



A novel hydrophobically-modified polyelectrolyte for enhanced dewatering of clay suspension

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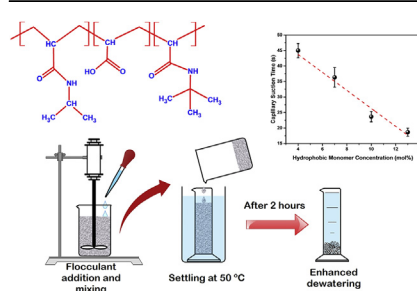
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HIGHLIGHTS

- p(NIPAM-AA-NTBA) is an anionic, hydrophobically modified, thermo-sensitive flocculant.
- Polymer chains caused bridging of clay particles and formed flocs.
- Hydrophobic monomer in this ternary polymer expelled water out of the flocs and improved the dewaterability of sediments.
- The flocculant produced clear supernatant with improved initial settling rate.

GRAPHICAL ABSTRACT



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ABSTRACT

This work investigates the effect of multifunctional poly (*N*-isopropyl acrylamide/acrylic acid/*N*-*tert*-butylacrylamide) [p(NIPAM-AA-NTBA)] ternary polymer on the sedimentation of kaolin clay – a major fraction of oil sands tailings. A series of linear, uncross-linked p(NIPAM), p(NIPAM/AA), and p(NIPAM/AA/NTBM) were synthesized as random copolymers, where all monomer units were randomly arranged along the polymer backbone and connected by covalent bonds. The ternary copolymer, used as a flocculant, exhibited thermo-sensitivity, anionic nature, and hydrophobic association due to NIPAM, AA, and NTBM, respectively. As the ternary polymer is thermosensitive, it undergoes extended to coil-like conformation, i.e. hydrophilic to hydrophobic transition, above its lower critical solution temperature (LCST). The comonomers NIPAM (above LCST) and NTBM help expel water out of sediments due to their hydrophobicity, while AA promotes charge neutralization of the kaolin clay particles. The effect of number average molecular weight, charge density, and concentration of NTBM on settling behavior of kaolin suspension was examined. Settling test at 50 °C resulted in significantly higher settling rates compared to that at room temperature. Further, the quality of water recovered in each experiment was tested in terms of its turbidity. These results indicate that this novel ternary polymer can be employed to enhance the recovery of water from oil sands tailings containing clays.

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1. Introduction

The largest oil sand reserves in the world are located in eight countries: Canada, Venezuela, USA, Trinidad, Madagascar, Albania, Russia, and Romania. In the process of water-based bitumen extraction, oil sand ores are mined, crushed, and mixed with warm alkaline water followed by the separation of bitumen using a

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flotation process (Masliyeh et al., 2004; Wang et al., 2014). The remainder of the slurry, referred to as tailings, is discharged into tailing ponds which occupy an enormous area of land. Fine kaolin clays are the major ingredients of oil sands tailings and remain suspended in water for many years due to its water chemistry. Kaolin clays exhibit a strong tendency to repel each other due to double-layer repulsion that results in the formation of a stable suspension (Botha and Soares, 2015; Long et al., 2006). Current polymer flocculation/dewatering technologies are centered on acrylamide-based polymers such as polyacrylamide (PAM), but sediments produced using PAM flocculants hold considerable amounts of water due to the formation of loosely packed flocs and their superhydrophilic nature. Additionally, recent studies have shown that the possible release of monomers from PAM-based flocculants pose a potential health risk as the released monomers can make way through the food chain (Bolto and Gregory, 2007). Alamgir et al. considered the use of organic-inorganic flocculant synthesized of $\text{Al}(\text{OH})_3$ -PAM; however, the potential risk of leaching aluminum into the environment could not be eliminated (Alamgir et al., 2012). The incorporation of inorganic salts, such as aluminum hydroxide, or aluminum chloride, into flocculants is being avoided due to their potential role in Alzheimer's disease (Divakaran and Sivasankara Pillai, 2001). Recently, many researchers used cationic polymers such as polyacrylamide quaternary amine comonomer to flocculate wastewater containing clay particles (Liu et al., 2017; Wilkinson et al., 2017; Zhang et al., 2016). These researchers used cationic comonomers such as 3-(methacryloylamino)propyl trimethylammonium chloride, 2-methacryloxyethyltrimethyl ammonium chloride, and dimethyldiallylammonium chloride to impart a cationicity to the flocculant. Lu et al. used glycopolymer-poly(2-lactobionamidoethyl methacrylamide) of various molecular weights to flocculate kaolin suspension (Lu et al., 2016). Our group recently exploited hydrolytically degradable polymers to dewater the clay slurries (Gumfekar et al., 2017; Rooney et al., 2016). Researchers have also used bio-based flocculants such as starch, chitosan cellulose to flocculate wastewater containing clays (Das et al., 2013; Divakaran and Sivasankara Pillai, 2001; Jia et al., 2016; Wu et al., 2016; Yang et al., 2014). Till date, oil sands industry has majorly relied on hydrolyzed polyacrylamide, copolymer of acrylamide and acrylic acid, and copolymer containing acrylamide and cationic monomers.

