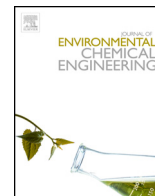




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Ammonium persulfate oxidized activated carbon fiber as a high capacity adsorbent for aqueous Pb(II)

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ABSTRACT

An activated carbon fiber was oxidized with ammonium persulfate solutions (APS) and used for the adsorption of Pb(II) from aqueous solutions. A comprehensive study on the effect of the oxidation process conditions—oxidant concentration, oxidation temperature and time—on the obtained fibers Pb(II) adsorption capacity and their chemical and physical properties was done. Samples were characterized by elemental analysis, Boehm titration, N₂ adsorption–desorption isotherms and infrared spectroscopy. Adsorption isotherms were obtained for the activated carbon fibers and for two commercial ion-exchange resins, and they showed good agreement with the Langmuir model. For the first time, an activated carbon fiber with a high adsorption capacity for aqueous Pb(II) was obtained (2.70 mmol g⁻¹, ca. 559 mg g⁻¹). This value was more than 10 times higher than that of the pristine fiber and superior to those of the commercial ion-exchange resins. The adsorption kinetics data of the oxidized activated carbon fiber were studied by different kinetic models and found to be better described by the pseudo-second order model, while the adsorption equilibrium could be reached in less than 2 h. It was confirmed that the adsorption process mainly occurred by an ion-exchange mechanism. The obtained results showed that APS oxidation is capable of producing activated carbon fibers with high adsorption capacities for aqueous Pb(II), making them promising materials for industrial wastewater treatment applications.

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1. Introduction

Lead is an important heavy metal used since ancient times in the manufacture of many products such as tubes and pipes; and it is still widely used to produce electronics, crystal glasses, and batteries [1]. Unfortunately, it is a very toxic pollutant, that when enters into the body accumulates in the organs causing several health problems related to the nervous system, anemia and high blood pressure. Exposure to high levels of lead can damage the brain and kidneys and ultimately cause death [2]. In China, because of the rapid economic growth and consequently the increasing demand for cars and power-assisted bicycles, the annual consumption of lead has been increasing at a high rate. It has been found that people living near lead mining areas present high blood lead levels, and they are affected by lead poisoning, especially

children [1,3]. Hence, the control of emissions from lead industries and treatment of their wastewater is crucial.

The most common method for lead wastewater treatment is precipitation; although it is cost effective, requires the use of many chemicals that ends in a sludge of big volume that needs additional exhaustive treatments [4]. Thus, it is of great importance to search for more effective, economical and environmental sustainable treatment methods, like reverse-osmosis, adsorption and ion-exchange [5]. Among these, adsorption is a very practical method, and the materials to be used as adsorbents can be chosen and developed to meet each specific treatment conditions. A very widely used and studied adsorbent is activated carbon (AC); its production can be economical and simple, having the possibility to use precursors from petroleum derivatives to biomass materials [6–10].

To improve the AC materials adsorption capacity and efficiency for their use in the removal of heavy metals ions, several modifications of their surface have been studied. These procedures include the introduction of acidic oxygen functional groups [11,12] and nitrogen groups [13], loading of nanoparticles [14–16] and the addition of anionic surfactants [17] and biopolymers like alginate [18]. However, the adsorption capacities of these materials are

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relatively low (0.08–0.67 mmol g⁻¹), and in most cases, their production requires several steps and long periods of time to be concluded.

Oxidation stands as a simple modification procedure that can be achieved in short periods of time; and it enhances the materials adsorption capacity for heavy metal ions, caused by the introduction into the carbon surface of oxygen-containing groups with ion-exchange properties [19,20]. While almost all of these studies have shown relatively low adsorption capacities for heavy metal ions, recently we have proved that after studying in detail the oxidant treatment conditions, it is possible to obtain high oxidized ACs with high adsorption capacities for aqueous lead [21,22].

In the present research, we investigated the oxidation process of activated carbon in the form of a fiber (ACF). This material has recently gained interest as an adsorbent for the removal of aqueous pollutants, since it possesses a large surface area, easy handling, and a bigger bulk volume than that of ordinary pelleted or powdered AC, so fast adsorption and desorption rates can be obtained [23–25]. Whereas there are some studies on the oxidation of ACFs for their use in heavy metals removal [24, 26–30], for the present a research that studies the influence of the oxidation process conditions in detail has not been done. This is the reason why the limited available data only report adsorption capacities for aqueous lead that do not exceed values of 0.3 mmol g⁻¹ [24,26,27].

Therefore, in the present study, we studied the influence of the oxidation process conditions of an ACF, in order to obtain materials that possess high adsorption capacities for aqueous lead and at the same maintain the exceptional characteristics of the ACFs. We evaluated the influence of oxidation time, oxidation temperature and oxidant concentration over the obtained fibers physical and chemical properties, as well as on their adsorption capacity for aqueous lead. This is the first time that a detailed study on this matter was done for ACFs oxidation and that exceptional high adsorption capacities were obtained for this type of adsorbents. Furthermore, we compared the maximum adsorption capacity for Pb(II) of the fibers with that of commercial ion-exchange resins and studied the adsorption process in order to better understand the action of the fibers as adsorbents for aqueous lead.

