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Research paper

Alginate clay hybrid composite adsorbents for the reclamation of industrial lean methyldiethanolamine solutions

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A B S T R A C T

Alginate based clay hybrid composites were used as adsorbents for the removal of total organic acid anions (TOA) as heat stable salt (HSS) and heavy metal ions (chromium and iron) from industrial lean methyldiethanolamine (MDEA, 50 wt%) solvents. Spherical calcium alginate based hybrid (CAH) composites were prepared using three different filler clays such as sepiolite, montmorillonite and bentonite. To overcome the inherent iron leaching problem associated with clay minerals, an appropriate clay treatment method was utilized before preparing the hydrogel composite. The structure of the clay (before and after modification) and composites was characterized by XRD, whereas the sorption mechanism of the CAH hydrogel composites was conferred in combination with the results obtained from SEM, EDX and FTIR analyses. The effect of quantitative contributions of polymer, bead size, agitation speed, cross linker concentration, reaction time and filler weight% were studied using batch adsorption. The initial experiments confirmed that all the hybrid composites could superiorly separate HSS and heavy metal ions from lean MDEA solvents. 2.0 wt% clay containing composite hydrogels prepared by using 1 mm dropper in a $CaCl_2$ solution of 1.5 M was found to have highest adsorption capacity. Thus, calcium alginate hybrid (CAH) composites showed efficient adsorption capacity for removing all the contaminants present in lean MDEA.

1. Introduction

Chemical absorption of natural gas using methyldiethanolamine (MDEA, 50 wt% used by GASCO Company, Abu Dhabi) is so far the preeminent industrial method used for the absorptive removal of H_2S and CO_2 (Pal and Banat, 2014, 2015). However, decontamination of industrial lean MDEA from heat stable salts such as total organic acids (produced by the reaction between aerial oxygen and CO_2/H_2S) and heavy metal ions like chromium, lead etc. (produced from the makeup water or due to the metal corrosion or erosion caused during the continuous running of the plant) always remain as a challenge to the gas industry (Cummings et al., 2007). Though few attempts are reported for the removal of total organic acids (TOA) from alkanolamine solutions using newer processes like distillation (Millard and Beasley, 1993), electro-dialysis (Meng et al., 2008) and ion-exchange techniques (Pal et al., 2013), they suffer from serious drawbacks like cost effectivity, higher energy requirements and continuous supply of huge volumes of water.

Reclamation using adsorbent materials have gathered wide attention due to their outstanding properties such as effective decontamination, high adsorption capacity, fast rate of adsorption, ability to remove even traces of contaminants and efficient regeneration capability. Currently, GASCO Company is using commercially available activated carbon as adsorbent to remove HSS anions. The low adsorption capacity along with the generation of toxic solid adsorbent wastes and associated environmental problems have paved the way for alternatives with focus on sustainable development. To meet this demand of the gas industry, remarkable endeavors have been devoted by the researchers for the design and synthesis of advanced sorbent materials (Pal and Banat, 2014, 2015). The use of biodegradable and bio-based polymers produced from renewable resources, such as proteins, cellulose and polysaccharides have gained wide attention, because of their low cost, biodegradability, low carbon footprint and reduced environmental impact.

Alginates, are naturally occurring biopolymers which are extensively used as starting material for making environmentally benign adsorbents. They are anionic polysaccharides obtained from brown seaweed and consists of linear chain of (1–4) β-D-mannuronic acid (M) and α-L-guluronic acid (G) residues arranged in non-regular block wise pattern (Gacesa, 1988). The ability of these materials to form hydrogels, a three dimensional network of hydrophilic polymers with high water retention capacity by ionically crosslinking with metal ions such Ca^{2+}

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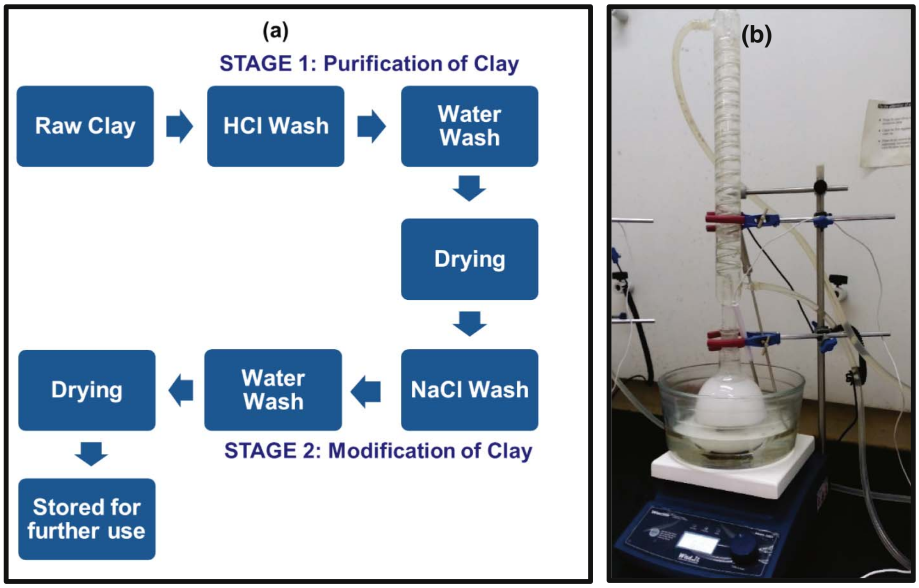

