

Effect of the concentration of NaCl and cylinder height on the sedimentation of flocculated suspension of Na-montmorillonite in the semi-dilute regime

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Abstract

In this study, we focused on the effect of the degree of floc growth on the moving velocity of the interface between sediment of flocculated montmorillonite and supernatant in the semi-dilute suspension, which is characterized by the start of an extremely slow movement of interface followed by an abrupt settling and ending in consolidation. Na-montmorillonite suspension coagulated under different ionic strengths, ranging from 0.5 M to 1.5 M of NaCl, was placed in a settling cylinder. The initial height of suspension was varied from 13 cm to 50 cm. The flocculated suspension was left to settle in the cylinder after the manual mixing of end-over-end. Changes in the height of the interface between the flocculated sediment and the transparent supernatant were measured as a function of elapsed time. It was confirmed that the maximum settling velocity increased with an increase in the height of cylinder within the range of our measurement. This tendency was found to be more significantly pronounced by the growth of flocs. This result indicates the presence of a feed-forward mechanism to enhance the upward motion of fluid or downward motion of flocculated sediment, or both. That means, sedimentation is accelerated by the growth of big flocs, and the growth of big flocs is accelerated by the sedimentation. These motions will eventually induce the generation of an upward plume or channel of water. The formed plume flutters slowly with rather a large scale. We term this phenomenon the “sedimentation turbulence.”

Na-montmorillonite, Sedimentation, Semi-dilute suspension, Cylinder height.

Sedimentation turbulence

34 **Introduction**

35 Colloidal substances, soil, and suspended solids in water are ubiquitous in an aquatic environment.
36 These substances can often be found in a flocculated state. Despite its ubiquitous presence and
37 importance to transportation in the aquatic environment, relatively little attention has been paid to
38 the sedimentation behavior of flocculated suspension. In paddy farming, this phenomenon can be
39 commonly found in the processes of puddling (Adachi 2019). Previous works have reported the
40 discharge of high-turbidity water into river water during or after puddling (Wakai et al. 2005; Sudo
41 et al. 2009). Fine sediment discharged from the paddy surface by excess water moving into the
42 river causes an increase of water turbidity in the downstream basin. Meanwhile, in the process of
43 irrigation, cohesive sediment tends to accumulate tentatively in the still-water part of irrigation and
44 drainage channels. But such tentative sediment can be easily flushed out by the change of flow rate
45 in the canal. In the puddling season, the river water will be polluted with chemical substances
46 originating from fertilizers and other agrochemicals, which are sorbed to the surface of clay
47 particles composing the flocculated sediment. Such contaminants can potentially endanger the
48 entire ecosystem (Winterwerp and Kestern 2004). In the natural aquatic environment, the majority
49 of fine particles are present in a flocculated form. Therefore, flocs can be regarded as a very
50 important unit of transportation as compared to individual particles, and flocculated material will
51 influence a wide array of environmental phenomena. The sedimentation process of flocculated
52 materials under gravity can be regarded as the most fundamental issue that is yet to be clarified in
53 the transportation process of chemical substances via water.

54 **Sedimentation behavior**

55 A framework of the theory of sedimentation was worked out by Kynch (1952). The theory has
56 been considered assuming mainly non-colloidal materials (Davis and Acrivos 1985). Imai (1981)
57 classified the sedimentation behavior of coastal sediment, which has the natural tendency to
58 flocculate, into three different regimes as a function of the suspension concentration. They are,
59 dilute, semi-dilute, and concentrated regimes. In the dilute regime, the formed flocs settle freely.
60 Since larger flocs settle faster and smaller flocs settles slower, smaller flocs are apt to remain in
61 the upper space. With such situation, the boundary between the sediment and the supernatant
62 becomes unclear. By contrast, in the semi-dilute regime, mutual interactions among the flocs will
63 form network-like structure and the settling process effectively induce an idle time before the flocs
64 start to settle. The difference between the dilute and the semi-dilute regime is the appearance of a
65 clear boundary between the sediment zone and supernatant zone. This is probably because all the
66 smaller flocs will be captured by the network-like structure formed by other bigger flocs. That is,
67 the network-like structure essentially acts as the filter of all small flocs. This means that in the
68 semi-dilute regime, flocs in the sediment appear to interact to form a phase of sedimentation that
69 has a clear interface with the transparent and supernatant zone. In the concentrated regime, an

70 initially formed network structure of flocculated sediment settles very slowly under its own
71 weight. In the present study, we focused on the second regime.

72 The progress of sedimentation in a semi-dilute regime can be further characterized into three
73 stages. They are, starting with an initial flocculation stage, followed by a rapid sedimentation
74 stage, and ended by a very slow consolidation stage. Michaels and Bolger (1962) presented an
75 analytical method of hindering sedimentation of flocculated kaolinite using the result of fluidized
76 bed reported by Richardson and Zaki (1954) in which we need to assume some representative size
77 of flocs. Although their data of the experiment showed a tendency for the maximum rate to
78 affected by the height of the suspension, they did not mention anything on this phenomenon. To
79 understand the sedimentation process of flocculated suspension, our insight in the present study is
80 that it is necessary to pay attention to the factor of floc growth during sedimentation.

