in both fresh and saline waters (Fig. 11A to D), and SSC re-equilibrates after the velocity drop (Fig. 6). At 15 cm/s the illite freshwater experiment shows an onset of larger particles (60 to 80 μm, Fig. 11E), whereas the saline illite experiment shows bedload particles in the very fine sand range (70 to 100 μm, Fig. 11F) in such abundance that the bed is 80% covered by sediment. For smectite at 15 cm/s, there is a minor uptick of coarser particles (50 to 80 μm, Fig. 11G) in freshwater, and a more noticeable coarsening (mostly 30 to 60 μm, Fig. 11H) in the saline experiment. In the latter case, however, many of these larger particles are smeared out (elongated) in the flow direction, an indication that they are still entrained in the flow instead of rolling and tumbling over the flume bottom. Thus, whereas the system is clearly inching towards CVS, it probably has not reached that threshold quite yet. Reaching 10 cm/s, illite experiments show a flume bed with general sediment coverage, and bedload particles (60 to 100 μm freshwater, Fig. 11I; up to 150 μm saline, Fig. 11J) that are increasingly difficult to differentiate from the already accumulated sediment. Bed coverage at this point is clearly better developed in saline experiments (Fig. 11J). For smectite at 10 cm/s, there is a visual coarsening of bedload particles to sand size (70 to 150 μm) for both freshwater and saline experiments (Fig. 11K and L). Onset of non-mobile bed coverage (images were chosen to show floccule size) is modest for freshwater experiments and well developed for saline experiments. For the final velocity step (5 cm/s),

