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Multi-scale features including water content of polymer induced kaolinite floc structures

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ABSTRACT

Despite their many uses, fine clay particles, such as kaolinite, present a problem in the management of tailings in various mineral industries such as the oil sands and phosphate processing industries. The effective flocculation, sedimentation and consolidation of these fine particles are major challenges. The structure of the flocs and the water entrapped within the flocs determine floc behavior and settling characteristics. The quantification of water entrapped within the kaolinite flocs has not been reported previously.

In this research, a new technique was developed for water content and size analysis of sedimented kaolinite flocs using High Resolution X-ray Microtomography (HRXMT). The results suggest a normal distribution of water content for these flocs, with mean water content of 53.9% by volume and a standard deviation of 11.8%. About 98% of the flocs were found to have water content in the range 30–80%. The size analysis revealed that about 90% of the flocs are less than 1.5 mm in size. The water content was found to decrease with an increase in size of the floc. The floc shape analysis was done for selected flocs. The flocs were found to be fairly irregular, with sphericity values around 0.1.

In addition to macroscopic analysis of individual flocs, flocs were also analyzed for their microstructure by cryo-SEM. Visualization of floc microstructure and polymer chains revealed the stabilization of kaolinite microflocs in the web formed by polymer chains. The structure of the polymer chains as well as the interaction between microflocs and polymer chains is a key to understanding floc growth and stability.

1. Introduction

Fine clays, such as kaolinite, pose a huge challenge for the management of tailings in various mineral industries such as the oil sands and phosphate processing industries (Zhang and Bogan, 1995; Chalaturnyk et al., 2002). The surface charge characterization of kaolinite clay using Atomic Force Microscopy (AFM) has been reported previously (Gupta and Miller, 2010; Liu et al., 2014). The authors that at low pH values (below pH 6.5), the alumina face is positively charged while the silica face and edge surface are negatively charged. However, all three surfaces (alumina face, silica face and edge surface) are negatively charged at higher pH values. The presence of oppositely charged surfaces at low pH values promotes aggregation between these fine clay particles leading to the formation of kaolinite clusters. These kaolinite particles become associated with edge-to-edge, edge-to-face or face-to-face interactions giving rise to a card-house type structure as suggested in the literature (Kie, 1954; Van Olphen, 1977). The card-house structure of kaolinite clusters has been discussed by various researchers over the past years and has recently been described using a dynamic coarse grain simulation model (Liu et al., 2015).

These clusters are about 10 μm in size, however, further increase in the size of aggregates can be obtained by the addition of coagulants or flocculants or both. Polyacrylamide based polymers (PAMs) are generally used as flocculants for tailings containing kaolinite and other fine clays. As the flocs settle under gravity, they form a sediment bed which contains a network of interconnected flocs and water channels. Further consolidation of the sediment is required to release the water for recycling and to minimize the sediment volume for land reclamation. The characterization of floc size, shape and entrapped water content will be helpful in understanding and influencing the tailings settling rate, sedimentation and consolidation.

The size, mass, surface area, and number concentration of particles substantially affect their removal by gravity sedimentation and deposition in packed-bed filters (Lawler, 1986; O'Melia, 1998). In addition to size, particle shape affects the behavior of

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aggregated particles, particularly with regard to collision efficiency (Jiang and Logan, 1991; Wiesner, 1992) and settling rates (Johnson et al., 1996; Li and Logan, 1997; Chakraborti et al., 2000). The particle size and solids weight percentage in the slimes determine the settling characteristics and amount of flocculant required to settle the slimes. The amount of water contained in flocs is directly influenced by the size of the flocs (Winterwerp and van Kesteren, 2004) and the rate at which flocculation occurs (Mietta, 2010). The water content of the flocs also influences the settling and self-weight consolidation (Hendricks, 2016). Previously, techniques such as Nuclear Magnetic Resonance (NMR) spectroscopy have been used to study the water content in different clays (Fan et al., 1999). The analysis of water content for individual kaolinite flocs is yet to be reported. The intra-floc water and size/shape analysis of flocs in kaolinite sediment is challenging due to the problems in imaging and segmentation of the sediment into individual flocs.

The use of X-ray microtomography (XMT) in mineral processing applications has increased over the past few years. Its use has been extended to include size, shape, texture, exposure and liberation of multiphase mineral particle populations (Garcia et al., 2009; Lin and Miller, 2005, 2010). More recently, High Resolution X-ray Microtomography (HRXMT) has been used for 3D particle characterization, multiphase particle segmentation, and data analysis at the University of Utah (Lin et al., 2010; Wang et al., 2015). HRXMT, along with image processing and visualization tools such as ImageJ (Schindelin et al., 2012), Medical Image Processing Analysis & Visualization (MIPAV) (McAuliffe et al., 2001) and Drishti (Limaye, 2012), are powerful tools for particle segmentation and structure analysis.