2. Materials and methods

2.1. Reagents

Activated carbon fiber KF1500LDA (KF) was purchased from Toyobo Co. Ltd., Japan. Ion-exchange resin IR120 Na form and KBr IR grade were acquired from Acros Organics (Belgium). Ion-exchange resin Amberlite IRC86 hydrogen form was purchased from Sigma-Aldrich (France). All other reagents were purchased from Kanto Chemical Co., Inc. (Japan), used as received and their solutions were prepared using pure water.

2.2. Oxidation of the ACF

A commercially available activated carbon fiber, KF, was used as the starting material. Ammonium persulfate solutions (APS) were prepared by dissolving different amounts of (NH₄)₂S₂O₈ in 1 mol L⁻¹ H₂SO₄. In a 300 mL beaker, 4 g of the fiber was put in contact with 240 mL of APS solutions of different concentrations. In order to prevent the fiber from floating, an arrangement as the one in Fig.S.1 was used. The temperature and agitation were regulated using a water bath stirrer. After oxidation, the oxidized samples were washed with pure water in a Soxhlet apparatus for various cycles and finally dried in an oven at 110 °C. The oxidized samples (Ox-ACFs) were denoted as *a*APS-*b*-*c*, where *a*, *b* and *c*, indicate the oxidant solution concentration in mol L⁻¹, the oxidation

temperature in °C and the oxidation time in hours, respectively. For example, the sample 1.4APS-30-72 was done under the following oxidation conditions: 1.4 mol L⁻¹ APS, at 30 °C for 72 h.

2.3. Characterization of the samples

2.3.1. Textural characterization of the fibers

Textural characteristics of the fibers were evaluated by B.E.T. method using N₂ adsorption and desorption isotherms at -196 °C, measured with a surface area analyzer SA3100 (Beckman Coulter, INC., U.S.A.).

To study the effect of the surface functional groups on the textural properties, the sample 1.4APS-30-72 was outgassed in a tubular furnace at 400 °C for 1 h, with a He flow of 200 mL min⁻¹; and later its N₂ adsorption and desorption isotherms were measured as described above.

2.3.2. Boehm titration

Modified Boehm titration method [31] was used to determine the amount of oxygenated surface functional groups. In a 100 mL Erlenmeyer flask, 0.1 g of each sample was placed with 25 mL of the following solutions: 0.1 mol L⁻¹ NaOH, 0.05 mol L⁻¹ Na₂CO₃, 0.1 mol L⁻¹ NaHCO₃ and 0.1 mol L⁻¹ HCl. The flasks were stirred for 4 days and then 5 mL of each solution was titrated with 0.05 mol L⁻¹ HCl, except for the ones with 0.1 mol L⁻¹ HCl which were back titrated adding first 10 mL of 0.1 mol L⁻¹ NaOH. The amount of acidic sites of various types was calculated assuming that NaOH neutralizes carboxy, phenol and lactone groups; Na₂CO₃ neutralizes carboxy and lactone groups; and NaHCO₃ neutralizes only carboxy groups. The number of basic sites was calculated from the amount of HCl that reacted with the sample.

2.3.3. Elemental analysis

The elemental composition of carbon, hydrogen and nitrogen was measured by PE2400 microanalyzer (Perkin-Elmer, U.S.A.). Oxygen composition was determined by balance assuming that all other elements were oxygen.

2.3.4. pH of point of zero charge

The pH of the point of zero charge (pH_{pzc}) of the samples was determined by the pH drift method [32]. In an Erlenmeyer flask, 0.1 g of the samples and 25 mL of 0.1 mol L⁻¹ NaCl solution were agitated at 100 rpm for 24 h at 25 °C. Before addition, the pH values of the NaCl solutions were adjusted between 1.5 and 12 by adding either 0.1 mol L⁻¹ HCl or NaOH. The initial solution pH was plotted against the final pH, and the point at which both values were the same was determined as the pH_{pzc}.

2.3.5. Fourier transform infrared spectroscopy (FTIR)

IR spectra were collected using an FTIR spectrometer IR Affinity-1 (Shimadzu, Japan) with a diffuse reflectance unit. The powder samples were mixed with KBr, and the instrument resolution was set to 4 cm⁻¹.

2.4. Adsorption experiments

Adsorption experiments for Pb(II) ion in aqueous solution were done in a batch system. Each sample was dried at 110 °C for 1 h in an oven before use. Pb(II) solutions were prepared by dissolving Pb(NO₃)₂ in pure water. In a conical flask, 50 mg of each sample was added to 25 mL of the Pb(II) solutions and agitated at 100 rpm over 24 h at 25 °C. The solution pH was adjusted between 4.5–5.0 with different concentrations of NaOH solutions in order to obtain the maximum amount of adsorbed Pb(II), as described in other studies [12,29,33] and as confirmed by our preliminary experiments (Fig S.2.). The Pb(II) ion concentration of the solutions before and after

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