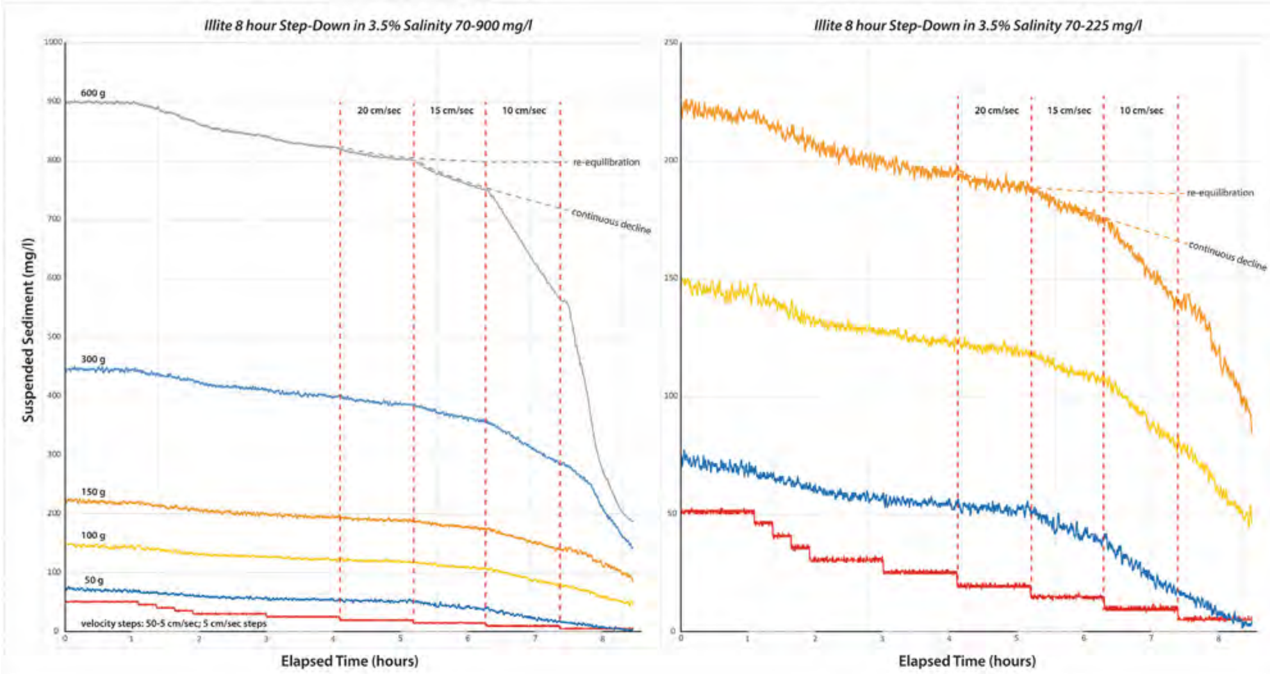


Fig. 8. Illite 'Short Drop' experiments for 3.5% salinity (left), and at right, more detailed plots of the 77 through 231 mg/L experiments. Just like in the 'Long Drop' experiment (Fig. 6), there is a clearly developed drop of SSC associated with a continuous decline at the 15 cm/s velocity step. The 'bumps' just at the switch to 5 cm/s appear due to rapid settling of sediment out of the upper half of the flow, temporarily raising SSC at the OBS sensor elevation (at flow half height). Whereas at higher velocities the flow shows uniform SSC across the depth of the flow (tested with the installation of multiple OBS sensors in other experiments), in the 923 mg/L experiment (600 g sediment addition), an unbalancing is clearly visible at the onset of the 5 cm/s drop. Optically, the upper centimetres of the flow became briefly more transparent, and the settling material pushed up SSC readings in the lower half of the flow temporarily.

bed buildup and sand-size floccules are common for both illite and smectite experiments (Fig. 11M to P). Bed buildup for illite starts at 15 cm/s for saline flows and for freshwater flows at 10 cm/s. In the case of smectite, most bed coverage is accomplished during the 10 cm/s velocity step.

Biotic influences

Early in our exploration of clay behaviour in moving suspensions (e.g. Schieber, 2011), the majority of experiments lasted several weeks to even months, and the general progression of experiments (reaching CVS, floccule-ripple formation, bed buildup) was the same regardless of which clay was used. Using fine-grained non-clay materials, such as dispersed diatomite, aragonite mud and precipitated calcite (Schieber *et al.*, 2013), and crushed quartz (≤5 μm; Yawar & Schieber, 2015), produced comparable results. Thus, the tacit understanding in those days was that the CVS for most muddy suspensions was in the neighbourhood of 25 cm/s (or a bed shear of about 0.2 to 0.25 Pa), and that this general behaviour could be counted upon as long as a sediment mixture was fine-grained enough.

Those earlier findings contrast with the differences observed here, regarding the sedimentation behaviour among kaolinite, illite and smectite; differences that have been replicated in multiple re-runs of these experiments. Because the observed differences appear to be real and in accordance with the physical properties of these minerals, they point to gaps of understanding vis-à-vis the larger body of experimental work published previously (e.g. Schieber, 2011; Schieber *et al.*, 2013; Yawar & Schieber, 2015). The IU Shale Research Lab team missed this important aspect of mud flocculation in earlier work because the focus was on behaviours that had not been described previously, and there was plenty of ground to cover