81 In the present study, we have chosen a system of the coagulated suspension of Na-
82 montmorillonite, with which extremely big flocs can be formed simply by the increase of the
83 concentration of NaCl (Adachi et al. 2019). This characteristic has been regarded as an ideal one
84 for studying the physical properties of flocculated suspension (Wu and Adachi 2016; Miyahara et
85 al. 2002; Kobayashi and Adachi 2008). Moreover, montmorillonite can be regarded as an
86 important clay mineral for industrial, agricultural, and environmental processes, and many aspects
87 of this clay remain to be studied (Uddin 2008). In our previous study, we highlighted the
88 relationship between microscopic heterogeneous interaction of clay particles and macroscopic
89 behavior of Na-montmorillonite sedimentation. We have confirmed that the increase of ionic
90 strength reduces the duration of the initial flocculation stage denoted as τ . The onset of collapse
91 appears to be escalated by the generation of small cracks in the weakly connected sediment
92 structure. This moment was regarded as the characteristic point corresponding to a certain degree
93 of flocculation at which the formed gel network of flocs is collapsed by its self-weight. On the
94 basis of this picture, we can simply conclude that τ is the time required to reach this certain degree
95 of flocculation (Wu and Adachi 2018). The duration of the initial flocculation stage becomes
96 smaller if the rate of flocculation is increased. This interpretation was consistent with the colloidal
97 stability analysis of Tombácz et al. (1988), who pointed out the dominance of edge-face
98 interaction detected as the difference in critical coagulation concentration, which appeared in a
99 lower pH under lower ionic strength. Our result in the previous study for the difference in τ also
100 correlated with this trend.

101 In the present study, we focus on the settling velocity of the second stage. We measured the
102 location of interface between sediment and supernatant as a function of elapsed time. When the
103 concentration of sodium chloride exceeds 0.5 M, the montmorillonite suspension is known to form
104 big flocs irrespective of pH value (Miyahara et al. 1998). This is suitable for naked-eye
105 observation. With this system, the behavior of maximum velocity of the second stage can be
106 intensively analyzed by changing the height of cylinder and the concentration of NaCl.

107 **Materials and methods**

108 **Montmorillonite suspension preparation**

109 The following procedure was applied to prepare well-characterized samples: 1) Commercially
110 available Kunipia-F (Kunimine Co., Ltd.) was dipped in a solution of 1.0 M NaCl to exchange
111 unknown adsorbed cations with sodium ions. The purity of the montmorillonite content of this
112 commercial product was confirmed by the courtesy of Agroenvironmental Research Institute, using
113 X-ray diffraction (MiniFlex, Rigaku Co., Ltd.). Based on that result, Sueto and Nakaishi (1992)
114 reported that this commercial product is typically pure montmorillonite and confirmed the
115 chemical composition of this clay supplied by the company (Kunimine Co., Ltd.). The purity of
116 the same product was also reported by Nanzyo and Kanno (2018). 2) The dispersion saturated with
117 NaCl was rinsed using the dialysis process with distilled water repeatedly. 3) The rinsing process
118 through dialysis was conducted until the ionic concentration of the resulted supernatant reaches
119 lower than 10^{-5} M measured by a conductivity meter (LAQUA pH/Ion/Cond. Meter F-74BW,
120 Horiba Scientific Co., Ltd.). 4) After dialysis, the original stock suspension was prepared. The
121 solid volume fraction of montmorillonite suspension was measured by drying out the water at 105
122 °C in the oven for one day. 5) The prepared dispersion of Na-montmorillonite was mixed with the
123 NaCl solution. For the mixing procedure, the end-over-end mixing method was used; whereby the
124 term indicates a simple mixing method of two liquids contained in two identical cylinders. In the
125 beginning, the two cylinders were filled with the same amount of liquid of suspension (A) and the
126 NaCl solution (B). Then, the entire liquid contained in one cylinder is poured into the other
127 smoothly. The solution in cylinder B was poured into cylinder A within 10 seconds. Instantly, the
128 solution in cylinder A was poured into cylinder B again within 10 seconds. The mixing procedure
129 was repeated three times and the glass cylinders were filled with the flocculated suspension up to
130 the required initial height.

131 **Measurement of maximum settling velocity**

132 The relationship between clear interface boundary and the elapsed time was established at room
133 temperature ($20 \pm 1^\circ\text{C}$). The solid volume fraction of montmorillonite suspension was 2.0×10^{-4} .
134 The NaCl concentration used in this study was adjusted to 0.5 M, 0.75 M, 1.0 M, 1.25 M, and 1.5
135 M. The height of settling cylinder used in this study were 13 cm, 20 cm, 25 cm, 35 cm, and 50 cm.
136 The flocculated suspensions were left to settle in the settling cylinder after the manual mixing
137 method of the end-over-end (Fig. 1). The experiment was conducted in triplicate and the
138 sedimentation behavior was delineated by descriptive analysis. Changes in the height of interface
139 between the flocculated sediment and the transparent supernatant for different levels of ionic
140 strength were measured as a function of elapsed time.

141 **Results**

142 The temporal variation of the interface between the supernatant zone and the sediment zone of
143 flocculated Na-montmorillonite are plotted in Fig. 2a-e for different concentrations of NaCl and
144 for each different cylinder heights. As demonstrated in the figure, all plotted data demonstrate the
145 pattern of inverse S-curve. This tendency is pronounced for the cases of higher cylinder and higher
146 concentration of NaCl. For the case of lower concentration of NaCl (0.5 M and 0.75 M), an initial
147 latency period was observed. This is consistent with the results reported previously (Wu and
148 Adachi 2018). The dependency of the maximum rate of sedimentation on the height of cylinder
149 confirmed our previous result obtained for the case of NaCl 1.0 M (Argo et al. 2016). But, here,
150 we confirmed the tendency for a wider range of NaCl concentration.

