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Preparation and characterization of the hydrogen storage activated carbon from coffee shell by microwave irradiation and KOH activation

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ABSTRACT

Coffee shell is an environmental concern to china along with steady growth of coffee production. This study attempt to characterize high specific surface area activated carbon (HSSA-AC). HSSA-AC was prepared from carbonized material which obtained from coffee shell by microwave irradiation. Textural properties and surface chemistry of HSSA-AC were found to be strongly depending on the activation time, KOH/C ratio and particle size. The textural properties of the samples were investigated by means of scanning electron microscope analyzer (SEM), cryogenic N_2 adsorption, whereas, surface chemistry was probed through Fourier Transform Infrared (FTIR) spectrometer (Maldhure and Ekhe, 2011) and Hydrogen storage performance was tested by H_2 adsorption. Maximum surface area of 3149 m^2 g^{-1}, Iodine adsorption value 2566 mg/g, Methylene Blue adsorption value 47.5 mL 0.1 g^{-1}, the hydrogen adsorption value 0.91 wt% at 14 MPa and yield 39% was observed in case of microwave treated sample at activation time 9 min, KOH/C ratio 5 and particle size 0.25–0.71 mm. Results revealed usefulness of microwave treatment in influencing surface area of HSSA-AC which could be used in a hydrogen storage material research application.

1. Introduction

High specific surface area activated carbon (HSSA-AC) is charcoal that has been treated to open up millions of tiny pores between the carbon atoms. HSSA-AC with high micropore volume and large specific surface area has a good adsorption performance (Wang et al., 2005), which is used as a potential adsorbent in processes such as the purification of industrial effluents(Pintar A, 2003), groundwater treatment (El-Sheikh et al., 2004)and the removal of volatile organic compounds from air and mercury vapors from a gas mixture (Vitolo and Seggiani, 2002). HSSA-AC has become the national economy, national defense construction and People's Daily life indispensable products. HSSA-AC demonstrated significant adsorption in gas and liquid phases due to its high micropore volume (Vmic), large specific surface area (SBET), favorable pore size distribution, thermal stability, capability for rapid adsorption and low acid/base reactivity (Li et al., 2009).

The chemical, physical and composite activation method has been used for HSSA-AC obtained (Wang and Li, 2002). Although the chemical method is one of the most usual and applicable methods for the production of HSSA-AC, this method has some disadvantages such as the equipment corrosion, wastewater pollution and hard recovery of activator (Hesas et al., 2013). The physical method generate less pollution, but AC obtained by this method has less surface area (Zhang et al., 2014).

The traditional thermal heating method is a thermal gradient from the surface to the interior of a particle, non-uniform temperature in material, high cost of heating, long preparation time. A microwave radiation method has been used recently by many

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Table 1
Analysis of the char.

The char	Ash content (%)	Moisture content (%)	pH	Iodine absorbed C_0 (mg g^{-1})
Coffee shell	5.22	8.86	10.30	68

researchers as an alternative method for heating. Compared with the traditional thermal heating method (Hesas et al., 2013). The microwave radiation method has some advantages such as uniform and fast heating and short activation time.

2. Methods

2.1. Adsorbate

The Iodine and Methylene Blue solution were chosen as the adsorbates testing HSSA_AC adsorption capacity. The Activation time, the KOH/C ratio and the particle size were investigated by determining the amount of Iodine and Methylene Blue adsorbed by HSSA-AC.

2.2. The preparation of HSSA-AC

Coffee shells used as raw material in this study were collected from the Puer, Yunnan, China. The material was manually chosen, air-dried, crushed and sieved to obtain a geometrical mean size ranging from 1.0 to 3.0 mm. The carbonization process was performed by loading 500 g of dried precursor into a muffle furnace, and heated up to a carbonization temperature of 500 °C.The char produced was mixed in potassium hydroxide solution with KOH/C ratio of 1:1,2:1,3:1,4:1,5:1 and 6:1.

A modified microwave oven with a microwave input power of 800 W was applied for the activation steps. The activation time of 5–25 min was selected as the heating period. The resultant HSSA_AC was washed with 0.1 M hydrochloric acid, and then rinsed repeatedly with hot and cold distilled water until the pH of the washing solution reached 6–7 (Foo and Hameed, 2012).

