



Research paper

Synthesis of a kaolin-based geopolymer using a novel fusion method and its application in effective water softening



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ABSTRACT

In this study, kaolin-based geopolymer was synthesized using a two-step method that consists of a fusion step (fusion of kaolin and sodium hydroxide), and a hydration and dealcalization step. The fusion step was performed at a temperature range of 400–800 °C for 10 to 20 h, and the hydration and dealcalization step was carried out by washing the fusion product with demineralized water for a short time (< 10 min). The synthesized geopolymer was characterized using XRD, FTIR, SEM, EDS and BET analyses. The sorption efficiency of the synthesized geopolymer was examined via removing Ca^{2+} and Mg^{2+} from both model media and groundwater. Results showed that sodium hydroxide to kaolin ratio, fusion time and temperature have profound effects on the performance of geopolymer in reduction of water hardness. Response Surface Methodology (RSM) was used to determine the optimal geopolymer synthesis conditions. RSM results indicated that the sodium hydroxide to kaolin ratio of > 2.2, the fusion time of > 14 h and the fusion temperature range of 500–700 °C provide the optimal synthesis conditions. Moreover, the synthesized geopolymer can efficiently adsorb Ca^{2+} and Mg^{2+} from both model media and groundwater. Based on the Langmuir isotherm model, the maximum Ca^{2+} adsorption capacity was 76.34 mg/g at 25 °C, increasing to 94.34 mg/g at 45 °C. Similarly, the maximum Mg^{2+} adsorption capacity increased from 39.68 mg/g at 25 °C to 51.55 mg/g at 45 °C. The adsorption experimental data at the above mentioned temperatures (i.e., 25 and 45 °C) fitted well with the pseudo-second-order model. Finally, efficient regeneration of the saturated geopolymer, using sodium chloride solution, suggested that the dominating mechanism of water softening by the synthesized geopolymer is ion exchange.

1. Introduction

Geopolymers or inorganic polymers are amorphous aluminosilicates with three dimensional AlO_4^- - SiO_4 tetrahedral frameworks. Geopolymers are formed by alkali activation of aluminosilicate precursors such as kaolin (van Jaarsveld et al., 2002; Van Jaarsveld et al., 2004; Skorina, 2014; El-Eswed et al., 2015), fly ash (Wang et al., 2007; Al-Zboon et al., 2011; Provis and Van Deventer, 2014), and furnace slag (Yunsheng et al., 2007; Suksiripattanapong et al., 2017). Generally speaking, the fusion method for geopolymerization consists of treatment of aluminosilicate precursors with sodium hydroxide followed by mixing the fusion product with water and aging the mixture (Li et al., 2006; Wang et al., 2007; Tchakoute et al., 2013; Provis and Van Deventer, 2014; El-Naggar and El-Dessouky, 2017). Geopolymerization process is time intensive and is accomplished using a variety of methods. Normally, depending on the precursor and the method of synthesis, it takes 1 to 3 days (Al-Zboon et al., 2011; Cheng et al., 2012.), and even up to 28 days (Komnitsas, 2011).

In recent years, geopolymers have been used as fireproof building materials, anticorrosive cement, inorganic composites and ceramics (Davidovits, 2011; Provis and Van Deventer, 2014; Zhang et al., 2016; Aguirre-Guerrero et al., 2017), heat-resistant coatings and adhesives, medicinal applications, high-temperature ceramics, new binders for fire-resistant fiber composites, toxic and radioactive waste encapsulation and new cements for concrete (Chakravorty et al., 2014).

Similar to zeolites, the inclusion of Al in the geopolymer matrix induces a negative charge onto the geopolymer framework. This inclusion requires the presence of alkali or alkaline earth cations to neutralize the framework charge. Hence, geopolymers have appreciable cation exchange properties; and can be viewed as amorphous analogs of zeolites with ion cation exchange properties (Sindhunata et al., 2006; Wang et al., 2007; O'Connor et al., 2010; Al-Zboon et al., 2011; Skorina, 2014). The preparation of geopolymers is simpler than the synthesis of zeolites. This is because it involves no nucleation or crystallization step (Rees et al., 2008; Cheng et al., 2012). Therefore, it seems that utilization of geopolymers in water and wastewater treatment should be

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more feasible (Al-Zboon et al., 2011; Cheng et al., 2012).

In recent years, the performance of geopolymers in removal of water pollutants such as heavy metals is examined. Skorina (2014) studied ion exchange properties of a pure potassium-based geopolymer. Ge et al. (2015) synthesized porous metakaolin-based geopolymeric spheres by a suspension and solidification method and studied their performance in removing Cu^{2+} from aqueous solution. Al-Zboon et al. (2011) used a fly ash-based geopolymer for Pb^{2+} removal from aqueous solution. Cu^{2+} and dye removal from aqueous solutions using fly ash-based geopolymer synthesized by fusion method are reported by Li et al. (2006) and Wang et al. (2007), respectively. Cheng et al. (2012) and Kara et al. (2017) introduced the heavy metal adsorption characteristics on a metakaolin-based geopolymer. These studies show that geopolymers are effectively capable of adsorbing heavy metals. However, studies conducted on the post desorption of pollutants from geopolymers are rather limited. The review of the literature shows that most case studies are aimed at immobilization and solidification of heavy metals (Wang et al., 2007; Yunsheng et al., 2007; Zheng et al., 2010; Medpelli et al., 2015). The difficulty of regeneration of the used geopolymers for heavy metals has been reported before (Cheng et al., 2012; Ge et al., 2015). It is mostly believed that the combined physical adsorption, formation of strong chemical bounds, and chemical encapsulation are responsible for the adsorption of heavy metals onto the geopolymer matrix (Cheng et al., 2012; Ge et al., 2015; Medpelli et al., 2015). Our literature survey shows that the performance of geopolymers in water softening process has not been studied yet. Water softeners are widely used as detergent builders, food supplements, and adsorbents in water and waste water treatment (Comstock and Boyer, 2014; Xue et al., 2014). Among the various zeolites, the high ion exchange capacity of zeolite 4A has led to its wide application as a water softener (Hui and Chao, 2006; Loiola et al., 2012; Comstock and Boyer, 2014; Xue et al., 2014; Ayele et al., 2015).