151 In Fig. 3, all of the obtained maximum settling velocities are plotted against the height of
152 cylinder. It is interesting to note that the acceleration of maximum velocity as a function of
153 cylinder height is obviously more pronounced for the case of higher concentration of NaCl.

154

155 **Discussion**

156 **Maximum velocity as a function of the cylinder height and the** 157 **concentration of NaCl**

158 According to Nakaishi et al. (2012), the difference of moving velocity during the initial stage
159 against cylinder diameter was ascribed to the drag at cylinder wall. While in the second stage, they
160 reported almost the same value of maximum settling velocity. That is, the value of maximum
161 velocity is not influenced by the diameter of cylinder. Our preliminary study also confirmed that
162 the settling velocity of aggregated Na-montmorillonite flocs in cylinders with diameters of 4.0 cm
163 and 2.0 cm showed no clear difference (Argo et al. 2016). Therefore, we can conclude that the
164 maximum velocity in the second stage increases with an increase of cylinder height and secondly,
165 this increase is much more enhanced by the increase in the concentration of NaCl.

166 Our observation of the sediment morphology confirmed that the sediment was coarser and less
167 homogeneous at higher salt concentration. This are notable characteristics of flocculated
168 montmorillonite coagulated with NaCl (Miyahara et al. 1998; Wu and Adachi 2016; Adachi et al.
169 2019). At lower ionic strength, flocs are obviously smaller, and the appearance of the cake is much
170 homogeneous. Miyahara et al. (1998) pointed out that Na-montmorillonite suspension is known to
171 form big flocs in an extremely high concentration of NaCl. This ensures that visibly large
172 montmorillonite flocs are formed to enhance the rate of sedimentation. If we can accept the reason
173 for the enhancement of maximum settling velocity is due to the formation of bigger flocs, the
174 reason for enhancement of the maximum settling velocity by the increment in cylinder height can
175 be ascribed to the development of bigger flocs during sedimentation. That is, the higher the
176 cylinder, flocs will have more chances to grow by traveling longer distances. This is the reason
177 that the maximum velocity is enhanced by the increase in the cylinder height and by the increase
178 of the concentration of NaCl.

179 Changing the concentration of NaCl will affect the macroscopic behavior, which in turn will
180 determine the coagulation behavior. Gibbs (1983) confirmed that increasing salinity will increase
181 the collision efficiency owing to the decrease in double-layer thickness and surface charge.
182 Increasing salt concentration decreases the repulsive electrostatic effect, breaking weak edge–face
183 bonds and residual strong edge–face bonds, and results in bigger flocs (Wu and Adachi 2016).
184 However, more important reason for the development of bigger floc in the sediment is the
185 remarkable increase of floc strength in the region of extremely high concentration of NaCl which
186 is clarified in our recent result on the floc strength of Na-montmorillonite studied in a well-defined
187 flow (Adachi et al. 2019). That is, if the floc strength is strong, it is more probable that big flocs
188 formed during the sedimentation stand for the break-up.

189 **Feed-forward mechanism enhancing sedimentation process**

190 Based on the result of the sedimentation behavior and naked eye observation, we summarize the
191 scenario of sedimentation of flocculated material in the cylinder as follows. Initially, during the
192 flocculation stage, colloidal flocs are considered to grow gradually to form a loose network
193 (Adachi et al. 2019) (Fig. 4). It is regarded as waiting time before the gel collapse (Fig. 6a-c).
194 Consequently, as the loose cake that is formed starts to collapse, the rapid sedimentation stage sets
195 in (Fig. 6d-e). During this stage, the bigger flocs (i.e., those formed in high salt concentration) will
196 settle down faster than the smaller ones. Owing to the velocity difference, the collision of settling
197 flocs will take place; this leads to the development of the flocs. In the case of higher cylinders, the
198 distance through which flocs fall becomes longer. The falling flocs will then have more chance to
199 collide with other small flocs and increase in size and mass, thereby accelerating their settling
200 speed. Consequently, the rapid sedimentation commences at a different period (Imai 1981; Dobias
201 et al. 1993). The flocs settle differently in terms of velocity, owing to their different size and
202 density, to fill the void formed from channeling, which then creates the counter-flow that is
203 visually observed to be similar to a small turbulent flow (Fig. 6e). It can be characterized as
204 an unstable, irregular, seemingly random, and chaotic flow, and it certainly causes unpredictable
205 movement of flocs (Pope 2000). We term this phenomenon the “sedimentation turbulence.” This
206 phenomenon is not well described in the previous study by Michaels and Bolger (1962) or
207 elsewhere. The flocs motion induces turbulent flow passing through in disordered flows, which
208 helps to rearrange the formation of the floc. The flocs formation is due to turbulent flow passing
209 through, which increases the settling velocity as the hydrodynamic drags and adds mass affected.
210 Consequently, the flocs collide with neighboring flocs, and they are rearranged to form bigger
211 flocs. This feed-forward mechanism continues until the flocs hinder each other and enter the
212 consolidation stage (Fig. 6f). The flocs settle and move further to fill the space left underneath to
213 develop early consolidation at the bottom of the cylinder (Winterwerp and Kestern 2004; Imai
214 1981). It is noticeable that the sediment at the bottom of the cylinder becomes denser and more
215 compact as the void ratio decreases over time (Fig. 5). During this final stage, the flocs settle
216 slowly owing to their self-weight reflecting a slow sedimentation rate. To illustrate the overall
217 progress of sedimentation as well as the sedimentation turbulence phenomenon, we have improved
218 the schematic diagram by Wu and Adachi (2016) after a careful observation (Fig. 6).