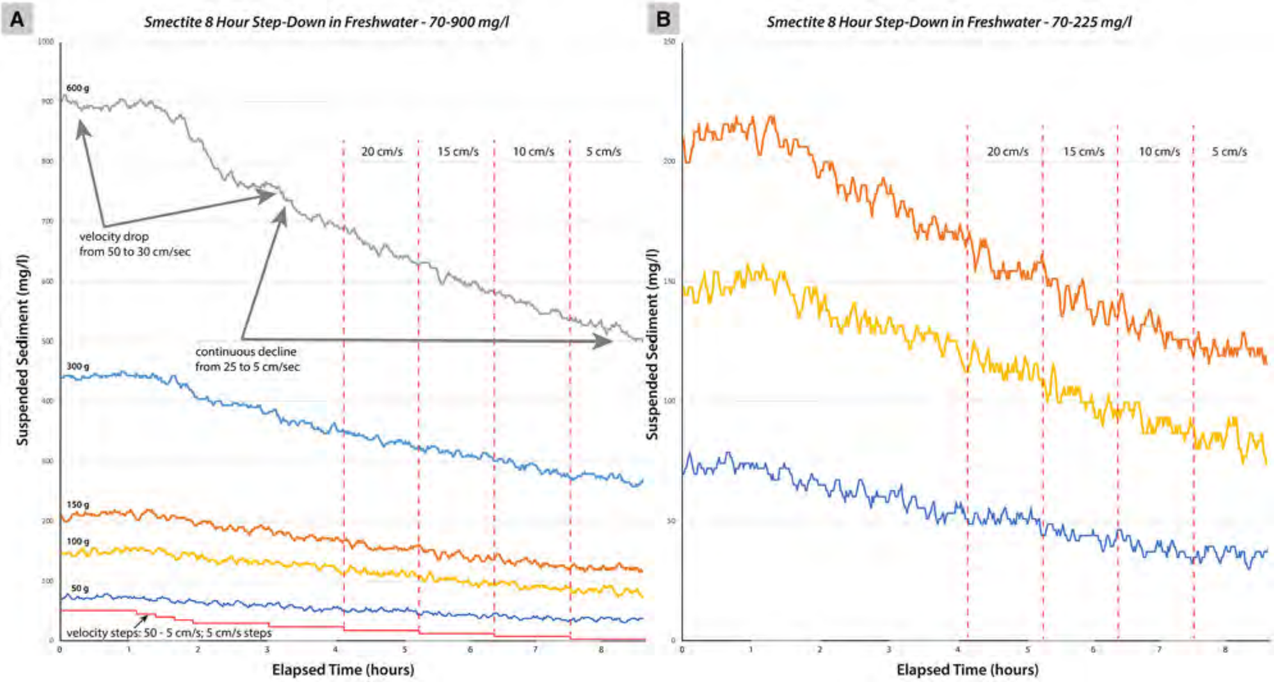


Fig. 9. Smectite 'Short Drop' experiments for freshwater (left), and at right, more detailed plots of the 77 through 231 mg/L experiments. Unlike in the 'Long Drop' experiment (Fig. 6), there is no clearly developed downstepping of SSC in response to velocity steps, just a steady SSC decline towards lower flow velocities. There is no feature that, with confidence, could be interpreted as a marker for reaching CVS, presumably because there was not enough time for its development. The increase of SSC seen over the first hour in some experiments is due to the fact that the clays of the previous run were recycled (we added sediment to get to the next load point) and had to be re-suspended at the start of the next run.

without worrying about cryptic biofilms. Electron microscopy on floccules retrieved from the flume was essential for their recognition, and the procedures for doing that were developed over the course of multiple years and not in place during the early stages of our research programme.

From the start (Schieber, 2011), each experiment was conditioned with more chlorine than is used to keep swimming pools free from algae and biofilms. Intent on studying abiotic systems, only clay (or other fine-grained materials) and water were added to the flumes that were cleaned thoroughly between experiments. No visible manifestations of biofilms were observed, but on Earth, microbial life is nonetheless found in the most unlikely places. Dried sedimentary deposits and floccules (collected from the flow) from various earlier experiments of longer duration had been stored in the lab's collection, and when examined under the SEM, clear evidence of biofilms was observed associated with floccules (Fig. 12). Thus, in spite of efforts to keep things 'clean', microbes had compromised our experiments. Further testing showed that in chlorine-conditioned experiments these kinds of cryptic biofilms become detectable if an experiment extends for more than 4 to 5 days. Thus, pretty much all of our longer-run experiments, even when bleach was also added intermittently, likely were impacted by biofilm growth. Given how readily these biofilms developed, and considering that they consist largely of water and are thus of little substance, the IU Shale Research Lab team is confident that clay behaviour in the great majority of experimental studies by coastal and civil engineers has likely been affected by cryptic biofilm development. The above-noted convergence of CVSs among various clay minerals and non-clay mineral fines, and the distinct CVS differences seen in short-duration experiments therefore are plausibly due to the influence of microbiota.

