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Microstructure regulation of super activated carbon from biomass source corncob with enhanced hydrogen uptake

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abstract

A series of super activated carbon have been prepared by potassium hydroxide activation of corncob. The as-obtained samples were characterized by SEM, TEM and N_2 -sorption. The results show morphologies and textural of activated carbon are highly depended on the activation temperature, heating rate, whereas the activation time is not a key factor. Morphologies and porous structure of activated carbons can be regulated by adjusting preparation parameters. A super activated carbon with BET surface area of 3530 m^2/g and total pore volume of 1.94 cm^3/g is obtained. However, the other activated carbon with smaller pore size exhibited the highest hydrogen uptake capacities exceeding 2.85 wt% at -196 °C and 1.0 bar, whose BET surface area is only 2988 m²/g. The correlation investigations show the micropore volume between 0.65 nm and 1.5 nm can be more important than BET surface area and total pore volume for hydrogen uptakes at -196 °C. The present results indicate that the corncob-derived activated carbons can be promising materials for hydrogen storage.

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1. Introduction

Hydrogen is considered as an important clean energy carrier, and will play an important role in future transportation energy sector. However, there are several major technical hurdles that must be overcome before a transition to hydrogen based on economy can be envisaged. One of the most difficult technical hurdles is the hydrogen storage. Several storage options are being extensively studied: compression storage at high pressure, cryogenic storage, and different ways of solid

storage: physisorption, chemisorption, or chemical compound formation.

Among the approaches listed above, physisorption has gained much attention due to its good kinetics, reversibility, and relatively high storage capacity. In general, physisorption employs high surface area sorption materials that can effectively adsorb hydrogen, such as activated carbon. Comparing with other novel sorption materials, such as metal organic frameworks (MOF), covalent organic frameworks (COF), and porous aromatic frameworks (PAF), activated carbon shows

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good chemical stability, ready availability, high packing density, and low cost, which has been a good candidate material for hydrogen storage $[1-3]$ $[1-3]$. In the past few years, some activated carbons with high specific surface area and large pore volume have been prepared by chemical activation, especially, preparation of activated carbon with high specific surface area from biomass wastes such as sawdust, bamboo leaves, coconut shell, corncob, hemp, etc., has attracted much attention $[4-7]$ $[4-7]$. Among these carbon sources, corncob is a good bio renewable precursor to prepare activated carbon with high specific surface area. Corn is one of the most widely cultivated crops all over the world. According to International Grains Council (IGC) report, the global corn annual production has been more than 800 million tons from 2010. The corn production is projected to get more than 200 million tons in 2012 in China alone. Corncobs are the byproducts in corn industry, with about 160-180 kg corncobs generated for every 1 ton of corn. So, the annual corncob yield will be enormous. In addition, corncob is easier to collect, transport and storage than other biomass wastes, which is also an important economic factor to consider when it comes to the commercialization of the corncob based carbon making process in the future.

How to efficiently and effectively use these corncobs to produce activated carbon with ultrahigh surface area has been a significant research topic. It not only can make considerable economic benefit, but also can meet the increasing demand of using activated carbon in the application areas such as energy storage, chemistry, environment, and so on $[8-11]$ $[8-11]$ $[8-11]$.

Previous researches on making corncob-derived activated carbon were mainly focused on common activated carbon which is generally used as adsorbents in wastewater treatment to remove organic pollutants [\[12,13\]](#page--1-0). These common activated carbons normally show a surface area of less than 2000 m^2/g and cannot be used for hydrogen storage to achieve the required gravimetric and volumetric energy storage target. Therefore, production of activated carbons with much higher surface area from corncob and investigation of their hydrogen storage performance become an interesting topic. However, up-to-date, only a few papers reported corncob-derived activated carbons with BET specific surface area reaching about 3000 m² g^{-1} [\[7,14\]](#page--1-0) in open literature. There is still a lack of comprehensive understanding about the relationship of process conditions, microstructure of the carbon, and the hydrogen storage performance of these carbons.

In this work, a series of super activated carbon from corncob with different processing conditions were prepared, and detailed microstructure and hydrogen storage performance of these carbon materials were investigated. It was demonstrated that activated carbon with super high surface area with controlled morphologies and textural could be prepared from corncob. The relationship between the porous structure of corncob-derived activated carbon and their enhanced hydrogen storage capacity was also illustrated in this paper.

2. Experimental

2.1. Preparation of activated carbon

Activated carbon samples were prepared by first carbonization followed by activation of the biomass sources corncob. The corncobs used are from Chifeng city of Inner Mongolia province located in northeast of China. The type of corn is No. 9 Jinkun. Detailed procedures are described as follows. First of all, after drying for 12 h at 120 $^{\circ}$ C, corncobs were grounded and sieved into powders with typical size of less than 830 μ m. Secondly, the corncob powders were placed into a furnace and heated to 400 °C with a heating rate of 3 °C/min for 4 h in flow nitrogen with 1.0 L/min. The carbonized powders were then further ground into small particles with size of less than 25 μ m by ball milling. Thirdly, 5.0 g of the carbonized powders were suspended in some KOH saturated solution and magnetically stirred for 2 h at room temperature followed by drying under vacuum at 120 °C overnight to evaporate most of the adsorbed water on the sample. Finally, the impregnated mixture was transferred to a horizontal steel tube furnace and activated at different temperatures (800–900 \degree C), different activation times (1–3 h) and different heating rates (3–10 \degree C/min) under flow nitrogen with 1.0 L/min. The final products were washed with deionized water until the pH value reached about 7.0 and then dried at 110 °C under vacuum. The as-prepared products were hereafter designated as CACx (Corncob Activated Carbons), and x is sample's number (see Table 1).

2.2. Characterization and analysis methods

The textural properties of the samples were studied by N_2 sorption at -196 °C with automatic instrument (ASAP2020, Micromeritics) over a wide relative pressure range from about

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