



Short Communication

Synthesis of macroporous silica biomass nanocomposite based on XG/MgSiO₃ for the removal of toxic ions

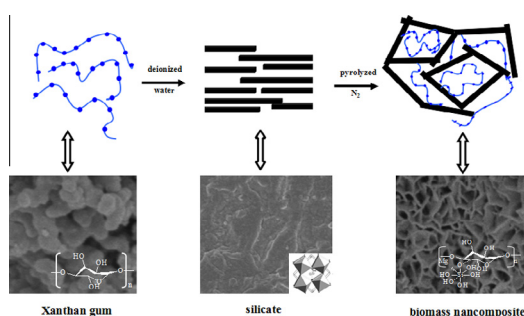
Wei Ma^{*}, Fanqing Meng, Zihong Cheng, Gang Xin, Shibo Duan

Department of Chemistry, Dalian University of Technology, Dalian 116023, PR China

HIGHLIGHTS

- A macroporous nanocomposite biomass has been synthesized based on XG/MgSiO₃.
- The macroporous nanocomposite biomass has revealed a ordered macroporous structure.
- The biomass nanocomposite is an efficient adsorbent for removal of toxic ions.
- The pyrolysis temperature effect on the adsorption capacity of biomass was studied.

GRAPHICAL ABSTRACT



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ABSTRACT

Biomass is known as a low-cost adsorbent and there is a need for the development of synthesis method further increase its efficient applications. In this work, a macroporous nanocomposite biomass was synthesized by natural polymer (Xanthan gum) and silicate. The resulting nanocomposite was characterized with XRD, SEM, TEM, BET and FTIR. The analysis confirmed that the functions silicate groups were successfully introduced and the nanocomposite not only showed a special macroporous structure but also showed a better cation exchange capacity which helps to retain cation ions. What's more, in order to investigate the adsorption capacity of the biomass, adsorption experiments were considered. The experiment results revealed that nanocomposite showed a high-efficiency adsorption capacity to remove toxic ions such as arsenic, chromium, mercury and cadmium.

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1. Introduction

Biomass as a renewable resource which is carbon-rich and low-cost. It has received more attention because of its application in agronomic and environmental fields (Verheijen et al., 2010). Simultaneously, biomass has a great potential for removing ions from aqueous solution contributed to its porous structure and abundant functional groups (Wang et al., 2013). As it has shown in many researches, biomass is particularly effective for the removal of organic compounds like dye (Angin et al., 2013),

antibiotics (Zheng et al., 2013) and naphthalene. What's more, it is also has great adsorption capacity for a variety of toxics like Ni (Han et al., 2013), Pb (Cao et al., 2009), Cr (Wang et al., 2015) and As (Wang et al., 2014).

However, recent study has proven that tedious separation process of the exhausted powdered biomass also has some insufficient such as the effluent and the regeneration/disposal limitation. Therefore, researchers began to use inorganic solids (e.g., zeolite, TiO₂, SiO₂, etc.) to modified the biomass to improve its efficient applications (Antonietti et al., 2004). A method was proposed to fabricate a ferric oxides loaded granular biomass adsorbent. Incorporation of some reducing agent like irons in the biomass matrix to modified it to get magnetic biomass nanocomposite

^{*} Corresponding author. Tel.: +86 (411) 8470 6303; fax: +86 (411) 8470 7416.
E-mail address: chmawv@yahoo.com (W. Ma).

matrix which will facilitate the separation of the exhausted biomass from the effluent (Devi et al., 2014).

Keeping the above facts in mind, it was decided to synthesize modified biomass to improve its adsorption capacity, yet seldom seen study the biomass's structure to company with the adsorption capacity. Therefore, in this study, a new method was provided to modified biomass to get a novel macroporous nanocomposite biomass by nature polymer and silicate. The physicochemical properties, adsorption performance and characteristics of the adsorbents were analyzed by characterization and adsorption experiments.

2. Methods

2.1. Materials

Xanthan gum (XG), a high molecular weight (5×10^6 g/mol) natural polymer which was purchased from Yide company of Dalian (China). Other reagents used in this work were of analytical grade and purchased from Dalian (China). All the reagents used in the study were analytical grade, and all solutions were prepared with deionized water.

2.2. Feedstock preparation

XG (5 g) and $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$ (1.2 g) was dissolved in 20 mL distilled water. The mixture was agitated at 250 rpm by a magnetic stirrer to maintain a uniform concentration in the flask. Then, 30 mL Na_2SiO_3 (30%) was dropwise added. The resultant residue was washed with distilled water to remove the unreacted reactant and was oven dried at 60°C . The dried biomass nanocomposite was stored in air-tight container for further use.

2.3. Biomass production

The feedstock was placed in a railboat reactor of 3 cm long, 1.5 m width and 1 cm height and pyrolyzed in a muffle furnace. Five different pyrolysis temperatures and time (300, 400, 500, 600, 700°C and 1, 1, 1, 0.5 and 0.5 h) were adapted to carbonize each feedstock. These pyrolysis temperatures were choose based on the results of earlier reports. (Zhang and Liu, 2015) Then the reactor was cooled to room temperature and crushed to about 1 mm for adsorption experiments.