219 Conclusion

220 The study presented here shows the effect of cylinder height on the sedimentation behavior of
221 flocculated Na-montmorillonite in the semi-dilute regime. Our results indicate that the maximum
222 rate of sedimentation increases steadily as a function of cylinder height. The sedimentation
223 behavior of flocs is dependent on floc size, which in turn is controlled by salt concentration and
224 collision rate between flocs. Our findings suggest that the size of the flocs increases owing to
225 prolonged traveling distance in higher cylinders, coupled with the sedimentation turbulence that
226 emerges during the gel collapse. Increasing cylinder height will allow longer traveling distance
227 and increase the chances of aggregation of falling flocs, and thus enhance the sedimentation. The
228 upward motion of fluid or downward motion of flocculated sediment, or both, is enhanced by the
229 feed-forward mechanism, which creates sedimentation turbulence. Sedimentation is accelerated by
230 the growth of big flocs, and the growth of big flocs is accelerated by the sedimentation. Additional
231 experiments on the sedimentation behavior of Na-montmorillonite using flow visualization
232 techniques will further elucidate this sedimentation turbulence phenomenon.

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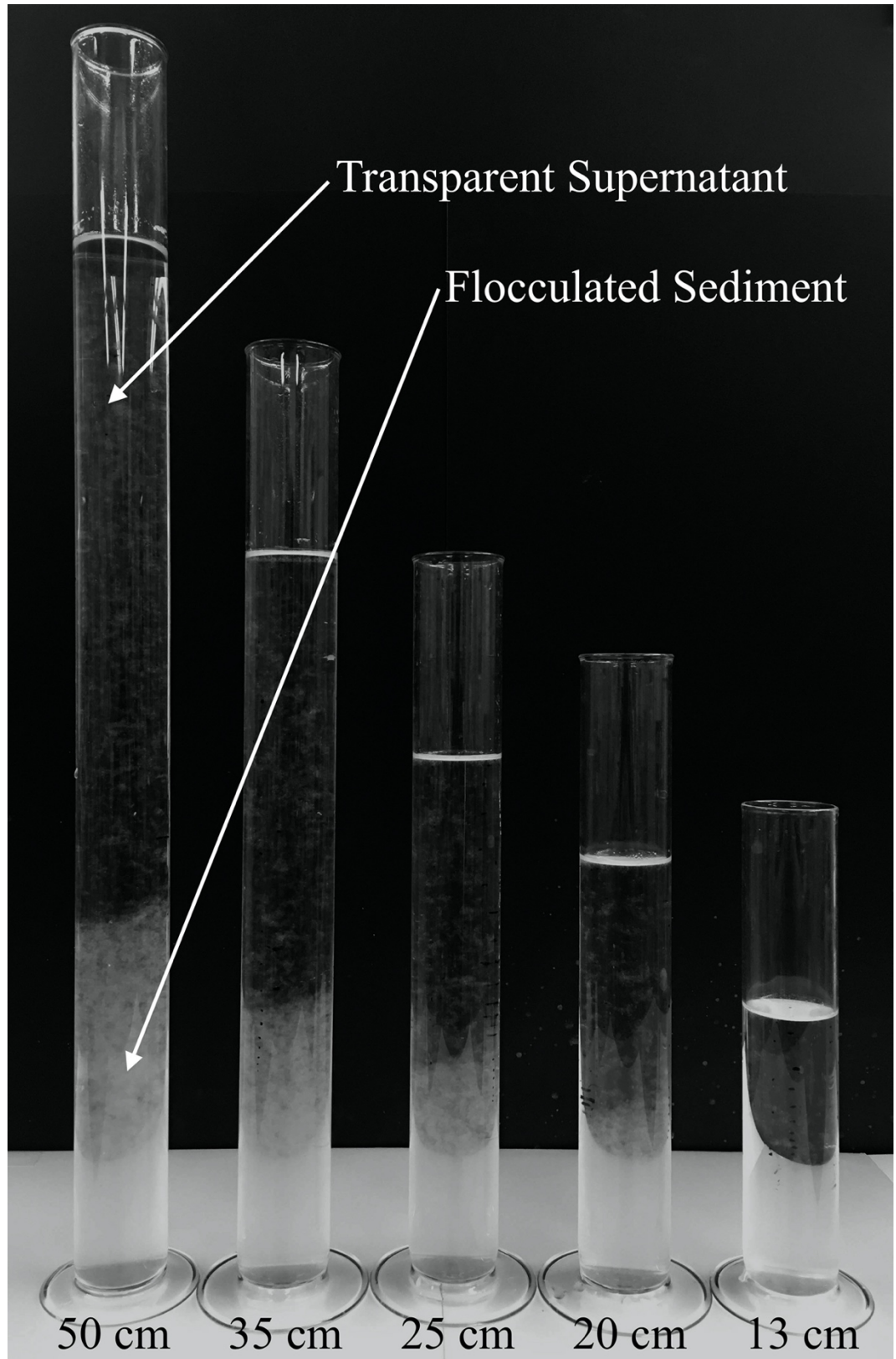
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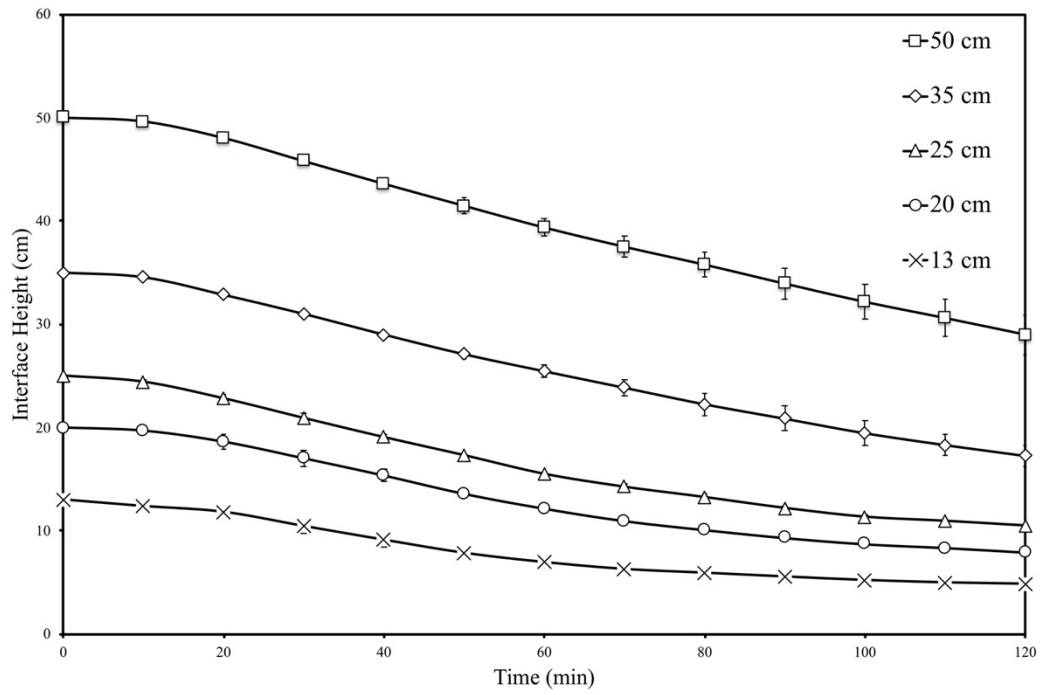
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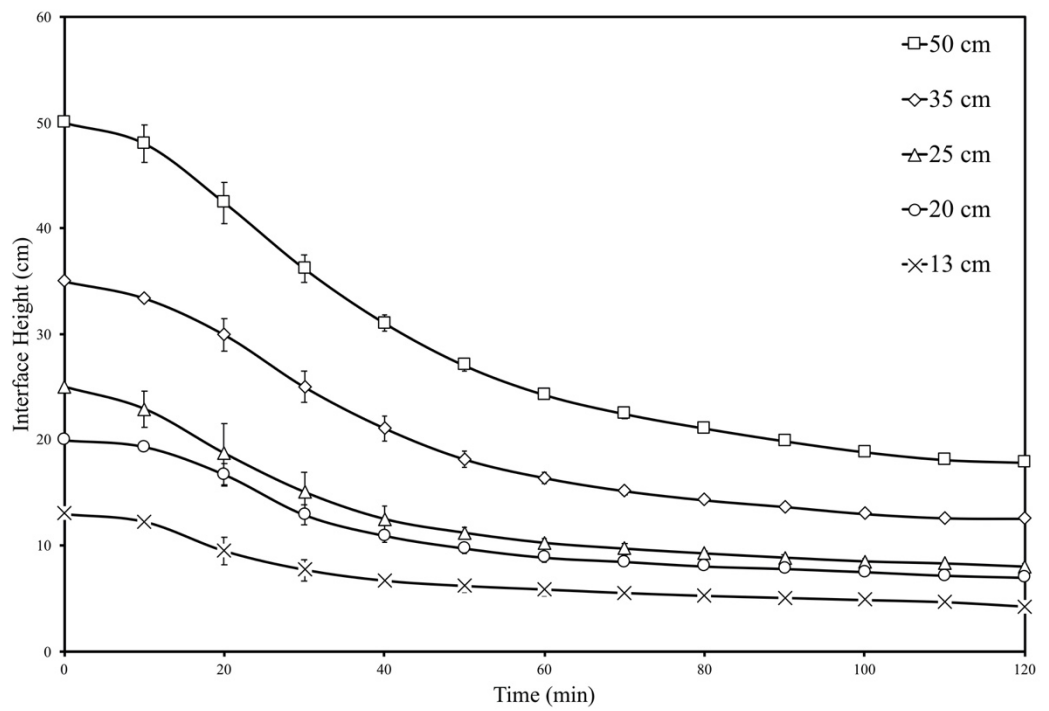
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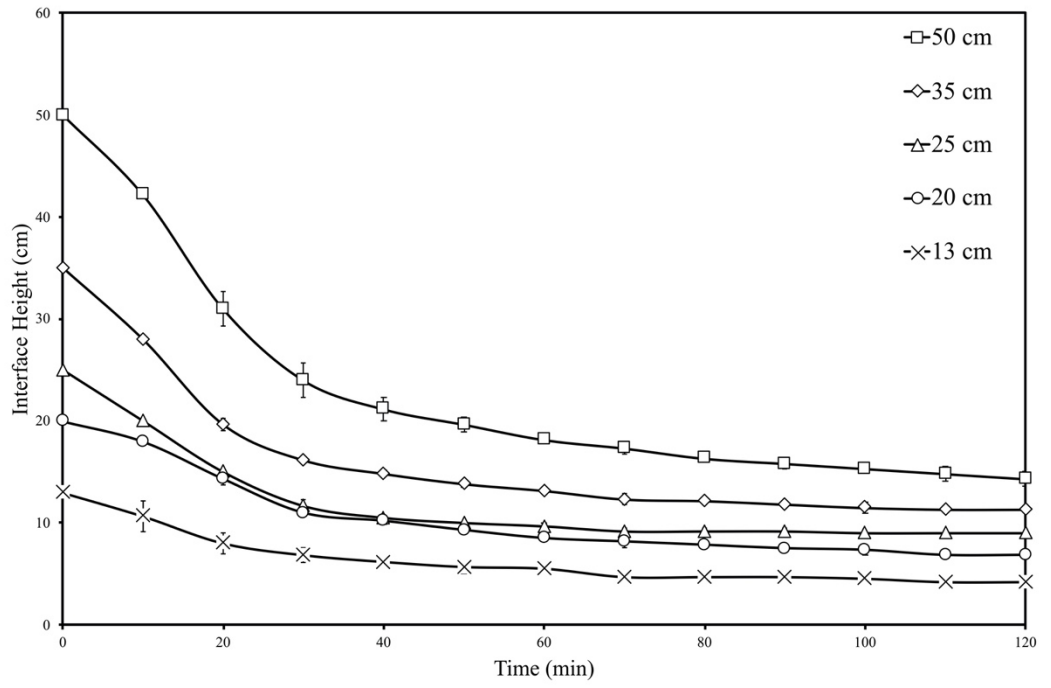
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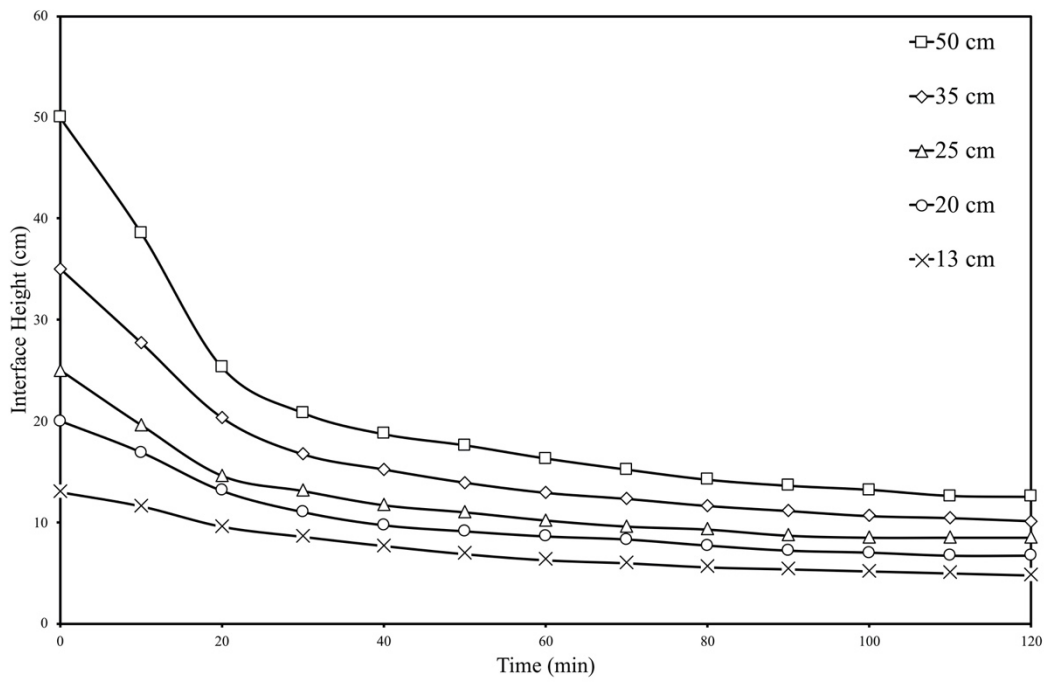
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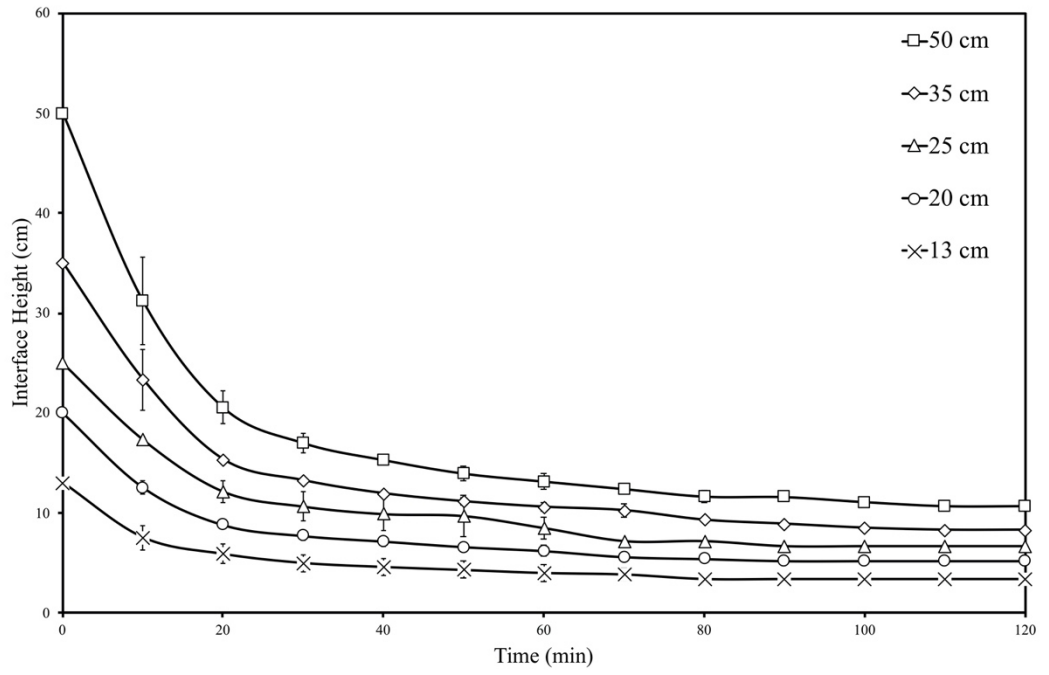
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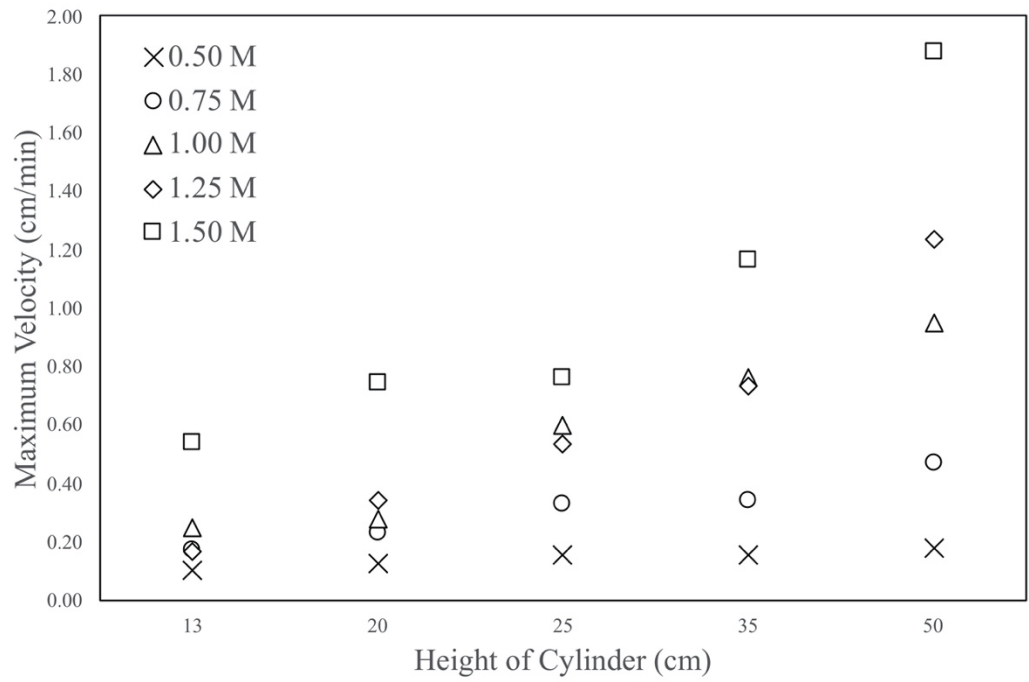


