



Lead and copper removal from aqueous solutions using carbon foam derived from phenol resin



Chang-Gu Lee^a, Jun-Woo Jeon^a, Min-Jin Hwang^a, Kyu-Hong Ahn^a, Chanhyuk Park^a, Jae-Woo Choi^{a,b,*}, Sang-Hyup Lee^{a,c,*}

^a Center for Water Resource Cycle Research, Korea Institute of Science and Technology, Hwarangno 14-gil 5, Seongbuk-gu, Seoul 136-791, Republic of Korea

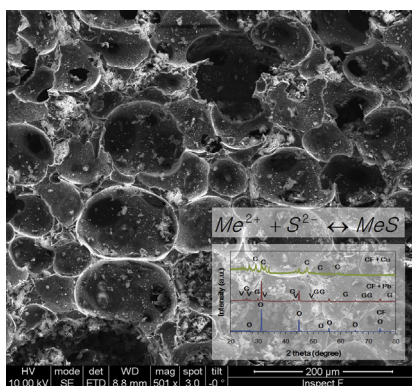
^b Department of Energy and Environmental Engineering, University of Science and Technology (UST), Daejeon 305-350, Republic of Korea

^c Green School, Korea University, 145, Anam-ro, Seongbuk-gu, Seoul 136-701, Republic of Korea

HIGHLIGHTS

- Phenolic resin-based carbon foam is a low-cost adsorbent alternative.
- Lead and copper removal from aqueous solution is possible with carbon foam.
- Key mechanism of lead and copper removal on carbon foam is surface precipitation.
- Maximum sorption capacities were 460.50 mg g⁻¹ for lead and 212.14 mg g⁻¹ for copper.

GRAPHICAL ABSTRACT



ARTICLE INFO

Article history:

Received 2 October 2014
Received in revised form 23 February 2015
Accepted 23 February 2015
Available online 24 March 2015

Handling Editor: Min Jang

Keywords:

Carbon foam
Lead removal
Copper removal
Batch experiments
Surface precipitation

ABSTRACT

Phenolic resin-based carbon foam was prepared as an adsorbent for removing heavy metals from aqueous solutions. The surface of the produced carbon foam had a well-developed open cell structure and the specific surface area according to the BET model was 458.59 m² g⁻¹. Batch experiments showed that removal ratio increased in the order of copper (19.83%), zinc (34.35%), cadmium (59.82%), and lead (73.99%) in mixed solutions with the same initial concentration (50 mg L⁻¹). The results indicated that the Sips isotherm model was the most suitable for describing the experimental data of lead and copper. The maximum adsorption capacity of lead and copper determined to Sips model were 491 mg g⁻¹ and 247 mg g⁻¹. The obtained pore diffusion coefficients for lead and copper were found to be 1.02 × 10⁻⁶ and 2.42 × 10⁻⁷ m² s⁻¹, respectively. Post-sorption characteristics indicated that surface precipitation was the primary mechanism of lead and copper removal by the carbon foam, while the functional groups on the surface of the foam did not affect metal adsorption.

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* Corresponding authors at: Center for Water Resource Cycle Research, Korea Institute of Science and Technology, Hwarangno 14-gil 5, Seongbuk-gu, Seoul 136-791, Republic of Korea. Tel.: +82 2 958 5820; fax: +82 2 958 5839 (J.W. Choi). Tel.: +82 2 958 6945; fax: +82 2 958 5839 (S.H. Lee).

E-mail addresses: plead36@kist.re.kr (J.-W. Choi), ysisanghyup@kist.re.kr (S.-H. Lee).

1. Introduction

Carbon foam is a sponge-like carbon material with high strength, light weight, high thermal/electrical management, and a large surface area with open cell structure (Chen et al., 2006a; Tondi et al., 2009). Because of these inherent properties, carbon

foam is used as catalyst support, filter for molten metal and corrosive chemicals, porous electrodes, and impact/energy/acoustic absorbers (Grujicic et al., 2006; Kim and Cunningham, 2010; An et al., 2011; Kang et al., 2011; Chun et al., 2012; Emmel and Aneziris, 2012; Shao et al., 2013; Amaral-Labat et al., 2013). Carbon foam is also applied as a biological filter in water treatment systems and as an adsorbent in gas- or liquid-phase environments (Chen et al., 2006b; Bao et al., 2011; Liu et al., 2013; Burke et al., 2013).

Initially, carbon foam was produced as a refractory thermal insulating material in 1964 by Ford (1964). Over the following decades, various types of carbon foam were produced depending on the precursors and manufacturing processes. They are classified as graphitic and non-graphitic carbon foam. Graphitic foam was derived from graphitization of coal, pitched at a temperature of at least 2600 °C (Song et al., 2012; Focke et al., 2014). They have very good thermal and electrical conductivity as a result of the high degree of graphitization. Non-graphitic foam was prepared by carbonization of organic polymers such as phenol formaldehyde and sucrose (Prabhakaran et al., 2007; Lei et al., 2010). They have low thermal conductivity and can be used as thermal insulation material. In addition, non-graphitic foam has potential applications as low-cost carbon foam due to the low-cost of manufacture. Low-cost adsorbent requires a simple manufacturing process, as well as a large amount of precursor that can be easily obtained. Recently, much research has been carried out to produce low-cost carbon foam though the use of commercially available phenolic resin (Manocha et al., 2010; Wu et al., 2011). The price of phenolic resin is less than US \$ 1 kg⁻¹.

