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Conversion of biomass from an invasive species into activated carbons for removal of nitrate from wastewater

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ABSTRACT

Development of activated carbons targeted at nitrate ion removal is investigated by chemical activation of sawdust from the invasive species *Parkinsonia aculeata* wood. Phosphoric acid and potassium hydroxide are comparatively used as activating agent. Its effect on main physico-chemical properties and on the effectiveness of the activated carbons in nitrate removal is compared with those determined for a commercial sample. Activated carbons developed with the base show the best effectiveness in nitrate removal, as determined from application of the Langmuir model to the experimental isotherms. The behavior is consistent with the development of surface basic groups and the more basic character of these carbons. Noticeable improvement in nitrate uptake for all the carbons is attained after treatment with a saturated urea solution, with removal levels of ~80–90%, despite reductions in porous structures. Nitrate adsorption onto the urea-treated carbons is in direct correlation with their enhanced contents of elemental nitrogen.

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1. Introduction

Nitrate contamination in surface and ground water has become a growing problem worldwide. Discharge of nitrogen containing compounds into the environment can cause severe damages. They include eutrophication of rivers and deterioration of water bodies, where pollution is caused by heavy algal growth, as well as hazards to human health. Concentrations of nitrate in drinking water exceeding the tolerance limits lead to the formation of nitrosoamine. This compound is related to cancer and increases the risk of diseases, such as methemoglobinemia, also known as cyanosis or blue baby syndrome, in new born

infants [1–4]. Accordingly, removal of nitrate from water is of utmost importance from health and environmental viewpoints.

High levels of nitrate in water may result from human and animal wastes, and from the excessive use of chemical fertilizers. Nitrates are extremely soluble in water and can move easily through soil into drinking water supplies [4–6]. Different physico-chemical and biological methods have been investigated for the removal of nitrate in water and wastewaters, including adsorption, biological denitrification, enhanced ultrafiltration, and electro dialysis. Among them, adsorption is very feasible because it allows simple and economical operation [7], although it requires highly efficient adsorbents. Due to

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their versatility and spread range of applications, activated carbons (ACs) in powder or granular forms are widely used in the removal of several different organic and inorganic pollutants from water. However, effectiveness of ACs in nitrate uptake is quite poor and has been scarcely investigated, mostly using commercial samples [1,2,8–12].

On the other hand, it is still necessary to search for easily available, renewable bioresources as inexpensive precursors for sustainable manufacture of ACs [13]. In particular, use of *Parinsonia aculeata* wood, a major invasive species in several regions worldwide, could represent an interesting option for ACs' production, simultaneously contributing to control the spread of this species through its conversion into a valuable product intended for wastewater treatment. *P. aculeata* forms dense thickets, preventing access for humans, native animals and livestock to waterways. It grows easily on most types of soils, withstands a great deal of heat, and survives in dry areas for prolonged spells. Moreover, seeds remain viable for many years and the seedpods float. The plant spreads by dropping pods into water or pods are washed downstream by seasonal flooding [14].

Within this context, the present study examines the development of ACs from *P. aculeata* wood sawdust, as unexplored precursor, for specific use in nitrate ion removal. The chemical activation process is applied. The effect of the chemical reagent used in the impregnation stage on main physico-chemical characteristics of *P. aculeata*-derived ACs and on their effectiveness in nitrate adsorption is investigated, comparatively using solutions of phosphoric acid and potassium hydroxide. Furthermore, modification of the ACs via post-treatment with saturated urea solutions is also explored in order to examine whether it leads to improve nitrate uptake. It should be mentioned that most works in the literature have been devoted to modify ACs by acid treatments in order to enhance the effectiveness of ACs in the removal of cationic species [8,15].

2. Experimental

2.1. Materials

P. aculeata wood was obtained from trees growing in a rural area placed in Buenos Aires province, Argentina. It is located at km 30.5 of route 215 (35° 4' 24" S, 58° 7' 41" W, 22.33 m elevation). Trunk's pieces of approximately 0.3 m length and 0.15 m cross-section diameter from a vertical distance of 1 m above ground were cut by our team with the help of some farmworkers using chainsaws. The pieces were *in situ* debarked and transported to our lab by road. Then, they were sun dried until constant weight was attained. Afterward, they were crushed, milled, and screen-sieved. Fractions of average particle diameter of 750 μm and 1500 μm were selected for preparation of the ACs with H_3PO_4 acid and KOH solutions, respectively. The samples were then washed and dried at 333 K up to constant weight.

2.2. Preparation of the activated carbons

2.2.1. Activation with H_3PO_4 acid

For phosphoric acid activation, sawdust fractions were impregnated with a 50% H_3PO_4 acid solution, using an acid/

precursor weight ratio of 2. The impregnated samples were first dried in an oven at 383 K for 2 h. Afterward, they were placed in a horizontal, fixed-bed reactor externally heated by an electric furnace, and thermally treated at a heating rate of 3 K min^{-1} up to 723 K under a self-generated atmosphere. Temperature selection was based on previous findings which demonstrate that maximum porosity development for ACs obtained by phosphoric acid activation from different lignocellulosic precursors is attained at 673–773 K [16–18]. Once the selected temperature was reached, it was held for 0.5 h. Then, the samples were allowed to cool down to reach ambient temperature. Further details of the equipment and procedure used have been reported earlier for other precursors [19]. To remove the excess of acid, the ACs were extensively rinsed with distilled hot water until neutral pH in the wash water was attained. Afterward, they were dried in an oven to constant weight.

2.2.2. Activation with KOH

The process involving carbonization of the precursor followed by subsequently KOH activation was applied since it reportedly leads to higher yields of ACs with higher surface areas and moderate ash content than those obtained by the one-step activation process [20]. A similar procedure to the one reported by Moreno Castilla et al. [21] was followed for ACs preparation.

Before proceeding to activation, carbonization of the precursor was carried out. For this purpose, 60 g of the precursor were heated under air flow (150 $\text{cm}^3 \text{min}^{-1}$) at a rate of 10 K min^{-1} up to 623 K. Once this temperature was attained, it was held for 1 h. Then, the carbonized precursor was mixed with a 13.4 mol dm^{-3} KOH solution using an impregnation weight ratio of 1. The latter is given by the ratio between the weight of KOH in solution and the weight of the carbonized precursor. The slurry was dried in an oven at 353 K up to constant weight. Afterward, it was placed in the same reactor used for acid activation, and heated under N_2 flow (150 $\text{cm}^3 \text{min}^{-1}$) at a rate of 10 K min^{-1} up to 573 K for 2 h. Then, the temperature was raised at the same heating rate to 1073 K, and it was held for 2 h, keeping the same N_2 flow rate. Finally, the sample was cooled down to ambient temperature maintaining the N_2 flow, and subsequently washed with 0.1 mol dm^{-3} HCl acid solution and with distilled water until absence of chloride ions in the washing water. The washed samples were then dried at 383 K to constant weight.

Yields in both cases were evaluated from weight differences. The ACs developed through chemical activation by using H_3PO_4 acid and KOH, are designated as ACA and ACB, respectively. For the sake of comparison, a commercial activated carbon (ACC) obtained by phosphoric acid activation of a woody precursor was also used and characterized.

2.3. Urea post-treatment of the activated carbons

The ACs obtained with both chemical reagents and the commercial sample were impregnated for 24 h at 383 K with a saturated urea solution, following a procedure similar to that reported previously [22]. An urea/ACs weight ratio of 2 was used since preliminary experiments showed that it led to enhance elemental N content of the derived ACs

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