



# Adsorption behavior of Ni (II) on lotus stalks derived active carbon by phosphoric acid activation

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## ABSTRACT

The adsorption behavior of Ni (II) on activated carbon prepared from lotus stalks by  $H_3PO_4$  activation has been investigated. Scanning electron microscopy and BET surface area were characterized to provide insight into the properties of lotus stalks activated carbon (LSAC). The adsorbent prepared was found to have a porous structure with surface area of 1220  $m^2/g$ . Adsorption results revealed that the removal speed of Ni (II) by lotus stalks derived active carbon was rapid. The pseudo-second-order kinetic model fitted well with kinetic data, showing high determination coefficients ( $R^2$ ) of over 0.999. The adsorption isotherms of Ni (II) on this adsorbent were well described by Langmuir and the maximum adsorption capacity calculated from Langmuir isotherm model was up to 31.45  $mg/g$ . The negative  $\Delta G$  and the positive  $\Delta H$  suggested the spontaneous and endothermic nature of the process. The effect of pH and ionic strength and desorption studies could help explain the electrostatic attraction and ion-exchange adsorption mechanism. Desorption studies also represented that LSAC had a good regeneration capacity. The results showed that LSAC could be used to effectively adsorb Ni (II) from aqueous solutions.

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## 1. Introduction

The elimination of toxic heavy metals from aqueous environment has received considerable attention in recent years due to their toxicity and carcinogenicity. Ni (II), one of the common toxic heavy metals, is used in a number of industries including electroplating, batteries manufacturing, forging, metal finishing and mining, posing serious threat on human health. Current treatment processes for Ni (II) removal from wastewater include precipitation, coagulation, ion exchange, membrane separation, lime softening and adsorption. Compared with other methods, adsorption technology has received much more attention because it is convenient and economical for reducing trace quantities of heavy metals [1]. Especially, active carbon which has high surface area, porous structure and functional group has been the most popular and widely used adsorbent in water treatment technology all over the world [2]. Many investigators indicate that active carbon is very effective for the removal of Ni (II) [3]. However, the widespread use of active carbon is restricted due to its high costs. To decrease treatment costs, many attempts have been made to find inexpensive alternative activated carbon precursors. At present, more attention has been focused on the utilization of agro-residues and hydrophytes as the precursors. The well developed porous caudex systems of these materials could offer a good precondition for the production of effective

activated carbon [4]. Materials such as sugarcane bagasse [5], peanut hull [6], apple pomace [7], sawdust [8], *Phragmites australis* [9], and *Polygonum orientale* Linn [4] have been utilized for this purpose.

Lotus, one kind of hydrophytes, grows in wetlands and lakes and is widely distributed in Africa and Asia. It can provide people with lotus nuts and lotus roots, leaving lotus stalks as solid waste. To our knowledge, no investigations have used lotus stalks as precursor to produce active carbon.

In this study, lotus stalks were carried out to produce active carbon. The characteristics of this active carbon were measured and described in order to compare with other active carbons. The adsorption behavior of Ni (II) by active carbon from lotus stalks was explored in aspects of kinetics and thermodynamics to describe some important thermodynamic parameters. In addition, the effects of pH and ionic strength were also considered. Desorption experiments were also conducted to examine the recycling use capacity.

## 2. Materials and methods

### 2.1. Preparation of lotus stalks derived activated carbon

Lotus stalks, obtained from Ji Nan, Shandong, China, were washed with water, dried at 80 °C for 12 h and crushed to the desired size (2–3 mm) using a grinder crusher. The dried mass was afterward saturated in  $H_3PO_4$  thoroughly (40 wt.%) at a ratio of 1/2 (g LS/g  $H_3PO_4$ ) for 12 h, heated in a muffle furnace with a heating rate of 15 °C/min to reach the desired temperature of 450 °C and maintained for 1 h.

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After heated, samples were naturally cooled to room temperature (20 °C), and washed with deionized water repeatedly until pH of the filtrate became steady. The samples were filtrated and dried at 80 °C for 5 h and crushed. The dry material particle size of 104–150 μm was stored in a desiccator for further experimental use.

## 2.2. Analytical methods

The surface of adsorbent was characterized by scanning electron microscopy (SEM, HITACHI S-520). The BET surface area and porous properties of LSAC were obtained from N<sub>2</sub> adsorption at 77 K using a surface area analyzer (Quantachrome Corporation, USA). The specific surface area ( $S_{BET}$ ), the total pore volume ( $V_{tot}$ ) and the pore width ( $D_w$ ) were provided by the manufacturer's software. The t-plot method was used to calculate the micropore surface area ( $S_{mic}$ ) and the micropore volume ( $V_{mic}$ ). The external volume ( $V_{ext}$ ) and the external area ( $S_{ext}$ ) were the deduction of  $V_{mic}$  from  $V_{tot}$  and the deduction of  $S_{mic}$  from  $S_{BET}$ , respectively. The acidic surface functional groups on LSAC were determined using Boehm's titration method [10] and [11]. The number of functional groups was based on the following assumptions: Carboxyl was neutralized by NaHCO<sub>3</sub>. Carboxyl and lactone were neutralized by Na<sub>2</sub>CO<sub>3</sub>. Carboxyl, lactone and phenolic were neutralized by NaOH. The Photometric Determination of Nickel with Dimethylglyoxime Using Potassium Sodium Tartrate and EDTA as Masking Agent was used to analyze the concentrations of Ni (II) in the liquor before and after adsorption using a UV–vis spectrophotometer (UV-754, Shanghai) at an absorbance wavelength of 530 nm.