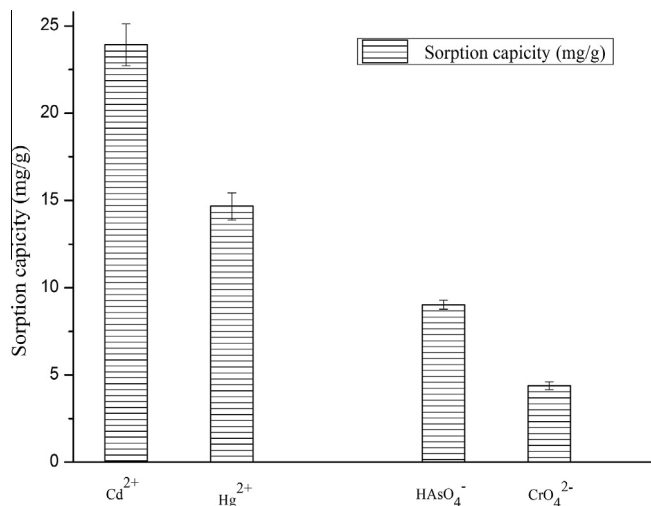


Fig. 1. The adsorption capacity analysis of the composite on different anion and different cation.

2.4. Biomass characteristic

Macroporous silica biomass nanocomposite and silicate were characterized by using Fourier transform infrared spectrometer (Bruker TENSOR 27 FTIR). Samples were prepared by the KBr disk method and the spectra were recorded in $400\text{--}4000\text{ cm}^{-1}$ range. The structure of the biomass was characterized by Transmission electron microscopy (TEM) on a Tecnai G2-F20 and microscope Scanning electron microscope (SEM) with a JEOL-JSM-5600LV Scanning Microscope. X-ray diffraction (XRD) (X'pert PRO analytical B.V., Netherlands) were recorded at 2θ ($^\circ$).

2.5. Adsorption experiments

Adsorption experiments were conducted using the biomass nanocomposite by batch experiments in a shaker. Unless otherwise stated the parameters were: temperature 293 K, solution volume 50 mL, sorbent dose 0.05 g, contact time 150 min, initial pH 6, ion concentration 20 mg/L. The initial pH effect was varied from 2.0 to 7.0 using diluted of H_2SO_4 or NaOH to investigate the effect of initial solution pH, and the influence of initial Hg (II) concentrations was investigated in the range of 5–25 mg/L which was carried out in different content (5, 10, 15, 20 and 25 mg/L), and other conditions kept the same. These adsorption processes were repeated three times for each current value. The concentrations of Hg^{2+} ions in adsorption experiments were measured using a PinA-Ade 900-T atomic adsorption spectrometer (PerkinElmer Inc., USA).

3. Results and discussion

3.1. Biomass nanocomposite properties

The FTIR spectra of the macroporous silica biomass nanocomposite and unmodified biomass were analyzed in the range of $400\text{--}4000\text{ cm}^{-1}$. The biomass nanocomposite sample showed a characteristic absorption band at 1089.89, 950, 792.294 and 466.773 cm^{-1} corresponding respectively to the stretching vibrations of ν (Si–O–Si), ν (Si–O), ν (Si–C) and ν (Mg–O) (Bruce and Sen, 2005; Besling and Goossens, 1998; Hofmeister et al., 2003) which implying modified functions magnesium silicate groups were successfully introduced. What's more, peak observed in raw nature polymer at 2928 cm^{-1} disappeared at temperatures 300°C and above, showing a methylene group $-\text{CH}_2-$ in nature polymer volatile at lower temperature. Besides, another acid group showed a declining trend is vibration of $\text{C}=\text{O}$ for lactone, peak at 1655 cm^{-1} on the raw nature polymer disappeared after heating. Vibration in the frequency range from 1100 to 1500 cm^{-1} reflect peaks for ester and carboxyl groups declined with the increase of pyrolysis temperature. The reduction of this acidic functional groups of raw nature polymer at high temperature shifted the basic sites in the form of unsaturated bonds with electron donating properties. These functional groups of pore surface negative charge property contributes to better cation exchange capacity that help to uptake cation ions.

The SEM and TEM images revealed that the structure of the biomass nanocomposite was ordered macroporous frameworks where the silicate and unmodified biomass was not found on the surfaces (Fig. S1). What's more, the macroporous nanocomposite was composed of the nano frameworks unit. In addition, the SEM images revealed that the macroporous structure of the nanocomposite was appeared when pyrolysis temperature up to 400°C . Moreover, the macroporous structure become more clearly and reveals a three-dimensional space structure when the pyrolysis temperature increased to 500°C . At 600°C , the macroporous structure of the biomass revealed the ordered diamond macroporous

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