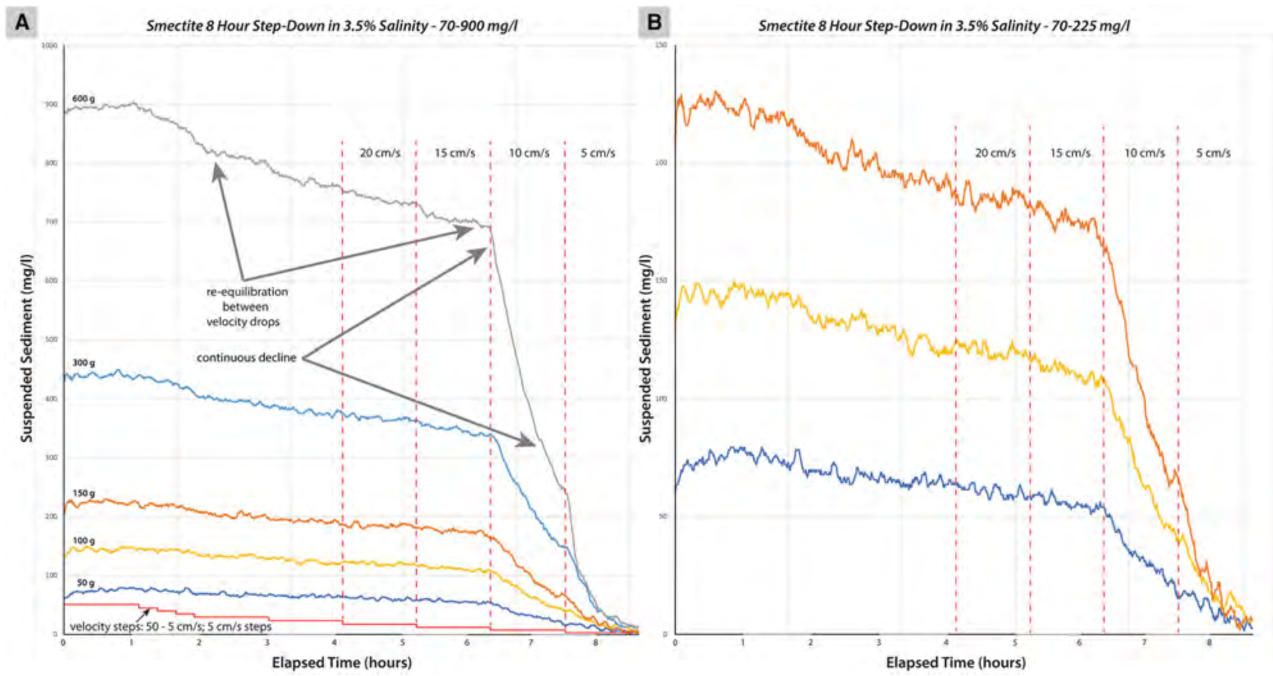


Fig. 10. Smectite 'Short Drop' experiments for 3.5% salinity (left), and at right, more detailed plots of the 77 through 231 mg/L experiments. Just like in the 'Long Drop' experiment (Fig. 6), there is a clearly developed drop of SSC associated with a continuous decline at the 10 cm/s velocity step. Whereas the freshwater experiments show a gradual decline while staying at comparatively high SSC levels, the saline experiments are marked by a rapid drop to low levels of SSC regardless of initial SSC. The drop curves for all initial SSC converge near the lower right corner of the diagram (10 to 20 mg/L), and the flows started to become transparent in the 30 min after the start of the 5 cm/s step.

DISCUSSION AND OUTLOOK

Factors affecting floc formation

The significance of these results is twofold. First, and most directly, they tell us that the mineralogy of flocculating materials does indeed influence the physical characteristics of the floccules and, by extension, the stability of floccules and how readily they can survive or grow in the basal portion of the flow in spite of elevated shear forces (Schieber *et al.*, 2023). Kaolinite bedload flocs form at higher flow velocities than illite (intermediate) and smectite (lowest).