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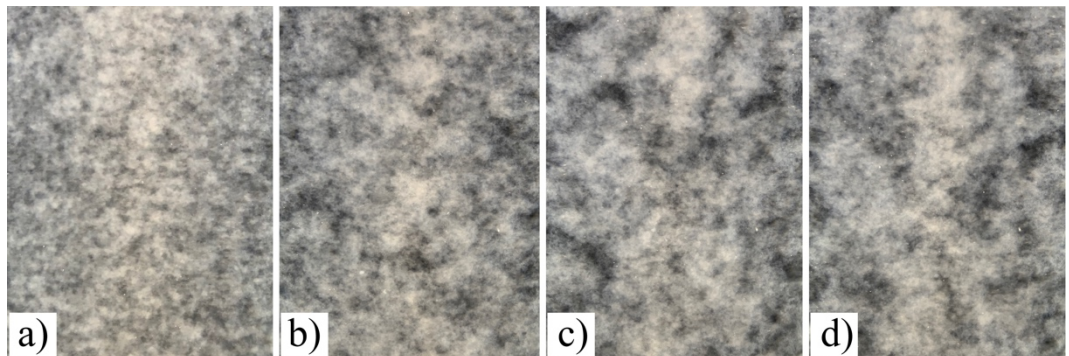
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Figure 3



