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## Comparative study on microstructure and surface properties of keratinand lignocellulosic-based activated carbons



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#### ABSTRACT

The paper probed the preparation of activated carbon by potassium silicate ( $K_2SiO_3$ ) activation from keratin waste (cowhair waste, CW) and lignocellulosic materials (*Cyperus alternifolius*, CA) and the comparisons of physicochemical properties of the resulting carbons. These impregnation conditions were as follows: one impregnated at room temperature for 12 h then dipped at high temperature for 30 min; the other was only impregnated at room temperature for 12 h, producing four activated carbons CWAC-1, CWAC-2, CAAC-1, and CAAC-2. The influence of activation time,  $K_2SiO_3$ /precursor weight ratio, and the pre-process on properties of activated carbons was discussed. The CWAC-1 produced at 700 °C with the  $K_2SiO_3$ /precursor weight ratio of 2:1 possessed the Brunauer–Emmet–Teller (BET) surface area of 1965  $m^2$ /g and total pore volume of 1.345 cm<sup>3</sup>/g, while CAAC-1 prepared at the same conditions attained the BET surface area of 1710  $m^2$ /g and total pore volume of 0.949 cm<sup>3</sup>/g. The surface area and total pore volume of CAAC increased with the impregnation ratio. Moreover, CWAC-1, CWAC-2, CAAC-1, and CAAC-2 exhibited high portion of micropores, illustrating the role of  $K_2SiO_3$ . The analysis with a Fourier transform infrared spectrometer indicates that CWAC has more functional groups than CAAC, as well as CWAC-1 and CWAC-2 which possess similar functional groups. © 2015 Elsevier B.V. All rights reserved.

#### 1. Introduction

Activated carbon (AC) is well known as an effective adsorbent which is widely applied in the separation and purity process for wastewater and exhaust gas operation [1,2]. It is also used as a catalyst carrier, raw material of capacitor and solid-phase extractors from aqueous solutions [3]. ACs have many advantages, including large surface area, various oxygen-containing functional groups and stable chemical properties. It can be prepared by any carbon-rich substances, and its properties depend on the raw materials and methods of preparation.

Generally, in the process of AC preparation, the activation methods contain chemical, physical and the combination of both of them. For chemical method, the precursor is mixed with compounds such as phosphoric acid [4], potassium hydroxide [5], zinc chloride [6] and potassium carbonate [7,8], and then activated at various temperatures. The previous studies [9] have been reported that carbon activated by potassium hydroxide could possess high specific surface area and large number of micropores, but the apparatus would be corroded and the life of which lowers due to the strong basicity. Actually, silicone material is widely employed as a catalyst and fire retardant to prevent the flame of material in origin [10], and potassium silicate ( $K_2SiO_3$ )

has lower alkalescency than potassium hydroxide. In general, carbon prepared by potassium hydroxide consists of the pyrolysis of the precursor material and activation of the char using activating agent. However, ACs activated by  $K_2 \text{SiO}_3$  can be prepared in one step, which is favorable to make the steps simple and reduce energy consumption.

Presently, lignocellulosic materials are composed of sugar, which have been found to be suitable for the preparation of AC due to its low cost and high carbon content, such as Zizania latifolia [11], cattail [12]. lotus stalks [13], cotton stalks [14], and oil palm shell [15]. Cowhair waste (CW), a kind of keratin waste, is abundant in tanneries, slaughterhouses, and textile factories. It has biological structures of unity and abundant keratin which provides nitrogen-containing functional groups. Converting the natural fibers into fine powders can be used into the adsorption in aqueous solution. Chen et al. have reported the characteristics of wool-based carbons activated by H<sub>3</sub>PO<sub>4</sub>, ZnCl<sub>2</sub>, and K<sub>2</sub>CO<sub>3</sub> [16]. However, to our knowledge, there is no study comparing the properties of keratin and lignocellulose-based ACs activated by potassium silicate. In this sense, the main aim of this paper is to study the preparation of keratin AC by K<sub>2</sub>SiO<sub>3</sub> activation with different preparation condition, and the comparison of keratin AC with lignocellulosebased ACs. Regarding the preparation method of AC, we have studied (i) how variables such as activation temperature and impregnation ratio affect the formation and properties and (ii) the effect of K<sub>2</sub>SiO<sub>3</sub> on the formation of pores. Regarding the precursors, cowhair waste