Carbonaceous adsorbents are known as one of the most effective methods for heavy metal wastewater treatment. A large number of studies were carried out on the removal of heavy metals in water using activated carbon or modified activated carbon (Zhu et al., 2009; Fu and Wang, 2011). Activated carbon is the most commonly used adsorbent, but it is relatively expensive. The price of activated carbon vary depending on the quality, it is about US \$ 9 kg⁻¹ (Kurniawan et al., 2006). In recent years, many studies have been conducted on the development of heavy metal adsorbents using low-cost material, such as agricultural by-product or biomass (Ahmaruzzaman and Gupta, 2011; Inyang et al., 2012). Carbon foam has rarely been used for heavy metal adsorption, despite its affordability (about US \$ 3 kg⁻¹) (Tondi et al., 2010a). Burke et al. (2013) conducted the only know study using carbon foam chemical oxidation for lead ion removal from aqueous solution. In the current study, a phenolic resin-based carbon foam was prepared as a low-cost adsorbent for lead and copper ion removal from aqueous solution. The chemical composition and surface characteristics of the prepared adsorbent were analyzed. Batch adsorption experiments were conducted to evaluate the removal capacity of heavy metals. The removal mechanisms of copper and lead were also analyzed.

2. Materials and methods

2.1. Preparation of carbon foam

Phenolic resin-based carbon foam was supplied from Smithers-Oasis Korea Co. Ltd. Firstly, the procedure involved the addition of 1.0 kg of phenol to a four-necked reactor, 1.5 kg of formaldehyde was then poured into the reactor with slow stirring followed by 40 g of a base catalyst, and the contents were stirred for a further 2 h at 75 °C. The mixture was cooled to 35 °C and neutralized to pH 6–7 using dilute sulfuric acid. The moisture content was then adjusted to 9% through dehydration under vacuum with reduced pressure. Secondly, 2 g of alkyl ether type surfactant was mixed

with the prepared phenolic resin and the mixture was stirred thoroughly in a cylindrical mold at a low stirring speed. Thirdly, 80 g of organic acid curing agent and 20 g of hydrocarbon foaming agent were added to the mixture and the stirring rate was increased to 1000 rpm. The synthesized foam was aged in a convection oven at 60 °C. Finally, the dried foam was put into an electric furnace and the air was removed using a vacuum pump. The sample was carbonized at a heating rate of 5 °C min⁻¹ under a nitrogen stream of 100 mL min⁻¹ up to 900 °C, held at the final temperature for 2 h, and then slowly cooled in the nitrogen atmosphere.

2.2. Characterization of carbon foam

The chemical composition of the carbon foam was determined using an X-ray fluorescence spectrometer (XRF, ZSX Primus II, Rigaku, Japan). The surface morphology of the sample was investigated using a scanning electron microscope (SEM, Inspect F50, FEI, USA) at 10.00 kV. Nitrogen gas (N₂) adsorption-desorption experiments were performed using a surface area analyzer (nanoPOROSITY-XQ, Mirae Scientific Instruments, Korea) after pretreatment of the sample at 200 °C. The surface area, mesopore volume, and micropore volume were calculated from the N₂ isotherm according to the methods of Brunauer-Emmett-Teller (BET), Barrett-Joyner-Halenda (BJH), and Horvath-Kawazoe (HK). The potentiometric titration method of Parks and Bruyn (1962) was used to determine the point of zero charge (pH_{PZC}). One gram of carbon foam was added to 250 mL beakers with 100 mL of electrolyte solution (0.1, 0.01, and 0.001 M NaCl). The solution was stirred for 2 h and then the pH was measured after each increment of 0.1 M HCl or 0.1 M NaOH. The results of the titration were plotted and the pH_{PZC} was determined using a graphical method. The crystalline structure of the sample was measured by an X-ray diffractometer (XRD, D-max 2500/PC, Rigaku, Japan) with Cu K α radiation and a fixed power source (40 kV and 200 mA). The diffraction data were collected over a 2 θ range between 20° and 80°, at a scanning rate of 0.5° min⁻¹. Fourier transform infrared spectroscopy (FTIR, Infinity Gold, Thermo Mattson, USA) was used to analyze the change in surface functional groups on the carbon foam before and after the sorption experiments.

2.3. Batch adsorption experiments

An initial evaluation of the sorption ability of carbon foam was performed using a mixed heavy metal solution containing lead, cadmium, zinc, and copper. The concentration of each metal was adjusted to 50 mg L⁻¹ through dilution using standard solution purchased from Kanto Chemical Inc. Approximately 0.03 g of sorbent was added to a 250 mL Erlenmeyer flask with 200 mL of the heavy metal solution at room temperature. After shaking in a rotary shaker for 24 h, the solution was sampled using a 0.45 μ m syringe filter. The concentration of each metal in the sampled solution was analyzed using an inductively coupled plasma-optical emission spectrometer (ICP-OES, Prodigy ICP, Teledyne Leeman Labs, USA).

Additional batch experiments were carried out to analyze the sorption properties of lead and copper. The desired concentration of lead and copper were prepared by diluting the stock solutions (1000 mg L⁻¹), which were made from lead nitrate (Pb(NO₃)₂, Sigma-Aldrich, USA) and copper chloride dihydrate (CuCl₂·2H₂O, Sigma-Aldrich, USA). The solution pH was adjusted close to neutral in consideration of the solubility of each metal. The solubility of lead and copper calculated from Visual MINTEQ 3.0 was shown in Fig. S2. The solution pH of lead and copper were adjusted 7 and 5, respectively. Kinetic batch tests were performed in 250 mL Erlenmeyer flasks containing 0.03 g of carbon foam with 200 mL of the diluted solution. Sorption experiments were performed

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