



Investigating the settling behaviour of saline tailing suspensions using kaolinite, bentonite, and illite clay minerals

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ABSTRACT

This study aimed to understand the dewatering behaviour of three types of clay minerals (i.e. kaolinite, bentonite, and illite) in saline water, and in particular the effect of factors including clay mineralogy, clay suspended solid concentration, water salinity, and polymer flocculant dosage on dewatering behaviour. It was demonstrated that the addition of polymer flocculants helped all three clay types to settle due to the polymer bridging effect. In saline water, this bridging effect worked best above a certain threshold level for illite but tailed off for the other two clay types. As well, high-salinity water was observed to assist bentonite and illite to settle faster but to hinder the settling rate of kaolinite. In addition to inter-particle forces, the clay aggregate structures formed through the addition of the flocculants were identified to play an important role in determining the dewatering behaviour of clay minerals in saline water. Cryo-scanning electron microscopy (SEM) identified that, depending on the clay type, the floc structures in saline water were either dense (water-excluding) or loose (water-trapping). Dense flocs were observed to settle more quickly than loose flocs. This study has practical applications in providing guidance for the processing of complex tailings containing clay minerals in saline water.

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1. Introduction

Clay minerals are ubiquitously present in most mine tailings. They create widespread problems in transport of tailings slurries and affect the long-term stability of tailings storage facilities [1]. Fine particle size, surface chemistry, and the internal structure of clay minerals affect water-holding capacity, and the ease of transport or rheology and viscosity of tailings slurry. They also affect the capacity of tailings to release or capture contaminants. Clay minerals are anisotropic phyllosilicates which comprise one alumina octahedral (O) sheet and one or two silica tetrahedral (T) sheets that fit together to form a basic repeating unit of either 1:1 (TO) or 2:1 (TOT) layers [2]. They usually have a complex surface chemistry arising from the heterogeneity of the charged edges and faces of these layers. The basal faces carry a permanent pH-independent negative charge, while the edges possess a pH-dependent charge [3]. Owing to the different basal/edge surface characteristics, clay particles can aggregate to form edge-edge (EE), edge-face (EF), and face-face (FF) aggregate structures [4].

The flocculation and thickening of fine clay particles have been extensively studied both at the laboratory and industrial scale to enhance the efficiency of solid-liquid separation [5–10]. However, to date, there have been limited economical and practical methods for recovering process water from these tailings for further use [11,12].

Poor solid-liquid separation arising from the presence of clay minerals continues to impose high operational and capital costs [13].

The increasing use of saline water has made the tailing management situation even more complicated. In some mines, for example coal mines, salts are present in the sedimentary geology or are generated by weathering processes which cause the process water to be saline. Also, many mine sites, particularly those in water-sensitive environments, are introducing new sources of water for processing purposes including saline groundwater and seawater. The use of saline water may negatively (or positively) affect the settling behaviour of clay particles in the tailings and therefore the turbidity of the recycled water. However, to date, research has focused mainly on the dewatering behaviour of tailings in fresh water. This means that the settling rate of saline tailings remains poorly understood.

In the absence of polymer flocculants, Nasser and James [14] found that, in acidic pH conditions (e.g. pH 2), kaolinite particles settle in flocculated form regardless of electrolyte concentration. This is due to the strong oppositely-charged electrostatic interactions of basal and edge planes. However, in basic pH ranges, the particles settle in both dispersed form (in 0.001 M NaCl electrolyte concentration) and flocculated form (in 0.1 and 1 M NaCl electrolyte concentration). Gorakhki and Bareither [15] evaluated the effect of salinity and cation valence on the sedimentation behaviour of kaolinite, bentonite, and soda ash mine tailings without addition of polymers and suggested that an increase in salinity led to higher sedimentation rates and final solids contents for bentonite. In contrast, a salt concentration of 100 mM

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was identified as a critical concentration whereupon the settling rate of kaolinite decreases regardless of salt type. This decrease was attributed to an increase in sedimentation fluid density and viscosity.

On the other hand, other studies reported that when polymer was present, it may interact with saline water so as to affect the dewatering of clay minerals. Siffert and Bocquet [16] investigated the adsorption of partially hydrolyzed polyacrylamides onto kaolinite, and found that adsorption was higher in a strongly saline medium than in pure water due to a change in polymer conformation. They found that in a CaCl_2 medium, the adsorption was more significant than in a NaCl medium. In addition, Mpofu et al. [8] studied the effect of hydrolysable ions Mn^{2+} and Ca^{2+} in combination with anionic polyacrylamide flocculant on the surface chemistry, particle interactions, and dewatering behaviour of kaolinite. They found that the specific adsorption of hydrolyzed metal ions led to an increased settling rate at pH 7.5 and 10.5. Ji et al. [17] studied the settling behaviour of coal tailings in the presence of three polymer flocculants, non-ionic polyacrylamide (PAM), anionic Magnafloc 1011 (MF) and cationic $\text{Al}(\text{OH})_3$ -polyacrylamide (Al-PAM) in both fresh water and saline solution. They identified that the settling of tailings depended significantly on solution salinity, as well as flocculant type and dosage. MF showed the best settling performance among the three polymers in both fresh water and saline solutions.

Based on previous studies, it is expected that saline water may interact with clay minerals, making commonly-used flocculants less effective and leading to a different dewatering performance in dewatering of clay-rich tailings. As clay tailings are increasingly treated in saline water [17–20], there is an increased need to understand the dewatering behaviour of saline clay-rich tailings. The dewatering behaviour of clay minerals in saline water is of importance to both fundamental understanding and industry practice.