From a perspective of colloid science, this is not unexpected. Floccule properties are known to vary due to the structure and distribution of surface charges, both parameters that are intimately linked to the physical makeup of the participating minerals. For example, the cation exchange capacity of kaolinite is lowest, that of illite is intermediate and that of smectite is highest (Lagaly, 2006). From a geometric perspective, the comparatively thick (and abundantly charged) edges of kaolinite particles cause flocculation in a sturdy house-of-cards arrangement (Van Olphen, 1977), whereas thinner illite flakes (Fig. 1) have less edge charge to offer and thus are likely to form weaker house-of-cards-type floccules (O'Brien, 1971). Smectite has the weakest floccules because of a smaller layer charge (Keren & Sparks, 1995) and clay particles that instead of platelet geometry resemble crumbled pieces of tissue paper (Fig. 2). This general concept of strong–intermediate–weak floccule strength of kaolinite, illite and smectite, respectively, also plays out empirically when the distribution of clay minerals is examined in river-to-shelf transects. In the latter, kaolinite is deposited most proximally, with illite intermediate, and smectite most abundant distally (Parham, 1966; Gibbs, 1983, 1985).

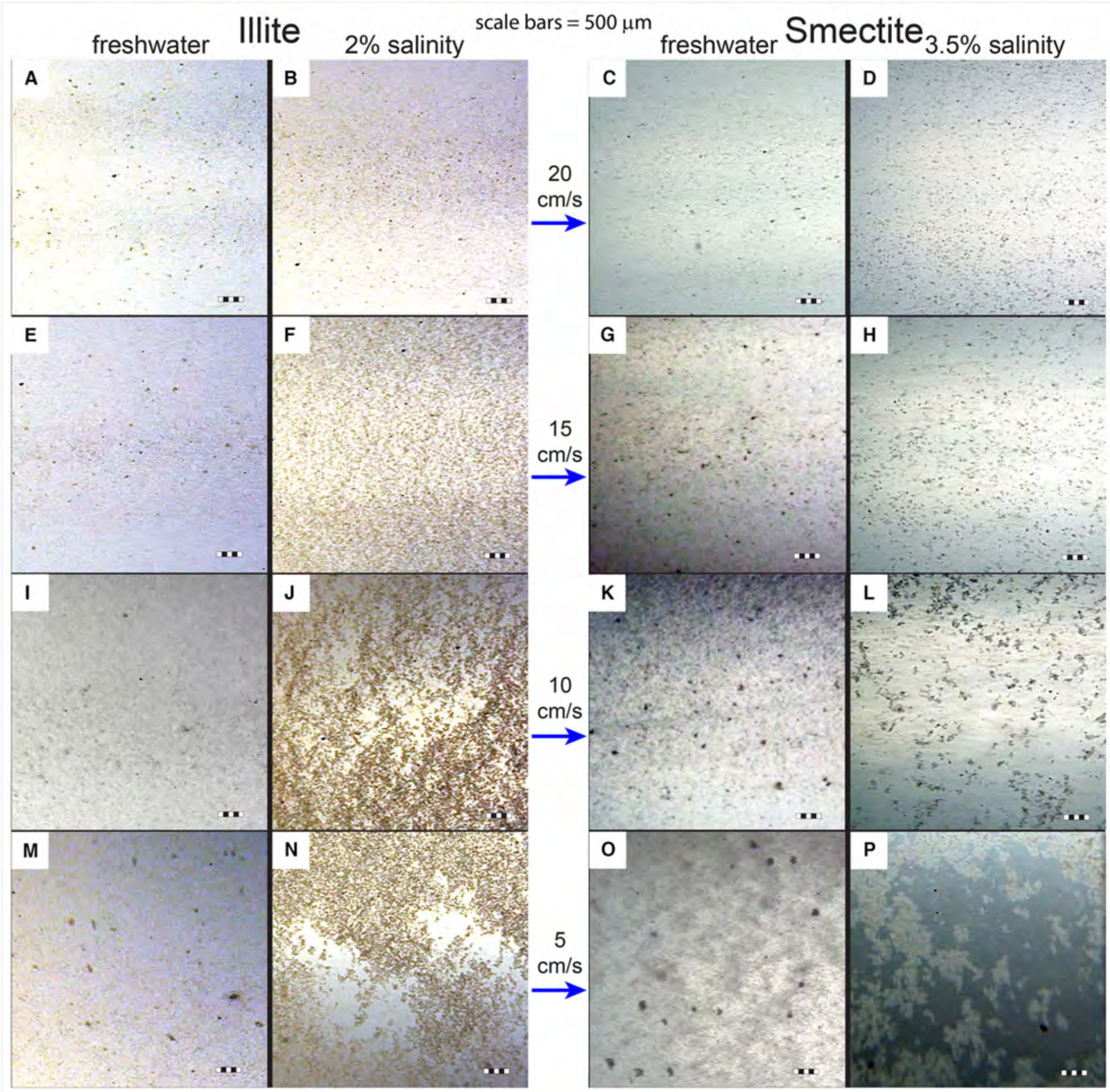


Fig. 11. High-resolution (7 µm/pixel) images of the flume bottom for illite (left) and smectite/Ca-montmorillonite (right) experiments in freshwater and 2% (illite) and 3.5% salinity (smectite) water. Only the velocity steps from 20 to 5 cm/s are shown, because higher velocity images are not materially different from the 20 cm/s images. The scale bar is 500 µm in every image, with 100 µm subdivisions. Flow direction is from left to right in each image.

Factors affecting mud accumulation

The above-observed mismatch between these new results and earlier experimental work done with the same flumes is the second significant aspect of this study. The observed differences (Fig. 6) are testament to the importance of mineralogy in flocculation processes, and the long observed convergence of CVS values of a range of fine-grained materials (clay and non-clay) towards a common value of around 20 to 25 cm/s points to the equalising influence of microbial slime (extracellular polymer substances, EPS) in flocculation processes.

