



pH-mediated interfacial chemistry and particle interactions in aqueous muscovite dispersions

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ABSTRACT

Interfacial chemistry and particle interactions of aqueous muscovite dispersions have been investigated in the pH range 2–9. Particle zeta potential, reflecting interfacial chemistry, indicated a strong pH-history and solid loading dependency. Pristine particles' zeta potentials measured from high to low pH indicated an isoelectric point (iep) at ~pH 4.5. Subsequent measurements from low to high pH showed differing electrokinetic potentials with iep shift to higher pH values, the extent of which depended upon particle volume fraction and aging time. Dispersion shear yield stress analysis revealed similar pH-history and solid loading dependency. Incongruent leaching was observed to be responsible for the interfacial chemistry change and rheological behaviour. Upon decreasing pH from 9 to 2, considerable leaching of the key elements in muscovite, Al(III), Si(IV), K⁺ and Fe(III), occurred. The species concentrations decreased dramatically upon subsequent pH increase to higher values due to their hydrolysis and specific adsorption. Dispersion shear yield stresses recorded from high to low pH sweep were pH-independent. Similar measurements from low to high pH values, however, showed a strong pH-dependency, with maximum yield values at the iep. The interfacial chemistry and particle interactions, both displaying bifurcation behaviour, showed good compliance with DLVO theory.

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1. Introduction

Due to their ubiquitous nature, clay minerals exert significant influence in a number of hydrometallurgical processing operations including uranium and copper leaching [1], precipitation and waste tailings dewatering [2–4]. The association of clay minerals such as muscovite with other ore phases as middling or liberated particles invariably presents intractable challenges which have a striking impact on concentrated mineral dispersions' particle interactions, flow behaviour, flotability, "pumpability", filtration and thickening processes, etc. Muscovite (ideally, $KAl_2(AlSi_3O_{10})(OH)_2$), sometimes referred to as sericite, is one of the layered mica group clay minerals with a 2:1 structure. The crystal structure comprises an Al–O–Al octahedral (O) layer sandwiched between two Si–O–Al tetrahedral (T) layers. Substitutions of lattice Si⁴⁺ by Al³⁺ in the tetrahedral layer and Fe³⁺ or Mg²⁺ and Ca²⁺ for Al³⁺ in the octahedral layer which invariably occur, results in a permanent net negative charge on the basal surfaces. The charge deficiency is compensated for by interlayer cations such as K⁺ and Na⁺ adsorbed between the TOT sheets, fitting closely into the hexagonal holes of the Si–O–Al sheet. The interlayer cations (e.g. K⁺) strengthen the bonding between basal planes of TOT sheets, which are nor-

mally held by attractive van der Waals forces, through the attractive electrostatic interactions [5]. These forces, consequently, render muscovite particles non-swelling in aqueous environments. Muscovite acquires hydrophilic character as a result of the aluminol groups (–AlOH) exposed at its edge surfaces. Despite the fact that muscovite is widely used in electronics and industrial applications, in the mining and minerals industry, it constitutes a non-valuable gangue mineral when associated with other desired minerals of higher economic value (e.g. copper, gold, uranium and platinum group).

During clay minerals processing in aqueous media at a given pH, the particle size, shape and surface characteristics together with the presence of hydrolysable metal ions play a pivotal role in defining the overall particle interactions in the dispersions [2,3]. The interplay between interfacial chemistry and mineral particles interactions in regulating the net interactions energy potential and hence, colloid stability and rheology at certain pulp pH values has been widely investigated [2–14]. To date however, there is still a dearth of knowledge and understanding of the nature of the interfacial chemistry and particle interactions displayed by concentrated dispersions of the various isomorphously substituted mica group clay minerals such as muscovite, under certain industrially relevant processing conditions. For a given pH and temperature ranges, whether or not a given muscovite clay dispersion displays shear thinning or thickening behaviour, is not predictable from the pristine particle surface chemistry, solution speciation and supernate

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ionic strength. As muscovite may leach or dissolve in an aqueous environment, pH- dependent hydrolytic and oxolation reactions of dissolved products (e.g. Al(III), Fe(III), Si(IV)) and their subsequent adsorption and precipitation onto particles' surface can significantly change the underlying interfacial properties [14]. Under certain processing conditions, the prevailing clay pulp chemistry and particle interactions may lead to unexpected, intractable rheological (e.g. high yield stress) issues and dewaterability challenges [2–4].

Of particular interest in the present work is the influence of pH-mediated pulp interfacial chemistry on particle interactions prevailing in dilute and concentrated (8–65 wt.% solid or 0.03–0.4 volume fraction), aqueous dispersions of muscovite at pH range 2–11. Specifically, the effect of pH and its history, multivalent metal ions which leach into the supernatant and aging time on interfacial chemistry and rheology is systematically investigated at 25 °C. To quantify the interfacial chemistry, particle zeta potential measurements were conducted at different pH values. The concomitant particle interactions were characterised via dispersion flow curves and shear yield stress measurements. The properties and behaviour of the muscovite investigated herein are pivotal to a number of separation processes in the minerals industry, including hydrometallurgical value mineral (e.g. uranium, gold and copper) extraction, leach and flotation tails dewatering and environmental waste minimization.