### 2.2.1. The preparation of standard curve

The Ni (II) stock solutions were prepared with deionized water using nickel single element dissolving in HNO<sub>3</sub> solution. Ni (II) working solutions were prepared by diluting Ni (II) stock solutions with deionized water.

### 2.3. Ni (II) adsorption and desorption experiments

A stock solution of 100 mg/L of standardized Ni (II) was prepared from NiCl<sub>2</sub>•6H<sub>2</sub>O. Experimental solutions of the desired concentration were obtained by further dilution. Adsorption kinetic experiments were performed in a 2000 mL beaker. Purified LSAC of 2 g was added to 2000 mL Ni (II) solution with different initial concentrations (20 mg/L, 30 mg/L and 40 mg/L) with 0.01 mol/L NaCl as background electrolyte. Then, the breakers were placed on magnetic stirrers at a speed of 125 rpm with a temperature control of 298 K. The samples were taken at determined time to filter using 0.45 μm millipore membrane filters for analysis.

The effects of contact time, temperature, solution pH, and ionic strength were investigated in batch adsorption experiments. A mass of adsorbent (0.1 g) was weighed into 250 mL glass bottles and contacted with 100 mL of Ni (II) solutions of different initial concentration with 0.01 mol/L NaCl as background electrolyte. The bottles were sealed and placed in temperature-controlled water bath oscillator with the agitation speed of 125 rpm (SHA-B, Shanghai, China) until equilibrium was reached. Finally, the supernatant liquids were filtered, and the metal concentration in each flask was determined using the method described above. All pH measurements were carried out using a pH meter (Model pHs-3C, Shanghai, China). The initial pH levels of the experimental solutions were adjusted to constant values by adding HCl or NaOH solutions. KCl, NaCl, NaNO<sub>3</sub>, CaCl<sub>2</sub> and NaH<sub>2</sub>PO<sub>4</sub> were chosen to investigate separately the effect on Ni (II) adsorption by LSAC.

#### 2.3.1. Desorption experiments

After the kinetics study, the spent LSAC (initial concentration was 20 mg/L) was collected and dried in a vacuum oven at 80 °C.

Ammonium citrate, HCl and water were used as desorption agent. The percentage of desorption was calculated as follows:

$$\% \text{Desorption} = \frac{m_d}{m_a} \quad (1)$$

where  $m_d$  is the amount of Ni (II) desorbed (mg/L), and  $m_a$  is the amount of Ni (II) adsorbed (mg/L).

## 3. Results and discussion

### 3.1. Characterization of lotus stalks derived active carbon

The scanning electron microscope (SEM) is widely used to study the morphological features and surface characteristics of the adsorbent materials. SEM micrograph (1000× magnification) of LSAC is shown in Fig. 1. The surface structure of LSAC shows a distinct regular tubular porosity, which is different from carbon derived from other materials. LSAC also has cavernous pores of about 60 μm diameter. This may be the original configuration of LS, which may favor the diffusion of Ni (II) into LSAC.

As Fig. 1 illustrates, the adsorption and desorption isotherms of N<sub>2</sub> at 77 K for the sample are clearly a mixture of types I and IV. The initial part of the isotherms follows the same path as the corresponding type I isotherms, which are given by microporous solids having relatively small external surface. Characteristic features of the Type IV isotherm are its hysteresis loop, which is associated with capillary condensation taking place in mesopores. The adsorption and desorption isotherms revealed the existence of microporous and mesopores simultaneously. According to the International Union of Pure and Applied Chemistry (IUPAC) classification of adsorption isotherms [12], the adsorbent pores are defined as three groups: macropore width (>50 nm), mesopore width (2–50 nm) and micropore width (<2 nm). The pore-size distribution (Fig. 2) of PLAC shows that majority of the pores fall into the range of mic-mesopores (<10 nm). Porous structure parameters for the sample are listed in Table 1. As Table 1 indicates, the derived activated carbon is a high quality adsorbent with surface area of 1220 m<sup>2</sup>/g. The pore volume is 1.191 cm<sup>3</sup>/g and the pore width (mode) is 1.410 nm. The sample exhibits various distributions in both the micropore (41.2%) and mesopore (58.8%). It is very suitable for adsorption of small molecules.

The adsorption properties of activated carbon depend not only on its pore structure, but also on its surface chemical properties. Activated carbon surface functional groups of the type and quantity can significantly affect the behavior of the surface of activated carbon. The functional groups of LSAC (Table 2) very usually fulfill the adsorption progress. H<sup>+</sup> provided by carboxyls and phenols can exchange with

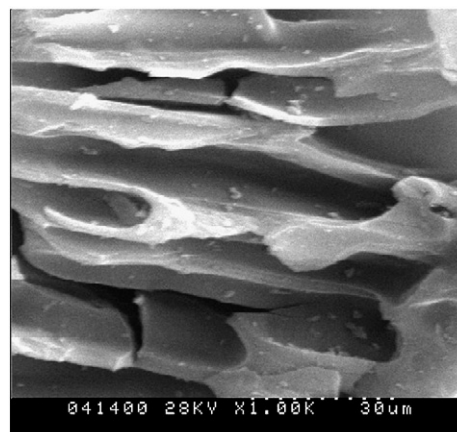


Fig. 1. SEM micrograph of LSAC (1000×).

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