The prevailing mechanisms in destabilizing colloidal suspensions using polyelectrolytes are adsorption and bridging, charge neutralization, and charged patching (Shaikh et al., 2017a). In adsorption and bridging mechanism, polymeric chains first adsorb onto surface of particles in colloidal suspension. Such polymer adsorbed particles bridge other particles in suspension to form a floc. This bridging phenomenon is observed up to a certain dosage of polymer beyond which flocculation deteriorates due to steric stabilization. At lower dosages, polymer is insufficient to form bridging links between particles. Overall, bridging produces stronger flocs than those produced with coagulation. The principle behind charge neutralization is the suppression of electric double layer of a particle due to flocculant's ionic nature. Suppression of electric double layer reduces electrostatic repulsion between particles causing them to aggregate. Charge neutralization is often confirmed by measuring zeta potential over the range of polyelectrolyte dosage. In flocculation induced by high molecular weight polyelectrolytes, both bridging and charge neutralization take place. Charge patching is often observed when high charge density cationic polymers are used in anionic suspensions because the high interaction energy favors flattened configuration of polymers on particle's surface instead of forming loops and trails. As a result, surface of particles exhibits regions/patches of cationic polymer and of existing anions. Such patches can interact with the

surface of another particle forming a bridge between them. This mechanism is found to be dominant in a suspension of low particle concentration (Bolto and Gregory, 2007; Tripathy and De, 2006).

Recently, researchers have used thermosensitive polymers such as pNIPAM for the treatment of various clay suspensions because these polymers can alter their conformation from hydrophilic to hydrophobic (Gao et al., 2006; Sakohara et al., 2011, 2008). To a large extent, the past research on thermosensitive polymers focussed on their theoretical study, whereas the recent literature has focussed on their applications. Additional functionality can be added to pNIPAM by copolymerizing NIPAM with desired monomers such as acrylic acid, 2-aminoethyl methacrylamide hydrochloride, ethyleneimine, allylamine etc (Gao et al., 2005; Han et al., 2015; Lu et al., 2015; Zintchenko et al., 2006). Polyelectrolytes, which exhibit charges on the backbone of the polymer, may form a multilayer assembly, aided by the thermosensitive polymer (Glinel et al., 2003). Additionally, polyelectrolytes cause the coagulation of clay suspension by neutralizing the charges on clay edges, which are responsible for its stability. When hydrophobic monomers are added to the p(NIPAM) electrolyte, the LCST may be changed and hydrophobic domains may be induced (Lowe et al., 1999). In the context of this work, such hydrophobic domains may further expel the water trapped in the sediments. The combination of both hydrophobic and ionic comonomers produces thermosensitive hydrophobically modified polyelectrolytes. By balancing hydrophobic and hydrophilic comonomers and adjusting charge density in the chain, the structure and physical properties of hydrophobically modified polyelectrolytes may be changed to meet desired performance targets.

Taking the advent of the aforementioned factors, we synthesized hydrophobically modified polyelectrolyte flocculants. The synthesized flocculants were employed above their LCST to dewater kaolin suspensions, and the dewatering performance was studied. The performance of flocculants is measured in terms of initial settling rate (ISR), the turbidity of the supernatant, and capillary suction time (CST) of sediments.

2. Experimental

2.1. Materials

N-isopropyl acrylamide (NIPAM), acrylic acid (AA), *N*-tert-butylacrylamide (NTBM), ammonium persulfate (APS), and *N,N,N',N'*-tetramethylethylenediamine (TEMED), purchased from Sigma-Aldrich (analytical reagent grade), were used without further purification. NIPAM, AA, and NTBM were used as monomers while APS and TEMED were used as initiator and accelerator, respectively. Kaolin was purchased in powder form from Sigma-Aldrich (SKU: 18616) and used as received. Deionized water was used as a solvent for all syntheses.

2.2. Synthesis of *p*(NIPAM), *p*(NIPAM-AA), and *p*(NIPAM-AA-NTBM)

The synthesis of polymers was carried out by modifying the procedures described elsewhere (Sakohara et al., 2011, 2008; Sakohara and Nishikawa, 2004). In a typical procedure for *p*(NIPAM) synthesis, a desired quantity of NIPAM was dissolved in DI water. The solution was constantly stirred and purged with nitrogen for 0.5 h to remove dissolved oxygen. Next, the desired amount of TEMED and APS was added to the monomer solution. The polymerization was carried out at room temperature for 24 h. In a free radical polymerization, the polymer molecular weight increases rapidly, but longer polymerization times were adopted to ensure the highest possible monomer conversion. After the

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