2. Experimental methods

2.1. Materials

Polydispersed muscovite particles (Geological Specimen Supplies, Australia) were used in this work as received. The particle BET surface area was 12.7 m²/g and the 10th, 50th and 90th percentile particle sizes, D_{10} , D_{50} and D_{90} , determined by laser diffraction (Malvern Mastersizer X, Malvern, UK) were 3, 30 and 140 μm, respectively. X-ray powder diffraction confirmed the high crystallinity of the sample whilst XRF analysis showed the oxide components to be SiO₂ (47%), Al₂O₃ (31.8%), K₂O (10%) and Fe₂O₃ (3.6%) and Mn, Mg and Ca as trace elements. Electron microprobe (CAMECA SX51) analysis confirmed $K_{0.91}Na_{0.07}(Al_{1.82}Fe^{3+}_{0.14}Mg_{0.08})(Al_{0.93}Si_{3.07}O_{10})(OH)_2$ to be the exact crystal structure. Dispersions with different solid contents (8–65 wt.%) were used. They were prepared by adding a known mass of dry muscovite particles to a known mass of 10⁻³ M KNO₃ solution used as background electrolyte. Concentrated KOH (Chem-Supply) and HNO₃ (Scharlau Chemie) were used to control and adjust the pH of fresh dispersions during zeta potential and shear yield stress measurements. Dispersions were homogenized by mixing for 10 min using overhead stirrer at 600 rpm before altering pH. High purity Milli-Q water (specific conductivity < 0.5 μS cm⁻¹, surface tension at 20 °C of 72.8 mN m⁻¹ and pH of 5.6) was used for preparation of all solutions and dispersions.

2.2. Zeta potential measurements

For interfacial chemistry characterization, the zeta potential of particles in low (8 wt.%) and high (50–57 wt.%) solid content dispersions were determined with an Acoustosizer II (Colloidal Dynamics Inc., Australia). This instrument measures the electrokinetic sonic amplitude (ESA) of particles in dispersion and converts it to dynamic mobility. The zeta potential was then determined from the dynamic mobility using the Smoluchowski model [15]. Fresh dispersions were made and their pH decreased from high (~9) to low (~2) and the zeta potential measured at different pH values.

For each new pH value, the samples were allowed to equilibrate for 5 min prior to data acquisition.

2.3. Rheological measurements

For rheological studies, dispersions with solid contents in the range of 50–65 wt.% were used. Flow curves and low shear yield stress values (<20 Pa) for dispersions were determined using a couette concentric rheometer (Haake RV1), whilst the higher shear yield stresses (>20 Pa) were measured by the vane technique (Haake VT550). The advantage of the latter technique is the minimised slip between the sample and the instrument fixture [16,17], giving a more accurate yield value particularly for structured dispersions (shear yield stress >20 Pa) [18–20]. For indirect shear yield stress measurements, the shear rate was increased from 0 to 1000 s⁻¹ and back to 0 over a 200 s period while shear stress measured continuously as a function of shear rate. As the dispersions showed a non-Newtonian, Bingham plastic behaviour, the shear yield stresses were estimated by extrapolation of the linear part of the flow curves using the Bingham plastic model:

$$\tau = \tau_B + \eta_B \dot{\gamma} \quad (1)$$

where τ and τ_B are the shear stress and Bingham shear yield stress (Pa), respectively, η_p is the plastic viscosity (Pa s) and $\dot{\gamma}$ is the shear rate (s⁻¹). For direct yield stress measurements, the vane was immersed in 0.2 dm³ dispersion and rotated at constant slow rate ($\dot{\gamma} = 0.021$ s⁻¹) and the yield stress estimated from the maximum torque (T_m) attained using following equation:

$$T_m = \frac{\pi D^3}{2} \left(\frac{H}{D} + \frac{1}{3} \right) \tau_y \quad (2)$$

where D and H are the vane diameter and height (m), respectively and the units of T_m and τ_y are N m and Pa, respectively. To investigate the compliance of the particles interactions with DLVO theory [21,22], the elastic floc model [23,24] was applied on the basis that a linear relation between measured shear yield stress and square of zeta potential will be observed if DLVO forces were dominant:

$$\tau_y = \frac{1}{20r\eta_s\dot{\gamma}} \left(\frac{A_h}{12d_1^2} + B(d_1)\zeta^2 \right) \quad (3)$$

where τ_y (or τ_B) is shear yield stress (Pa), A_h is Hamaker constant (J), $\dot{\gamma}$ is the shear rate, d_1 is interparticle separation distant (m), r is the particle radius (m), η_s is the suspension viscosity (Pa s), $B(d_1)$ is a fitting constant which is negative in value and dependent on the elasticity of the interparticle bonds and ζ is the particle zeta potential (V).

2.4. Aging behaviour

Dispersions with 8 and 57 wt.% solid contents were prepared and their pH decreased from the pristine value (~pH 9) to pH 2 or 3 and then increased to the initial value after a fixed period of aging (5 min or 1 h). Both the particle zeta potential and shear yield stress of the dispersion were recorded at selected pH values. At each pH unit, dispersions were allowed to equilibrate for 5 min before the measurements were performed and an aliquot of suspension taken for solid-supernatant separation by centrifugation. The resulting supernatants were analysed by inductively coupled plasma (ICP) for speciation. To achieve good reproducibility of results, all rheological, zeta potential and aging measurements were replicated at least three times and the pure errors determined and reported at 95% confidence interval.

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