Our extensive literature search shows that the time of hydration step in the synthesis of geopolymers using the standard fusion method is long (in order of many hours to a few days). The reduction of this time would be highly appealing in geopolymerization processes on a commercial scale. On the other hand, diversifying the applications of geopolymers and finding new applications for this new generation of materials would increase the demand for their production. This in turn would enhance the reuse of wastes that contain precursors for synthesis of geopolymers.

In the present communication, we report a convenient and novel fusion method for the synthesis of kaolin-based geopolymer in which the time of hydration step is reduced considerably; and the synthesized kaolin-based geopolymer is unprecedentedly used for water softening. This paper is organized as follows. After the introduction, Section 2 explains materials and methods including geopolymer synthesis, characterization, application of the synthesized geopolymer in water softening, and regeneration of the saturated geopolymer and its reuse. The third section presents and discusses the experimental results followed by the investigation of kinetics of the reduction of hardness of water by the synthesized geopolymer. Some conclusions are drawn and highlighted in the final section.

2. Materials and methods

2.1. Geopolymer synthesis

Kaolin powder (supplied by Kersial, Czech Republic) was used for the synthesis of geopolymer, since it is a rich source of aluminum and silicon. The weight percent of the major chemical ingredients of the kaolin were SiO_2 , 46.57%, Al_2O_3 , 37.20%, Fe_2O_3 , 0.81%, TiO_2 , 0.16%, CaO , 0.36%, K_2O , 0.98%, and its loss on ignition (LOI) was 13.92%. In this study, geopolymer was synthesized in two steps. The first step was fusion of kaolin and sodium hydroxide at elevated temperatures, and the second step consisted of hydration and dealcalization that was

performed by washing with distilled water. In this method, there is no need to transform the kaolin into metakaolin. In order to synthesize geopolymer, the kaolin was activated by sodium hydroxide (NaOH, purchased from Merck). NaOH and kaolin were mixed at a certain ratio, (NaOH/kaolin weight ratio between 1 and 4), by grinding in a mortar. Then, the mixture was placed in a furnace at a specified temperature (400 to 800 °C) for 10 to 20 h. Subsequently, the fusion product was cooled, ground, and rinsed with distilled water (800 cm³ distilled water was required for dealcalization and hydration of 1 g of fusion product). This step was carried out till the neutral pH was reached. Finally, the prepared sample was dried at 100 °C for about 24 h and was saved in a desiccator for characterization and application experiments.

2.2. Geopolymer characterization

The synthesized samples that have been subject to dealcalization and hydration were characterized using X-ray diffraction (XRD, Philips X'pert power system) in the 2θ range of 10–60° for phase determination. Fourier Transform Infrared Spectroscopy (FTIR) spectra were recorded on a JASCO FT/IR-680 FTIR spectrometer over the range of 400–4000 cm^{-1} . Scanning electron microscopy (SEM) coupled with chemical analysis by energy dispersive spectroscopy (EDS), Philips XL30, was used for evaluation of the morphology of kaolin and geopolymer and the Brunauer–Emmet–Teller (BET) surface area was measured by nitrogen adsorption–desorption isotherm at 77 K (BELSORP-mini II, BEL Japan). The pore size was calculated using the Barrett–Joyner–Halenda (BJH) method.

In this study, the purpose was to use the synthesized geopolymer to remove calcium (Ca^{2+}) and magnesium (Mg^{2+}) ions from aqueous solution. The effective parameters in the synthesis of geopolymers using the fusion method are the sodium hydroxide to kaolin ratio, fusion time, and temperature. Initial results showed that these parameters have a significant impact on the geopolymer performance in removing the water hardness. In order to optimize the synthesis parameters, statistical design of experiment (DOE) was performed through response surface methodology (RSM) using Design-Expert 9.0 Software (trial version). Based on the Box–Behnken design, the number of experiments required to investigate three parameters at three levels was 17 with 5 center points. Table 1 lists the range and levels of effective parameters which were chosen based on initial tests. Low, middle, and high levels of each factor were given the values of -1 , 0 , and 1 , respectively.

2.3. Evaluation of the effectiveness of geopolymers in hardness removal

Ca^{2+} and Mg^{2+} solutions were prepared using calcium chloride and magnesium chloride salts (Merck). Batch adsorption experiments were performed in stopper bottles to study the performance of the water hardness removal of the geopolymer. According to the statistical design, 17 samples of the synthesized geopolymer were used to remove Ca^{2+} from a 100 ppm solution. The adsorbent to solution ratio was set to 0.001 $\text{g}\cdot\text{mL}^{-1}$. The batch adsorption tests were performed at fixed temperatures (25 and 45 °C) with constant stirring. The pH was adjusted at 6. Ca^{2+} and Mg^{2+} concentrations were measured by titration method, using EDTA solution. To calculate the removal percentage (R), the following equation was used.

Table 1
The levels and range of different synthesis variables in coded and uncoded form.

Factors (independent variables)	Level range (Coded levels)		
	1	0	−1
Fusion temperature (°C), X_1	800	600	400
Fusion time (h), X_2	20	15	10
NaOH/Kaolin ratio, X_3	4	2.5	1

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