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Research article

Applications of magnetic hybrid adsorbent derived from waste biomass for the removal of metal ions and reduction of 4-nitrophenol

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ABSTRACT

The use of industrial waste to synthesize materials of technological interest is a rational way to minimize or solve environmental pollution problems. This work investigates the adsorption of cadmium and lead ions by magnetic hybrid adsorbents synthesized using the *in natura* biomasses coconut mesocarp (CCFe), sawdust (SAFe), and termite nest (TEFe) for the organic phases and magnetic cobalt ferrite as the inorganic phase. The formation of a cobalt ferrite phase was confirmed by XRD. The use of XRD and FTIR analyses revealed the presence of organic matter in the structure of the material. Removal assays performed at different pH values (2.0–8.0) showed the effectiveness of the adsorbent for the removal of Pb^{2+} at pH 3.0 and Cd^{2+} at pH 4.0. The adsorption processes showed fast kinetics, with removal of 79–86% of Pb^{2+} and 49% of Cd^{2+} within only 5 min, and removal of 92–96% of the metal species at equilibrium. In the case of cadmium, the hybrid sorbents (CCFe, SAFe, and TEFe) showed high removal capacity after three reuse cycles, while the removal of lead decreased from 99% to 40%. The adsorbent matrices saturated with the recovered cadmium and lead ions showed excellent catalytic performance in the reduction of 4-nitrophenol, with 99.9% conversion within 43–56 s. The materials showed high capacities for reuse in three successive reduction cycles. The findings highlight the effectiveness of an industrial symbiosis approach to the development of new technologically important materials.

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1. Introduction

There is increasing interest in the use of byproducts or waste biomaterials from large-scale industrial operations and agriculture, which can provide both economic and environmental benefits (Owlad et al., 2010; Xu et al., 2016; Cheng et al., 2016). Amongst other uses, such biomaterials can be employed for the sorption of pollutants, benefiting from their unique chemical composition, widespread abundance, renewability, and low cost (Araújo et al., 2013; Cunha et al., 2015; De Jesus et al., 2017).

The use of these wastes to produce new materials provides them with added value and contributes to reducing environmental impacts, since they are traditionally often either incinerated, hence increasing the production of carbon dioxide and other greenhouse gases, or are dumped in the environment (De Jesus et al., 2017).

A technical limitation on the use of these materials as adsorbents concerns the procedures necessary to separate adsorbents from aqueous solutions, especially when non-biodegradable materials are involved, in order to avoid problems of secondary pollution. This separation process is often considered as one of the main technological challenges in environmental remediation (Reddy and Yun, 2016).

Adsorption using new classes of hybrid materials has emerged as a promising option for the treatment of pollutants, and materials that possess magnetic properties are especially attractive. In addition to their magnetic characteristics, these materials can be synthesized with functional groups that enable them to interact with different classes of pollutants.

Magnetic hybrid materials synthesized using biomass as the organic phase have considerable potential for the specific sorption of a range of pollutants, including metals (Witek-Krowiak et al., 2011; Xu et al., 2011; Bhatnagar et al., 2013). The use of magnetic materials as adsorbents enhances the technique, because the adsorbent can be easily removed from the aqueous medium by the simple application of an external magnetic field, enabling recovery of the adsorbate and reuse of the adsorbent following the

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desorption process. This facilitates the implementation of industrial symbiosis procedures, which constitute one of the foundations of sustainable development. (Ballav et al., 2014; Dey et al., 2014).

Heavy metal pollutants are of concern due to their persistence in the environment, and cadmium and lead are among the heavy metals often found in different environmental media (Araújo et al., 2013; Novais et al., 2016; USGS, 2010).

The adsorbents and adsorbates obtained after the treatment of effluents are potential secondary pollutants and can be defined as wastes. One option for their productive use requires the development of techniques to enable their incorporation in the production chain.

There have been few studies concerning the use of wastes rich in metal species as raw materials for the synthesis of catalysts. Greater focus is needed in this area, because the reuse of wastes to manufacture technologically important products is one of the pillars of sustainable development. It provides added value to the waste, attracting the attention of businesses that can benefit from an additional revenue stream. Furthermore, catalysts are used in many sectors of industry, leading to a substantial demand for the metal species employed in their manufacture (Cunha et al., 2015; Cruz et al., 2017).