Fig. 1. (a) Various stages involved for the treatment of clay and (b) clay modification setup.

have envisaged its application in interdisciplinary fields (Rowley et al., 1999; Goh et al., 2012; Lee and Mooney, 2012). Recently, (Banat et al., 2014) investigated to utilize the highly active carboxyl and hydroxyl groups present in these hydrogels for achieving simultaneous remediation of heavy metals and total organic acids from industrial lean MDEA solutions. However, uncontrollable degradation owing to poor mechanical property has further limited the practicability of these hydrogels.

Several efforts have been devoted by the researchers not only to enhance the mechanical performance of alginate hydrogels but also expand its areas of applications. Reinforcing inorganic materials such as clay into the alginate matrix have gained significant amount of interest over the past years owing to their high surface area, strong adsorption capability and potential complexation capacity (Barreca et al., 2014; Chiew et al., 2016). These hybrid materials called composite hydrogels are believed to have exceptional mechanical properties, high adsorbent density, enhanced adsorption efficiency and biocompatibility; reflecting the combined effect of alginate and clay materials and have attracted great attention in water treatment applications (Luo et al., 2014; Cataldo et al., 2015; Gopalakannan and Viswanathan, 2015). The immobilization of fine clay materials as reinforcing phase within the polymeric matrix overcomes the challenges faced in separating the clay for regeneration when used as an adsorbent itself (Tan and Ting, 2014). In addition, alginate-clay composites are the most widely used material for applications such as drug delivery (Ahmed, 2015; Jana et al., 2016; Oliveira et al., 2017), pervaporation separation membranes (Bhat and Aminabhavi, 2006; Suhas et al., 2014; Xing et al., 2016), insulation foams (Chen et al., 2016; Darder et al., 2017) etc.

Therefore, clay encapsulated alginate hybrid composites ascertains to be a promising candidate for purifying the amine solvent through clean and safe process. However, future research needs to be carried out to confirm the improvement in mechanical properties along with guaranteed removal efficiency. To best of our knowledge, the application of modified clay encapsulated calcium alginate hydrogels has not yet been investigated for the simultaneous removal of TOA and heavy metal (chromium and iron) from industrial lean MDEA solvents. Even with the pivotal role of clay minerals in the composite hydrogel, it is of great importance to lay emphasis in avoiding the leaching of metals such as iron from the clay into the solution, which can further results in corrosion and erosion of the system (Cummings et al., 2007).

Thus, this study aimed at distinguishing the role of various types of clay such as Bentonite, Montmorillonite and Sepiolite as fillers on the adsorption potential for reclaiming industrial lean MDEA using batch adsorption experiments and subsequently screening the appropriate clay materials as best fillers for calcium alginate/clay hybrid (CAH) composite hydrogels. Major efforts were paved in identifying the best clay treatment methods in obtaining a polymer/clay hydrogel composite that is free from metal leaching. The influence of various experimental factors such effect of hydrogel bead size, rate of agitation, cross linker concentration, polymerization time and filler weight % on the adsorptive performance were also investigated. XRD, TGA, SEM, EDX and FTIR analysis were conducted to identify the morphological changes on the composites before and after adsorption. The best CAH composites were selected based on the adsorption studies conducted using lean MDEA containing different initial contaminant concentration. Finally, the possible mechanism responsible for the removal of the contaminants from lean MDEA and a comparison of the different CAH composites with the activated carbon used by GASCO was exemplified.

2. Experimental

2.1. Materials

Sodium Alginate of 91% food grade with minimum viscosity of 45 cP (cps) for 1% solution in water were purchased from Loba Chemie Pvt. Ltd., India. Calcium chloride dihydrate was obtained from Merck KGaA, Germany. All the chemicals purchased were of reagent grade and were used as obtained. For adsorption studies, lean MDEA solvent containing 50 wt% methyldiethanolamine (MDEA) and activated carbon were provided by GASCO Company (Habshan, Abu Dhabi) and used without any modification. Deionized water was used for all the experiments. Three commercially available clays (Bentonite sodium form, Alfa Aesar; Montmorillonite (K-10) and Sepiolite, Sigma Aldrich) were used in this study to compare their efficiency in removing TOA and heavy metal ions from industrial lean MDEA.

2.2. Methods

2.2.1. Clay pretreatment procedure

Preliminary adsorption results indicated that pretreatment and further modification of clay are required to eliminate the leaching of heavy metals such as iron to the lean MDEA solutions. The pretreatment process was carried out for all the clays except Bentonite as it is obtained as sodium form.

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