The structure of clay aggregates plays an important role in dewatering of tailings. Although extensive studies have examined how to improve the recovery of water from mineral tailings, low settling rates and consolidated waste slurries with a low solid density are commonly observed in thickeners [21]. While an increased floc size can increase the settling rate, compact consolidation of the resulting sediment can be diminished if the flocs are not also dense in structure [5]. Mpofu et al. [7] studied the effect of polymer structure type on flocculation, interfacial chemistry, particle interactions and dewatering behaviour of smectite dispersions in fresh water. According to Mpofu et al. [7], compared to PAM, polyethylene oxide (PEO) produces better inter-particle bridging, a higher settling rate and higher floc compaction behaviour upon shear. For PAM, the hydrogen bonding interactions appear to be weakened as a consequence of electrostatic repulsion between the polymer's COO^- pendant groups and the negatively charged clay particle surfaces. This, in conjunction with possible steric hindrance due to PAM polymer chain branching, leads to the formation of loose, open and fragile flocs and therefore a slower settling rate.

It is interesting to note that flocs structures may be affected by external parameters, such as water salinity. It was found that high-salinity water can influence the efficiency of polymeric flocculation of clay minerals, leading to a different solid-liquid separation performance compared to fresh water [8,17,20,22]. This is probably because the presence of salts alters the interaction between flocculants and clay particles leading to different clay aggregate structures. It is therefore posited that saline water may cause clay minerals to aggregate differently, in ways which release or entrap water, affecting their dewatering performance.

In this study, kaolinite, a non-swelling clay mineral with a 1:1 (TO) structure, bentonite, a swelling clay mineral with a 2:1 (TOT) structure, and illite, a non-swelling clay mineral with a 2:1 (TOT) structure, were used to represent three typical types of clay minerals commonly found in mine tailings. The aim was to understand how clay minerals interact with saline water so as to affect dewatering behaviour of clay-rich tailings. The settling behaviour of pure kaolinite, bentonite and illite clay minerals were studied and the relationship between the

dewatering rate of clay minerals and the driving factors (clay type, clay suspended solid concentration, water salinity, and polymer flocculant dosage) were summarised. In addition, through the use of cryo-SEM, the effect of water salinity on clay aggregate structure was investigated in the presence of a range of water salinities. This study may provide strategies to improve the separation of clay-rich tailings in the presence of saline water.

2. Material and methods

2.1. Raw materials

The kaolinite Q38 mineral was purchased from Sibelco Australia Limited Company, South Australia. A quantitative XRD analysis showed that it contained 85 wt% kaolinite, 11 wt% muscovite, and 4 wt% quartz [23]. Murray and Lyons [24] and Du et al. [25] indicated that there are two types of kaolinite minerals, well-crystallized and poorly crystallized kaolinite. Kaolinite Q38 belongs to the latter category. Poorly crystallized kaolinite particles are poorly ordered and have more complex surface structures and higher specific surface areas than their well crystallized counterparts, as identified by SEM [25]. Bentonite was purchased from Sibelco Group, Australia. Quantitative XRD analysis showed that the sample contained 63 wt% montmorillonite, 25 wt% albite, and 12 wt% quartz. The illite sample was purchased from New Directions Laboratory, Australia and contained 75 wt% illite, 15 wt% kaolinite, and 10 wt% argilla. One type of anionic polyacrylamide/polyacrylate (PAM) copolymer with a trade name of Magnafloc 1011 (MF1011) powder was supplied by BASF Australia Limited and was used as the polymer flocculant. Polymer stock solution (1 g/L) was prepared in deionised water daily. MF1011 was selected for this study as the flocculant because Ji et al. [17] investigated the settling behaviour of clay-rich coal tailings in saline solutions and identified that anionic PAM MF1011 exhibited good settling performance in saline water.

In addition, three types of saline water with low-, medium-, and high-salinity were used for the dewatering tests in this study to represent the common range of saline process water used in the coal preparation plants in Queensland. The range of salinities was based on a survey conducted by Ofori, Firth, McNally and Nguyen [26] of Australia coal preparation plants regarding the quality of process and make-up water. The minimum, medium and maximum values of the concentration of major ions in low-, medium-, and high-salinity water indicated the variation in composition of process water used in the coal processing plants. In the lab, synthetic saline water was prepared by mixing a known amount of analytical grade salts such as $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$, $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$, KCl, Na_2SO_4 , NaCl, NaHCO_3 , and $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ in deionised water. The salts were purchased from Sigma-Aldrich and used directly without further purification. The saline water was well mixed using a mechanical stirrer and used for settling, zeta potential, and cryo-SEM tests. The composition of low-, medium-, and high-salinity water is summarised in Table 1. The water composition was also compared with some other process saline water used in metallic mines [19,27, 28] listed in Table 2. These showed even higher concentrations of sodium and chloride ions.

The size distribution of the clay particles was measured by a Laser Diffraction Malvern Mastersizer (Model No MSX14) and the results are shown in Fig. 1. Kaolinite had greater than 80% particles smaller than 19.2 μm . Bentonite had greater than 80% particles smaller than 28.6 μm and illite had greater than 80% particles smaller than 14.8 μm .

Nitrogen sorption experiments were carried out on a Micromeritics TriStar 3020 analyser after degassing under vacuum on a Micromeritics VacPrep061 at 200 °C for a minimum of 6 h. The specific surface areas were calculated from the adsorption isotherms via a multi-point Brunauer–Emmett–Teller (BET) model. The BET surface area of kaolinite, bentonite, and illite were 14.30 $\text{m}^2 \text{g}^{-1}$, 37.15 $\text{m}^2 \text{g}^{-1}$, and 56.89 $\text{m}^2 \text{g}^{-1}$, respectively. It was interesting to find that the size of the kaolinite was finer than bentonite whilst its specific surface area

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