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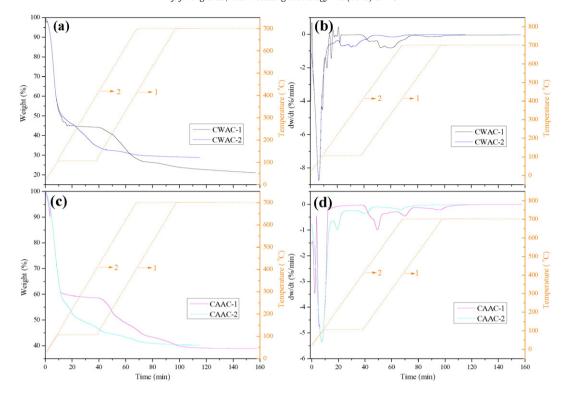


Fig. 1. TGA and DTG diagrams for CW-K<sub>2</sub>SiO<sub>3</sub> and CA-K<sub>2</sub>SiO<sub>3</sub> in N<sub>2</sub> atmosphere.

(CW) and *Cyperus alternifolius* (CA) on developing functional groups for both ACs, textural properties of ACs were analyzed and probed.

#### 2. Materials and methods

#### 2.1. Synthesis of the AC

All the chemicals used in this study were analytical reagents, and deionized water was used as the experimental water. CW used in this study was obtained from a local leather factory, and cut into 5 mm length for further study. CA was acquired from artificial wetlands in Weishan, Shandong Province, China. Before preparing AC, CA was first

washed with distilled water and then grounded into particle size in the range of 0.25–0.425 mm.

Two different kinds of ACs were produced by  $K_2SiO_3$  activation with varying impregnation ratios (IR) of 1:1–3:1 (g  $K_2SiO_3$ :g CW/CA). One sample was impregnated at room temperature for 12 h, then heated from room temperature to 105 °C and maintained for 30 min. The samples were placed into a tube furnace, then heated to the desired temperature (400–800 °C) at a same heating rate (15 °C/min) under  $N_2$  atmosphere (100 mL/min) and kept at that temperature for 1 h. The ACs are referred to as CWAC-1 and CAAC-1, and the best preparation condition was determined by acquiring the properties of ACs. Thus, another mixture of  $K_2SiO_3$  and CW/CA with the best impregnation ratio (2:1) was only dipped for 12 h at room temperature, and then activated

**Table 1**Surface areas and pore volume of ACs produced by CW under different conditions.

AC	Activation temperature (°C)	Impregnation ratio (IR)	$\frac{S_{\rm BET}}{({\rm m}^2/{\rm g})}$	$\frac{S_{\rm mic}}{({\rm m}^2/{\rm g})}$	$\frac{V_{\text{tot}}}{(\text{cm}^3/\text{g})}$	$\frac{V_{\rm mic}}{({\rm cm}^3/{\rm g})}$	$\frac{V_{\rm mic}/V_{\rm tot}}{(\%)}$	$\frac{D_{\rm P}}{({\rm nm})}$
500	2:1	657		0.337	0.2837	84.18	2.046	
			632					
600	2:1	802		0.494	0.315	63.77	2.460	
			707					
700	2:1	1965		1.345	0.7954	59.14	2.646	
			1462					
800	2:1	4		0.039	0.0016	4.10	35.617	
CWAC	700	1:1	33		0.099	0.0109	11.01	10.005
				25				
	700	1.5:1	1566	4.400	0.855	0.6609	77.30	2.145
	700	2.4	1005	1466	4.045	0.7054	50.14	2010
	700	2:1	1965	1.462	1.345	0.7954	59.14	2.646
	700	2.5.1	1020	1462	1 170	0.7462	C2 4F	2.400
	700	2.5:1	1820	1489	1.176	0.7462	63.45	2.488
	700	3:1	339	1489	0.594	0.1373	23.11	6.980
	700	5.1	339	205	0.554	0.1373	23.11	0.560
CWAC-2			419	203	0.394	0.162	41.12	3.649
			713	269	0.534	0.102	71.12	5.045

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