There is currently considerable interest in the development of efficient and inexpensive catalysts suitable for the reduction of aromatic nitro compounds, especially nitrophenols, which are important pollutants present in industrial and agricultural waste waters. The United States Environmental Protection Agency (USEPA) has established a maximum permitted concentration of nitrophenol in natural waters of 10 ng L^{-1}, due to the toxic and carcinogenic properties of the compound. Furthermore, the catalytic reduction of nitrophenol produces aminophenol, which is one of the principal inputs used in the synthesis of many industrial dyes, pharmaceutical products, and certain other biologically active compounds, and is also employed as a corrosion inhibitor in paints and fuels (Takasaki et al., 2008; Ibrahim et al., 2016; Costa et al., 2016).

The purpose of this work was therefore to develop sustainable magnetic adsorbent matrices based on hybrid materials synthesized using *in natura* biomasses that would otherwise represent environmental liabilities. The materials were studied in terms of their efficiencies in the removal of Cd^{2+} and Pb^{2+}, and evaluation was made of the catalytic potentials of these recovered species and the saturated adsorbents in the reduction of 4-nitrophenol to aminophenol.

2. Materials and methods

2.1. Biomasses and pretreatment

Samples of coconut mesocarp and sawdust were obtained from industries located in the city of Aracaju, the capital of Sergipe State, Brazil. A termite nest was collected from an Aroeira tree (*Schinus terebinthifolia*). The biomasses were washed with distilled water and dried at ambient temperature.

2.2. Synthesis procedure

The *in natura* coconut mesocarp, sawdust, and termite nest used to synthesize the magnetic hybrid matrices were denoted CCFe, SAFe, and TEFe, respectively. The synthesis was performed with 3.125 g of biomass in 50 mL of an aqueous solution of $Fe(NO_3)_3 \cdot 9H_2O$ (100 mmol) and $Co(NO_3)_2 \cdot 6H_2O$ (50 mmol). The pH of the medium was adjusted to 9.0 and the system was kept under agitation for 30 min, at ambient temperature. According to the reduction potential diagrams of cobalt and iron, at pH 9.0, the Fe and Co nitrates dissolved in solution produce solid precipitates such as FeO(OH), $Co(OH)_2$, and $Co(OH)_3$ (Garcia et al., 2008). The gel obtained was heated to 100 °C for 24 h in order to eliminate water and obtain a xerogel (Garcia et al., 2008).

2.3. Characterization

X-ray diffraction (XRD) measurements (Rigaku RINT Ultima +2000/PC) were used for qualitative analysis of the crystalline phases present in the samples. The measurements were performed at room temperature, using CuKα radiation, in continuous scan mode in the range from 10° to 80°, with a scanning speed of 1° min^{-1}.

Infrared spectra of the biomasses were obtained using a Varian 640 IR instrument. The frequency was scanned between 4000 and 400 cm^{-1}, with resolution of 4 cm^{-1}, and 32 scans were acquired for each sample.

BET surface area measurements were performed at 77 K with a NOVA 1200 analyzer. All the sorption isotherms were obtained using ultrahigh purity gases (99.999%). Prior to the measurements, the samples were degassed for 2 h at 100 °C.

Photomicrographs were obtained using a scanning electron microscope (TM 3000, Hitachi) operated under low vacuum at an acceleration voltage of 15 kV and filament current of 1850 mA.

2.4. Adsorption assays

The pH dependences of the adsorptive capacities of the hybrid materials were studied using batch adsorption experiments in the pH range from 2.0 to 9.0, which were conducted individually for each metal. Each test was performed using 10 mL of metal solution (at an initial concentration of 100 mg L^{-1}) and 100 mg of the hybrid material, with agitation at 150 rpm, 25 ± 0.2 °C, for 60 min in amber glass flasks. After this period, the adsorbent was separated from the solution using a neodymium magnet and the concentration of lead or cadmium in the solution was quantified by atomic absorption spectrometry. A control solution was prepared without bioadsorbent, and all the experiments were performed in triplicate.

The results were expressed in terms of removal percentages, calculated using the following equation:

$$\% \text{ removal} = [(C_i - C_f) / C_i \times 100] \quad (1)$$

where C_i and C_f are the initial and final concentrations of the metals in solution, respectively.

Kinetic tests were performed as described in the previous section, after adjustment of the pH to the value that provided the highest removal percentage, with aliquots collected at predetermined time intervals (up to 420 min).

The experimental data obtained for adsorption of Cd(II) and Pb(II) were tested using the pseudo-first order, pseudo-second order, and intraparticle diffusion kinetic models, described by Equations (2)–(4), respectively:

$$\log(q_e - q_t) = \log q_e - \frac{k_1}{2.303} t \quad (2)$$

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{t}{q_e} \quad (3)$$

$$K_i = \frac{q}{t^{0.5}} \quad (4)$$

where q_e and q_t are the quantities of Cd(II) and Pb(II) adsorbed at equilibrium and at time t, respectively; t (min) is the time

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