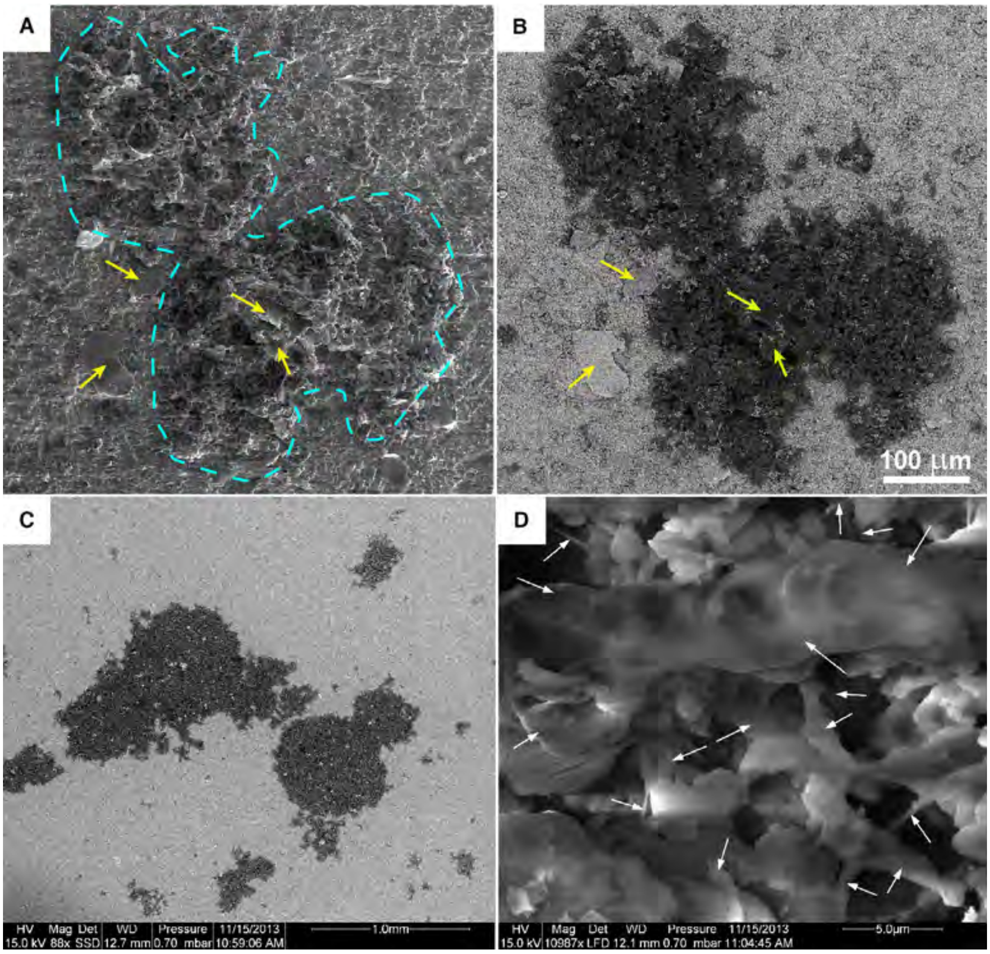


Fig. 12. Examples of organic matter associated with floccules from experiments. (A) Secondary electron image of a dried and collapsed floccule (marked by blue dashed line) on top of a roughed-up glass plate. Clay mineral flakes (yellow arrows) can be seen both outside and inside the floccule. (B) A backscatter electron image of the same floccule as in 'A'. The arrows point to the clay flakes marked in 'A'. Note that the clay flakes inside the floccule look much darker than those on the outside. The dark colour is due to low-density organic matter which permeates the floccule and coats mineral particles. (C) Backscatter electron image of several large floccules that dried out in a glass slide. (D) High magnification detail from these floccules (secondary electron image). Arrows point to likely organic filaments, as well as dried-out strings of extracellular mucus between mineral grains.

The mismatch was discovered during efforts to scale experiments to the typical duration (hours to days) of common sedimentation events (tidal cycles, river floods, storm surges, turbidites, etc.). Whereas the first-generation OBS sensors used at the time were not sensitive enough to capture the differences convincingly, when experiments were repeated with better sensors and precisely timed velocity drops, the differences between clay-mineral types became clear (this study and its precursor; Schieber *et al.*, 2023). Searching for the cause of the differences between short versus long duration experiments then led to the discovery of cryptic biofilms as the likely explanation.

On the bright side, however, this also means that many of the 'compromised' long duration earlier experiments conducted in the IU Shale Research Lab were, without intent, closer to natural conditions than originally anticipated, because microbial coatings are a common aspect of surface sediment particles (e.g. Aller, 1998; Ahmerkamp *et al.*, 2020). Although the original intent was to understand the processes active in clay and mud deposition from moving suspensions by starting the exploration with abiotic systems first (Schieber, 2011), so as to better understand the impact of organic-biological factors once organic matter and microbes were added, the aforementioned 'general' CVS of 25 cm/s may still be a useful parameter when evaluating flows under natural, biomediated conditions (e.g. Zeichner *et al.*, 2021). In our flumes, microbes were able to override mineralogy-imposed sedimentary thresholds in longer duration flume experiments (Fig. 12), in spite of added bleach at the start of the experiment, and in spite of having to live on a water and rock powder diet. It seems plausible, therefore, that biogenically driven aggregation/flocculation of muds was already exerting control over mud transport and deposition when microbes proliferated in the oceans and on land surfaces (Schopf & Packer, 1987; Djokic *et al.*, 2017; Baumgartner *et al.*, 2019) as far back as 3.5 bybp in the Archean.

