



Batch sorption dynamics and equilibrium for the removal of lead ions from aqueous phase using activated carbon developed from coffee residue activated with zinc chloride

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ABSTRACT

Lignocellulosic materials are good precursors for the production of activated carbon. In this work, coffee residue has been used as raw material in the preparation of powder activated carbon by the method of chemical activation with zinc chloride for the sorption of Pb(II) from dilute aqueous solutions.

The influence of impregnation ratio (ZnCl₂/coffee residue) on the physical and chemical properties of the prepared carbons was studied in order to optimize this parameter. The optimum experimental condition for preparing predominantly microporous activated carbons with high pore surface area (890 m²/g) and micropore volume (0.772 cm³/g) is an impregnation ratio of 100%. The developed activated carbon shows substantial capability to sorb lead(II) ions from aqueous solutions and for relative impregnation ratios of 75 and 100%, the maximum uptake is practically the same. Thus, 75% represents the optimal impregnation ratio.

Batch experiments were conducted to study the effects of the main parameters such as contact time, initial concentration of Pb(II), solution pH, ionic strength and temperature. The maximum uptake of lead(II) at 25 °C was about 63 mg/g of adsorbent at pH 5.8, initial Pb(II) concentration of 10 mg/L, agitation speed of 200 rpm and ionic strength of 0.005 M. The kinetic data were fitted to the models of pseudo-first order and pseudo-second order, and follow closely the pseudo-second order model. Equilibrium sorption isotherms of Pb(II) were analyzed by the Langmuir, Freundlich and Temkin isotherm models. The Freundlich model gives a better fit than the others.

Results from this study suggest that activated carbon produced from coffee residue is an effective adsorbent for the removal of lead from aqueous solutions and that ZnCl₂ is a suitable activating agent for the preparation of high-porosity carbons.

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1. Introduction

Water pollution due to toxic heavy metals has been a major concern for chemists and environmental engineers. Most metals are rather insoluble in water with a neutral or basic pH; and rather than dissolving, they are rapidly adsorbed onto particulate matter or assimilated by living organisms. To prevent deteriorating surface water quality, legislation governing the levels of heavy metals such as copper, zinc, lead and cadmium, discharged from industries is becoming progressively stricter (Cochrane et al., 2006). The treatment of heavy metals bearing effluents has been studied by several

methods such as reduction precipitation, ion exchange, electrochemical reduction, evaporation, reverse osmosis, adsorption and direct precipitation (Xu et al., 2008; Kobyta et al., 2005). Most of these methods require high capital cost and recurring expenses such as chemicals, which are not suitable for small-scale industries. The process of adsorption is by far the most versatile and widely used technique for metal ions removal (Sen Gupta et al., 2009; Panuccio et al., 2009; Volzone and Beatriz Garrido, 2008; Salim et al., 2008; Sawalha et al., 2009). Activated carbon has been the water industry's standard adsorbent for the reclamation of municipal and industrial wastewater for potable use for almost three decades. Despite its prolific use in water and waste industries, activated carbon remains an expensive material. In recent years, research interest in the production of low-cost alternatives to activated carbons has grown.

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Activated carbons (ACs) exhibit a great adsorption capacity, owing to their highly developed pore structures characterized by large surface areas. However, the chemical nature of their surface often plays a dominating role in their performance as adsorbents, catalysts supports, or catalysts (Bansal et al., 1988). The chemistry of the surface depends on the presence of heteroatoms, mainly oxygen, which form organic functional groups such as carboxylic acids, lactones, phenols, carbonyls, quinones, aldehydes, ethers, anhydrides and even others (Boehm, 1994). Growing international demand for these adsorbents, mainly because of their use in applications related to their environmental mitigation, has led to search for new and available low-cost feedstocks of renewable character.

In practice, coal and agricultural by-products of lignocellulosic materials are two main sources for commercial activated carbons production. Agricultural wastes have emerged as a better choice. There is a quite large number of studies regarding the preparation of activated carbons from agricultural wastes which include nutshells, fruit stones, bagasse, coir pith, oil palm waste and agricultural residues from sugarcane, rice and peanut shell, sawdust and canes from some easy-growing wood species (Rao et al., 2009; Ahmadpour and Do, 1997; Lussier et al., 1994; Mohan and Singh, 2000; Kadriavelu et al., 2001; Aydın et al., 2008; Blanco Castro et al., 2000; Srinivasan et al., 1998; El-Shafey, 2007; Marquez-Montesinos et al., 2001; Basso et al., 2002).