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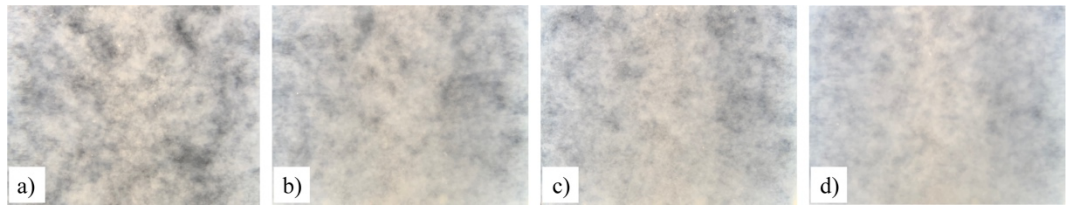
Figure 4



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Figure 5

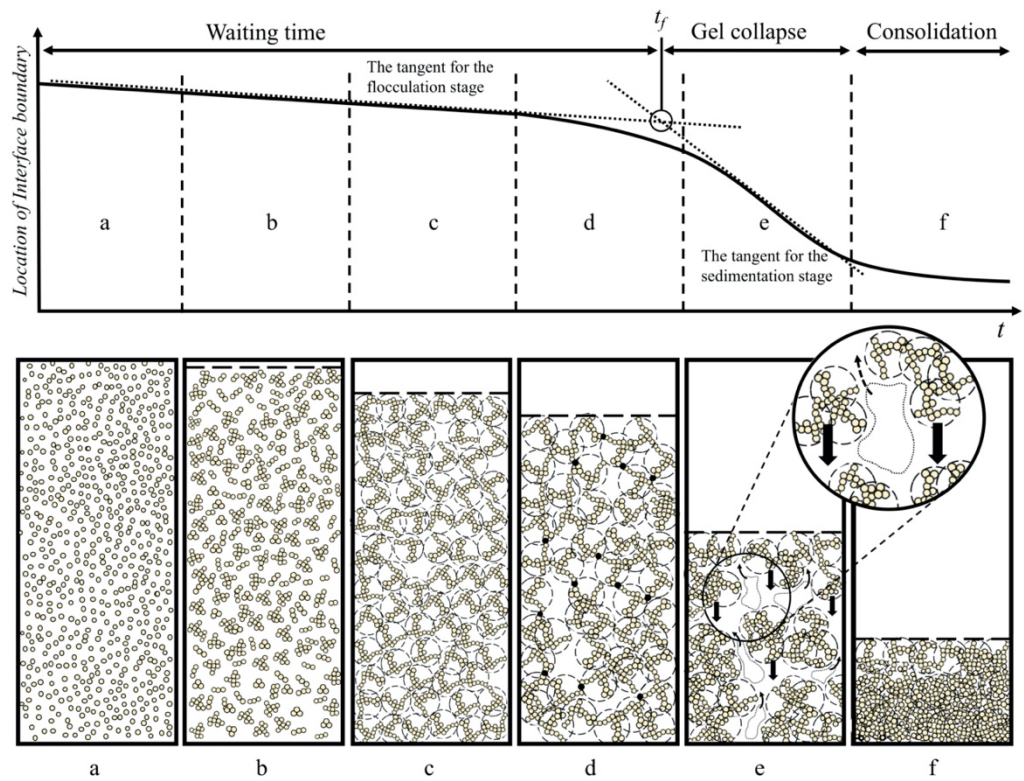


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Figure 6



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Figure 1	Clear interface boundary between flocculated sediment and the transparent supernatant in different cylinder height
Figure 2	Interface height as function of elapsed time for different levels of ionic strength, with error bars representing the standard deviations; a) 0.5 M, b) 0.75 M, c) 1.0 M, d) 1.25 M and e) 1.5 M
Figure 3	Maximum velocity as function of cylinder height at different levels of ionic strength
Figure 4	Flocculation and settling stage of Na-Montmorillonite at 20 cm of cylinder height over time; a) 1 minute, b) 5 minutes, c) 9 minutes and d) 12 minutes
Figure 5	Consolidation stage of Na-Montmorillonite at bottom of 15 cm cylinder over time; a) 14 minutes, b) 16 minutes, c) 30 minutes and d) 42 minutes
Figure 6	<p>Schematic representation explaining the progress of sedimentation of flocculated materials in the semi-dilute regime.</p> <ul style="list-style-type: none"> a) Just after mixing, uniform dispersion is formed. b) Small flocs are formed and suspended independently. At this stage, we can already identify the clear interface boundary between the flocculated sediment and the transparent supernatant. It starts to settle down slowly. The rate of sedimentation is very small reflecting the small size of flocs. During this stage, the flocculation process is ongoing. c) The space-filling structure of sediment is formed. d) At some contacting points in the network (marked as black circles), the stress is concentrated due to self-weight of the sediment. The point will resist against the compression by self-weight of the network. Due to this resistance, the apparent sedimentation rate is essentially low. Once the network could no longer yield the stress, the network starts to break down and mark the onset of gel collapse. The intersection point for tangents of linear parts between the flocculation stage and sedimentation stage with maximum sedimentation rate is denoted as the flocculation time, t_f. e) Abrupt gel collapse during this stage initiate the rapid sedimentation stage. Development of crack void while flocs fall down during gel collapse created the water channelling in the network. Downward motion of flocculated sediment (represented by big arrows) will induce the generation of upward plume of water (represented by small arrows) which is a bit fluttering. In the high cylinder, the traveling distance of falling flocs become

	<p>longer. The development of flocs along the long traveling distance coupled with the sedimentation turbulence creates a feed-forward mechanism which further enhanced the sedimentation process.</p> <p>f) Flocs are accumulated at the bottom and consolidate very slowly.</p>
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