The main parameters in the process of activation were the microwave radiation time, the microwave power level, the impregnation ratio and the diameter of particles. The effects of these parameters on the physical and chemical properties of HSSA-AC, such as the pore structure, the adsorption capacity, the carbon yield and the surface functional groups were studied.

2.3. Characterization of HSSA-AC

The surface functional groups of HSSA-AC were determined by Fourier Transform Infrared spectrometer (Nicolet, iS10,USA). The pore structure characteristics of HSSA-AC were determined by nitrogen adsorption–desorption isotherm at 77 K using an automatic Micromeritics Autosorb iQ Station 1 volumetric adsorption analyzer. The specific surface area (SBET) was calculated by the Brunauer–Emmett–Teller (BET) equation; the total pore volume (VT) was evaluated by converting the adsorption volume of nitrogen at relative pressure of 0.98 to equivalent liquid volume of the adsorbate. The micropore volume, micropore surface area and external surface area were deduced using the t-plot method. The surface morphology was examined using the scanning electron microscope (Foo and Hameed, 2012). Hydrogen adsorption-desorption properties of HSSA_AC were determined by hydrogen adsorption–desorption isotherm at 298 K using an automatic Micromeritics 3H-2000PH1 volumetric adsorption analyzer (see Table 1).

3. Results and discussion

3.1. Optimization of preparation conditions

3.1.1. Optimization of the activation time

The optimization process was performed by taking respectively seven samples of Coffee Shell derived char weighing 10 g into microwave oven with power of 800 W and KOH/C ratio of 1:1. The heating times for seven samples were 3 min, 5 min, 7 min, 9 min, 11 min, 13 min, 15 min, 17 min, 20 min and 25 min Table 2 show the results of yield, Iodine and Methylene Blue adsorption value.

Table 2 presents the variation of Iodine adsorption values with respect to time at the concentrations 1013–1305 mg g^{-1}. It is clear that the adsorption process for HSSA-AC increased sharply at the initial stage, indicating of the availability of HSSA-AC. The process is gradually slower when the activation time over 9 min.

From the table we can see that the Methylene Blue adsorption values of HSSA-AC had a tendency to increase slowly.

The variation of HSSA-AC yield is summarized in the table. The yield shows a trend of decrease when time increased.

With the increase of the reaction time, more carbons in samples were consumed and more micropores, mesopores and macropores were formed, so the Iodine and Methylene Blue adsorption value increased when the yield decreased.

Despite the adsorption capacities of HSSA-AC was increasing with time growing, adsorption became slower over 9 min. The yield of HSSA-AC was lower and microwave energy consumption was more and more big. Thus, the optimum activation time was confirmed as 9 min.

3.1.2. Optimization of KOH/C ratio

Seven 10 g of Coffee Shell derived char were weight respectively which mixed with KOH into microwave oven at 800 W and then held 9 min. The KOH/C ratio for seven samples were as follows: 1:1,2:1,3:1,4:1,5:1 and 6:1. Table 3 show the yield, Iodine and Methylene Blue adsorbed of resultant HSSA-AC.

With the increase of the KOH/C ratio, the Iodine adsorption value was from 1153 mg g^{-1} to 2156 mg g^{-1}. However, As shown in Table 3, KOH/C ratio ranging from 5:1 to 6:1, the Iodine adsorption value reduced to 1685 mg g^{-1} That is because the Iodine adsorption represents the amount of micropores and overdose of KOH lead to overactive and changed pore structures. The micropores of activated carbons were destroyed and formed more mesopores and macropores.

Table 2
Optimization of the activation time.

Time (min)	5	7	9	11	13	15	17	20	25
Iodine adsorbed (mg g^{-1})	1013	1093	1153	1102	1124	1111	1150	1285	1305
Methylene blue adsorbed (mg 0.1 g^{-1})	13.8	15.6	16	15.1	15.5	15	15.4	17.5	19.5
Yield (%)	64.5	61.4	60	59.5	57.2	57	55	46	43