Although HRXMT has been used previously for studying multiphase solid particles, it has never been used for studying the structure and water content of individual flocs in a sediment bed. In this research, water content and size analysis of kaolinite flocs have been accomplished by using HRXMT and various in-house software. The kaolinite floc sediment bed was treated as a two phase system containing water and kaolinite and the principles of multiphase particle segmentation were applied to the sediment bed to isolate and identify individual flocs.

In addition to the macro scale characterization of flocs, the investigation of floc microstructure is also reported in this work. The flocs are formed by various mechanisms on addition of polymer to the kaolinite suspension. Visualization of floc microstructure would help us understand the interaction between the polymer chains and the kaolinite particles which eventually influence the floc size and shape. In the past, various aspects of kaolinite floc structure and rheological properties have been studied in detail using Scanning Electron Microscopy (SEM) (Mpofu et al., 2003; Kim and Palomino, 2009; Zbik et al., 2008; Du et al., 2009). The results from all these studies are interesting and give information about floc structure during different stages of sedimentation (Zbik et al., 2008), including the effect of polymer concentration (Kim and Palomino, 2009), and the effect of polymer type (Mpofu et al., 2003; Kim and Palomino, 2009). Most of these studies focus on the type of primary particle interactions, i.e. edge-edge, edge-face, face-face interactions at different conditions. Kim and Palomino (2009) concluded that with an increase in polymer concentration face-face interactions increase resulting in a higher density of flocs. They also investigated the effect of polymer type and concluded that cationic polymers produce more porous flocs than the anionic polymer induced flocs. Mpofu's research suggests that free settling flocs show predominant edge-edge interaction whereas settled flocs show more face-face interaction (Mpofu et al., 2003). Although we have some idea about the structure of kaolinite clusters, we have limited information on the structure of polymer chains and how these chains bridge the smaller aggregates and create flocs millimeters in size. In this research, an attempt has been made using SEM to visualize the kaolinite floc microstructure as well as polymer bridges connecting these microstructures. Such visualization will enhance our understanding of kaolinite floc formation with a polyacrylamide flocculant. This information can, in turn, help in selection of appropriate polymers and operating conditions for flocculation.

2. Materials and methods

Acid washed K2 500 kaolinite (U.S.) obtained from Fisher Scientific was used in this study. The size analysis of primary particles was done using Dynamic Light Scattering (DLS) instrumentation manufactured by Wyatt Technologies. DLS utilizes time-dependent fluctuations of scattered intensity, which arise from Brownian motion, in order to determine the diffusion constant. The hydrodynamic radius R_h is then calculated directly as described in the DLS theory. Details about the DLS theory and other working principles are described in the literature (Berne and Pecora, 2000). Kaolinite suspensions at pH 8.6 containing 0.1% solids were used in this research. About 1 ml of suspension was put in a cuvette and the system was sonicated for about 10 s. The cuvette was then inserted into the DLS system and the size analysis was done. Two peaks were observed during the analysis, at about 100 nm and at 600 nm (Fig. 1). The analysis suggests a bimodal size distribution of kaolinite primary particles.

Polyacrylamide based polymer (AF 303) was obtained from Hychem Inc. The polyacrylamide monomer contains two functional groups, the acryl and the amide group. The extent of substitution of these groups determines the charge density. The polymer used has molecular weight in the range of 12–14 million and a negative charge density of 5%. The polymer selection and solution preparation was done based on recommendations from Pocock Industrial, Salt Lake City, Utah. The polymer was added to suspensions in an aqueous solution state at 0.1 g/l concentration. The solid polymer particles in powder form were added to DI water and a stock solution with a polymer concentration of 1 g/l was prepared. The solution was allowed to equilibrate while being stirred for 8 h at 400 rpm. Right before addition to the kaolinite suspension, some of the polymer solution was diluted to 0.1 g/l and then added to the suspension. Fresh stock solution was prepared every five days as the polymer solution showed some aging if stored for a longer period of time. The aging was evident by a change in viscosity and/or a change in color to off-white. The stock solution was always refrigerated and stored at lower temperatures.

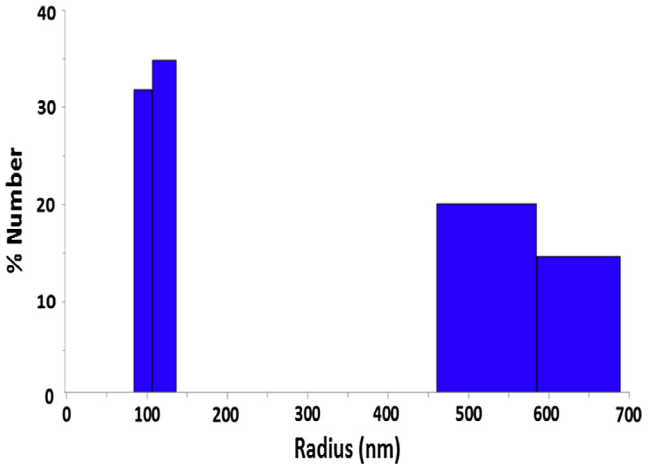

Fig. 1. Size analysis of kaolinite primary particles using DLS. A kaolinite suspension at pH 8.6 and solid concentration 0.1% was analyzed.