We hypothesise that adding suitable quantities of natural biomass (e.g. marine snow; Schieber *et al.*, 2011) or organic coagulants to the mix has the realistic potential to further enhance floc formation and move CVS to even higher values than typically encountered in our experiments. There is likely an optimal proportion of coagulant for each type of additive, a topic that the IU Shale Research Lab team is actively exploring using the same experimental parameters as in the here-presented kaolinite, illite and smectite experiments. So far, preliminary results are as encouraging as they are confounding. Anybody expecting linear behaviour will be sorely disappointed. With an eye towards the broader goal of gaining a general understanding of mudstone deposition, it is worth remembering that 'mudstone' is defined by grain size (<63 μm), not mineralogy (Lazar *et al.*, 2015). Thus, mudstones do not need to contain clay-size particles or clay minerals (Schieber *et al.*, 2022). The obvious question then is how other fine-grained materials (silicate minerals most commonly) are deposited from clay-mineral-free flows. Will it be necessary to develop alternative sedimentological concepts for such flows? Or will fine-grain size and organic binders afford enough flocculation to make up for the lack of clays, such as to result in similar depositional parameters as observed in clay-bearing mudstones (Schieber, 2011)? A hint that the latter option may be viable is provided by experiments with fine-grained aragonite (Schieber *et al.*, 2013), where formation of bedload floccules, floccule ripples and bed accretion was observed at a CVS similar to that found for kaolinite. Interestingly, the aragonite experiments were conducted with material that had been exposed for 1 month to sodium hypochlorite (10% solution), as well as with material that still had all its original organic coatings; and yet, when added to flume experiments, both materials behaved in a comparable fashion. It should be an interesting exercise to examine whether other silicate minerals, when fine-grained, also show flocculation characteristics that mirror their structure and charge distribution, and will converge in their behaviour with those of clays when organic matter is added or biofilm growth is facilitated. Ultimately, experiments with mixtures of illite and smectite (ca. 60 : 40) with biofilms in saltwater might be most representative of the most common conditions of mudstone accumulation on Earth (Schieber *et al.*, 1998; Potter *et al.*, 2005).

Understanding these differences could even benefit the exploration of other planets and the search for extraterrestrial life. For example, if one were to observe lateral mineral segregation in Martian mudstones that consist of impact-pulverised silicates (Schieber *et al.*, 2022), it might be taken as an indicator that mineral physics dominated depositional patterns. Conversely, if different fine-grained minerals show little segregation along the transport path, it might indicate that organic binders helped to even out the differences due to mineralogy.

CONCLUSIONS

The data presented show clearly that in an abiotic setting, depositional parameters for clay minerals are strongly influenced by mineralogy (Table 1). With regard to deposition from moving suspensions, the CVS that coincides with the formation of sand-size bedload flocs, floc ripple migration and mud bed accretion, is highest for kaolinite (~25 cm/s), intermediate for illite (15 cm/s) and lowest for smectite (10 cm/s).

The development of organic polymers and biofilms, even when not visibly obvious, significantly changes the mineral-imposed depositional parameters. Organic coatings and binders tend to equalise the differences due to mineralogy and make muddy suspensions behave similarly in spite of mineralogical differences.

Whereas understanding of depositional parameters for clay-bearing muds is based on insights from colloid science and flume studies, the same cannot be said at the moment for muds that consist primarily of fine-grained non-clay-mineral materials. However, less of an issue on Earth, where clay minerals tend to be ubiquitous, for understanding mudstones on other planets, experimental exploration of the deposition (from moving suspensions) of fine-grained non-clay materials might be beneficial. It could on one hand allow refinement of the interpretation of mudstone successions that are devoid of clay-size particles or clay minerals, and also provide clues with regard to the possibility of past microbial life.

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DATA AVAILABILITY STATEMENT

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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Supporting Information

Additional information may be found in the online version of this article:

Data S1. Methods.

Data S2. 2% salinity 'Short Drops' for illite and smectite.

Data S3. Bedload particle/floccule sizes from 50 to 5 cm/s.