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Enhancement of Pb^{2+} removal by activating carbon spheres/activated carbon composite material with H_2O vapor

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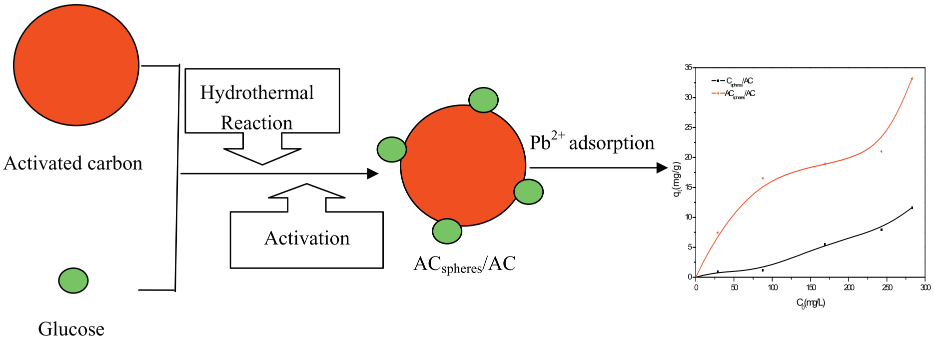
HIGHLIGHTS

- $C_{spheres}$/AC was activated by H_2O vapor.
- The surface area of $C_{spheres}$/AC was remarkably increased from 89.9 to 920.9 m^2/g after activation.
- The Pb^{2+} adsorption capacity was enhanced observably after activation and the maximum adsorption capacity was achieved to 33.2 mg/g.
- The adsorption process was obeyed the monolayer adsorption mechanism and involved physical and chemical adsorption.
- The surface area was main factor that affected the adsorption capacity.

GRAPHICAL ABSTRACT

H_2O vapor was used to activate the $C_{spheres}$/AC composite material which was prepared from activated carbon (AC) and glucose by hydrothermal reaction. The surface area of $C_{spheres}$/AC was remarkably increased from 89.9 to 920.9 m^2/g after activation, about 10 times, while the hydroxyl groups decreased from 46.2 to 35.6%. The Pb^{2+}adsorption capacity was enhanced observably after activation and the maximum adsorption capacity was achieved to 33.2 mg/g, about 3 times of $C_{spheres}$/AC. The adsorption process of Pb^{2+} involved both physical adsorption and chemical adsorption, while the surface area was the main factor that affected the adsorption capacity.



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ABSTRACT

H_2O vapor was used to activate the carbon spheres/activated carbon ($C_{spheres}$/AC) to enhance its adsorption performance and prepare a composite of activated carbon spheres embedded in activated carbon ($AC_{spheres}$/AC). The influence of H_2O vapor activation on morphology, pore structure and surface element composition of $C_{spheres}$/AC were studied by scanning electron microscope, fourier transform infrared spectroscopy, N_2 sorption and X-ray photoelectron spectroscopy. It was found that the specific surface area was remarkably increased from 89.9 to 920.9 m^2/g after activation, about 10 times, while the hydroxyl groups decreased from 46.2 to 35.6%. The adsorption behavior of Pb^{2+} was investigated and evaluated by Freundlich and Langmuir models. The result showed that the adsorption capacity was enhanced observably after activation and the maximum adsorption capacity of $AC_{spheres}$/AC was achieved to 33.2 mg/g, about 3 times of $C_{spheres}$/AC.

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The adsorption process occurred on surface of absorbent was obeyed monolayer mechanism and in micropores was multilayer adsorption. The specific surface area was main factor that affected the adsorption capacity.

1. Introduction

Water pollution caused by heavy metal (Pb^{2+}, Hg^{2+}, Mn^{2+}, et al.), has become a serious environmental problems in the world. Adsorption is considered as a reliable technology for controlling water pollution [1]. Because of their outstanding adsorption performance, activated carbon (AC) has been treated as an essential material and widely used as adsorbents and catalysts supports in a growing number of application fields such as chemicals purification, food processing, environment protection [2–4]. As an adsorbent, the adsorption performance of AC is linked with the presence of the surface functional groups and the specific surface area. The specific surface area is an important factor in physical adsorption, while the surface functional groups affect chemical adsorption and surface reaction primarily [5,6]. Therefore, modification of surface properties would be an effectively method to enhance the adsorption capacity of AC which could expand its application and tackle the environmental challenges.

Recently, research of carbon materials with special morphology and function has become a hotspot. Carbon spheres, prepared from polyhydric sugar through hydration method, have been highlighted in various research fields because of their abundant functional groups such as —C—OH and —COOH [7,8]. Similar with most carbon materials, carbon spheres have high mechanical strength and good chemical stability [9]. A more important fact is that the intrinsic properties of carbon spheres can be purposeful controlled by changing the production or modifying condition [10–13]. But due to its undeveloped pore structure and large filtration resistance, the application of carbon spheres in pollution controlling is limited seriously.

