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Zeolite A synthesized from alkaline assisted pre-activated halloysite for efficient heavy metal removal in polluted river water and industrial wastewater

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ABSTRACT

High quality zeolite A was synthesized through a hydrothermal process using alkaline-assisted pre-activated halloysite mineral as the alumina and silica source. The synthesis conditions employed in this study were finely tuned by varying the activating temperature, sodium hydroxide content, water content and Si/Al ratio. The obtained zeolite A showed excellent adsorption properties for both single metal cation solutions and mixed cation solutions when the concentrations of the mixed cations were comparable with those in polluted natural river water and industrial wastewater. High adsorptive capacities for Ag^+ (123.05 mg/g) and Pb^{2+} (227.70 mg/g) were achieved using the synthesized zeolite A. This observation indicates that the zeolite A synthesized from alkaline-assisted pre-activated halloysite can be used as a low-cost and relatively effective adsorbent to purify heavy metal cation polluted natural river water and industrial wastewater.

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Introduction

The widespread contamination of drinking water sources by heavy metal ions, such as lead, chromium, zinc, nickel, and copper, is of major concern because of their toxicity, persistence and bioaccumulative nature (Hafiza et al., 2015; Fu and Wang, 2011; Ciszewski and Grygar, 2016; Turhanen et al., 2015; Simeonidis et al., 2016). These kinds of heavy metal pollution can lead to serious human health hazards through the food chain, and especially from drinking water, which can harm the whole biological environment (Hafiza et al., 2015). The pollution of rivers by heavy metals has been taking place in many countries in recent years. The concentrations of heavy metals in the water of several famous rivers are higher than the guidelines issued by the World Health Organization (WHO) (Temnerud et al., 2013; Li et al., 2011; Shiller, 1997; Liu and Li, 2011; Kurniawan et al., 2006). Moreover, the pollution in many small rivers is even more serious.

In past decades, a lot of effort has been made toward the purification of natural river water for drinking purposes.

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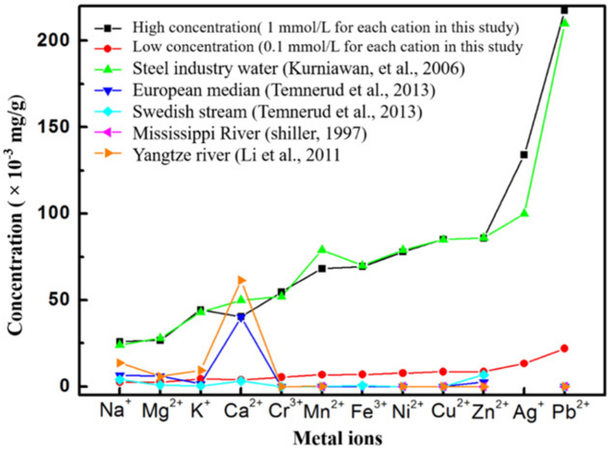

Fig. 1 – Comparison of metal concentrations in natural river water, industrial wastewater and the simulated solutions used in this study.

Various methods, such as adsorption, flotation, phytoremediation, electrochemical treatment, membrane filtration, and chemical precipitation, have been used for heavy metal removal in drinking water (Shannon et al., 2008; Chong et al., 2010; Thompson, 2015). Of the methods, adsorption is an environmentally friendly and low-cost method which has been widely used in the treatment of polluted water. There are many adsorbents that can be used in industry to remove heavy metal cations in processing natural water or industrial wastewater, including ion exchange resins (Eldin et al., 2016), activated carbon (Jurado-sánchez et al., 2014), clay minerals (Zhu et al., 2016), and zeolites (Ghasemi et al., 2016). Zeolites are often chosen not only because of the porous structures of these materials, but also because they can provide excellent heavy metal adsorption capacity without the high price of more complex adsorption media. Among the different types of zeolites, zeolite A is of particular interest as an adsorbent. It is expected to have the best adsorption capacity and selectivity among the most common low-cost zeolites (Dyer and Emms, 2005; Higgins et al., 2002). During the past years, zeolite A has been employed in a few different systems as an absorbent to remove hazardous materials, including heavy metals at different concentration levels, however, studies on the use of Zeolite A to remove heavy metals cations in complex cation solutions at levels similar to those in river water have been scarce.

Herein, we employed an alkaline pre-activated hydrothermal synthesis method (Li et al., 2012; Liu et al., 2015; Yue et al., 2014), by using halloysite mineral, which has a crystal structure similar to kaolinite but with much higher purity and quality, as a source material to prepare pure and highly crystalline zeolite A. Parameters affecting the synthesis such as temperature, amount of alkali for activation, and amount of water in the mixtures used for crystal growth, were carefully investigated. Subsequently, the prepared zeolite A was used as an adsorbent to remove heavy metal cations from aqueous solutions, both for single metal cation solutions and mixed solutions, where the selectivity for different heavy metal cations was determined. Parameters affecting adsorption, such as the effect of competitive innocuous cations and initial metal cation concentration, were studied as well. The low (0.1 mmol/L for each cation) and high (1 mmol/L for each cation) concentrations in this study were comparable to those in contaminated water in typical rivers over the world and industrial wastewater, respectively (Fig. 1). The results indicated that the synthesized zeolite A exhibited high adsorption selectivity and capacity for removing heavy metal cations in both contaminated natural river water and industrial wastewater.

1. Experimental section

1.1. *Characterization*

Both the raw minerals and the synthesized zeolite A were characterized by powder X-ray diffraction (PXRD), Fourier Transform Infrared spectroscopy (FT-IR), and scanning electron microscopy (SEM). PXRD patterns were obtained by using a PANalytical X'Pert X-ray diffractometer with Cu Kα (λ = 1.5406 Å) radiation operating at 45 kV and 40 mA over the 2θ range of 5°–50°. Fourier transform infrared spectra (FT-IR) were recorded on a Varian 670-IR spectrometer in the range 400–4000 cm^{-1}. Scanning electron microscopy (SEM) was performed using a SU-7000F SEM, operating at an accelerating voltage of 10 kV. Transmission electron microscopy (TEM) images and energy dispersive X-ray (EDX) spectrum were obtained with a JEOL FEG 2100 microscope. N_2 adsorption and desorption isotherms were measured using a Micromeritics ASAP2020 device at 77 K. A ThermoFisher ICAP 6500 inductively coupled plasma mass spectrometer (ICP) was used to measure the concentrations of cations after absorption.

1.2. *Raw materials*

Halloysite is an aluminosilicate clay mineral, consisting of alumina octahedral sheets and silica tetrahedral sheets in 1:1 stoichiometric ratio. In our experiments, natural halloysite mineral was purchased from Sigma and used as the silica and alumina source. Both SEM and TEM images (Fig. 2a–b) showed that the halloysite mineral particles had a cylindrical shape, and the nanotubular particles were hollow and open-ended. The typical powder X-ray diffraction (PXRD) pattern in Fig. 2c indicates the seriously disordered features of the halloysite contributed by the layer stacking. The energy-dispersive X-ray spectrum (EDX) in Fig. 2d shows that the raw mineral had a Si/Al ratio of 1:1 without any other metal cations.

1.3. *Synthesis of zeolite A*

The activation process of the raw minerals, which is used to destroy the layered edge-shared AlO_6 octahedra, is crucial for synthesis of pure zeolite A. Both the temperature and alkaline content can influence the activation process. Normally, high temperature up to 1000°C is needed in order to break the Al–O bonds in an AlO_6 configuration (Lee et al., 1999). In order to decrease the energy requirement, alternatively, alkali can be introduced in the combustion step to activate the mineral, as

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