Basically, there are two different processes for the preparation of activated carbon: physical and chemical activations. In comparison to physical activation, there are two important advantages of chemical activation. The first one is the lower temperature at which the process is accomplished and the second one is that the global yield of the chemical activation tends to be greater since burn-off char is not required. Among the numerous dehydrating agents, zinc chloride in particular is the widely used chemical agent in the preparation of activated carbon. Knowledge of different variables during the activation process is very important in developing the porosity of carbon sought for a given application. Chemical activation by $ZnCl_2$ improves the pore development in the carbon structure, and because of the chemical effects, the yields of carbon are usually high (Ahmadpour and Do, 1997).

In this work the results obtained on the preparation of activated carbons from coffee residue with zinc chloride activation and their ability to remove lead ions from dilute aqueous solutions are reported. The effect of impregnation ratio on the characteristics of activated products is studied to find the optimum conditions to prepare activated carbons with well-developed porosity. The influence of several operating parameters, such as pH, ionic strength, contact time, and temperature on the sorption capacity is investigated. Kinetic models are used to identify the possible mechanisms of such sorption process. The Langmuir, Freundlich and Temkin models are used to analyse the sorption equilibrium.

2. Experimental methods

2.1. Preparation of activated carbon

The coffee residue collected from coffees houses was first washed with distilled water to remove water-soluble impurities, surface adhered particles and then dried at 60 °C. Afterwards, the granular particles of the coffee residue are impregnated with $ZnCl_2$ solution. 200 g of dried precursor material are thoroughly mixed with $ZnCl_2$ at $ZnCl_2$ /coffee residue ratios of 1:4, 1:2, 3:4 and 1:1 in separate jars. Impregnation was carried out at 85 °C for 7 h to ensure the access of $ZnCl_2$ to the interior of the precursor and then

the temperature of the mixture was increased to the boiling point until complete dryness for 24 h.

The samples are carbonized separately for 1 h at 600 °C in a furnace heated from room temperature to 600 °C at 10 °C/min under a nitrogen flow of 150 mL/min. The chars obtained are cooled under nitrogen (N_2) flow to room temperature and the weight losses due to carbonization are determined. The materials were thoroughly washed with deionized water and soaked in 3 mol/L hydrochloric acid solution for 2 h in order to eliminate the residual zinc from the pores of the carbons. The materials were washed again with hot deionized water and finally cold water until the washed solution was free of zinc ions. Then they were dried in hot air oven at 110 °C, crushed and sieved to a particles size diameters less than 63 μm . Weight loss of the carbon samples is calculated on a chemical free basis and chemical recovery (CR) is estimated according to:

$$CR = \frac{(W_{pi} - W_{pf})}{W_c} 100 \quad (1)$$

where: W_{pi} and W_{pf} are the weights of products before and after washing, and W_c is the used weight $ZnCl_2$. Chemical recovery and weight loss are regarded as indicators of the process efficiency in the chemical activation process. The results are listed in Table 1.

2.2. Physical characterization

Specific surface area and pore volume of the samples are determined by nitrogen adsorption-desorption isotherms measured by Gas Sorption Analyzer (Quantachrome NOVA WIN2-Version 9.0). Prior to each measurement, carbon samples are out-gassed at 200 °C under nitrogen flow for 3 h. The nitrogen adsorption-desorption data are recorded at liquid nitrogen temperature (77 K). The specific surface areas are calculated using BET equation (Slasli, 2002). The total pore volume (V_{tot}) was estimated to be the liquid volume of N_2 at a relative pressure (p/p_0) of 0.95. The Dubinin-Radushkevich (D-R) equation represented below:

$$\log V = \log V_{mic} - D \left(\log \left(\frac{p}{p_0} \right) \right)^2 \quad (2)$$

was applied to calculate the micropore volume (V_{mic}). The mesopore volume (V_{mes}) was obtained by deducting the micropore volume from the total pore volume. The average pore diameters were estimated from the BET surface area and total pore volume ($D_p = 4V_{tot}/S_{BET}$) assuming an open-ended cylindrical pore model without pore networks (Gomez et al., 2006).

The apparent density is calculated by filling a calibrated cylinder with a given activated carbon weight and tapping the cylinder until a minimum volume is recorded. This density is referred to as

Table 1
Physical characteristics of carbon samples.

Parameter	Impregnation ratio $ZnCl_2$ /coffee (w/w)			
	25%	50%	75%	100%
Apparent density (g/cm^3)	0.48	0.51	0.51	0.58
Real density (g/cm^3)	1.01	1.34	1.34	1.22
Conductivity ($\mu S/m$)	9	13	11	15
Moisture (%)	Nil	Nil	Nil	Nil
Particle size (μm)	≤ 63	≤ 63	≤ 63	≤ 63
Total pore volume V_{tot} (cm^3/g)	0.353	0.413	0.552	0.772
Average pore diameter (nm)	2.26	2.44	2.58	3.47

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