As we known, AC has huge specific area but less surface functional groups, while carbon spheres are rich in functional groups especially —OH. Therefore, combined carbon spheres with AC can be an effective modification method to increase acidic functional groups [14,15]. Liu et al. prepared carbon spheres/activated carbon using activated carbon and glucose by hydrothermal treatment. The obtained carbon spheres/activated carbon showed small specific surface and nearly was a non-pore materials, because carbon spheres loaded on the surface of activated carbon and blocked the pore structure [16]. So far, there were a great number of studies regarding the synthesis of carbon spheres, which focused on the effect of synthesis temperature, time and concentration on the particle size of carbon spheres. But the effect of activation and modification of $C_{spheres}$/AC on adsorption performance were lacked. Therefore, activating of $C_{spheres}$/AC will be an effective method to enhance its adsorption performance and create a novel composite adsorbent. In this study, $C_{spheres}$/AC was activated by H_2O vapor and characterized using scanning electron microscope, fourier transform infrared spectroscopy, N_2 sorption and X-ray photoelectron spectroscopy. The adsorption capacity of Pb^{2+} ions on samples and adsorption mechanism were investigated.

2. Materials and methods

2.1. Synthesis of $C_{spheres}$/AC

2.0 g activated carbon (produced by MU LIN SEN Activated Carbon Co. Ltd.) was added to 1.0 M glucose aqueous solution (120 mL) and sonicated for 20 min, then shaked at 25 °C for 24 h to form homogeneous dispersion. The homogenous dispersion was put into a Teflon-sealed autoclave vessel (250 mL) and treated at 160 °C for 4 h, then cooled and washed with alcohol and distilled water. After dried at 105 °C for 4 h and marked as $C_{spheres}$/AC.

2.2. Activation of $C_{spheres}$/AC

Under the nitrogen atmosphere, $C_{spheres}$/AC was put into tube furnace and heated to activated temperature (750, 850 °C) at heating rate about 5 °C/min. After reaching the activation temperature, nitrogen gas was stopped and H_2O vapor (0.75, 1.0 mL/min) was used to activate for 60 min. The samples were prepared and marked as $AC_{spheres}$/AC1 (750 °C, 0.75 mL/min), $AC_{spheres}$/AC2 (750 °C, 1.0 mL/min), $AC_{spheres}$/AC3 (850 °C, 0.75 mL/min) and $AC_{spheres}$/AC4 (850 °C, 1.0 mL/min).

2.3. Characterization

The surface morphology of samples were observed using Hitachi S-3400 scanning electron microscope. Before observation, samples were coated with gold in E-1010 Ion sputter. Specific surface area (S_{BET}) and pore distribution of the samples were determined by nitrogen adsorption-desorption isotherms measured in a Micromeritics ASAP 2020 apparatus at 77 K. Before analysis, the samples were degassed under N_2 flow at 350 °C for 2 h in a vacuum at 27 Pa. The S_{BET} of the samples were estimated by BET method, the total pore volumes were calculated from the volume of N_2 adsorbed at $p/p_0 = 0.1$ and the pore size distribution of samples were calculated by the Barret Joyner Halenda (BJH) model [17]. Fourier transform infra red spectroscopy (FTIR) analysis of samples was performed on a Nicolet DXC20 FTIR spectrometer. Samples with particle size <45 μm were dried for 24 h at a temperature of 100 °C and then mixed with finely divided KBr at a ration of 1:100. The element composition and surface chemistry of samples were detected by X-ray photoelectron spectroscopy (XPS) on a PHI5000V using C1s corrected energy. A pass energy of 100 eV was used in recording the scan the analysis. The sample was dried at 283 K for 24 h before analysis.

2.4. Adsorption procedure

The adsorption experiments were carried out in a volumetric flask using a horizontal axis mechanical agitator. Each 0.2 g sample contacted with 100 mL Pb^{2+} solution for 24 h. Pb^{2+} solutions were prepared by dissolving a given capacity of $Pb(NO_3)_2$ in distilled water. Pb^{2+} concentration was detected by Inductively Coupled Plasma Emission Spectrometer (ICP). The adsorption capacity of Pb^{2+} was calculated by the following Eq. (1).

$$q_e = \frac{(C_0 - C_e) \times V}{m} \tag{1}$$

where q_e is the adsorption capacity (mg/g), C_o and C_e are the initial and equilibrium concentration of Pb^{2+} in solution (mg/L), V is the volume (mL) and m is the weight of sample (g).