



Synthesis of activated carbon derived from rice husks for improving hydrogen storage capacity



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ABSTRACT

In this work, we prepared activated carbon derived from rice husks (RHC) using chemical activation with KOH ratio. The results showed that significant increase in specific surface area and optimum pore size for hydrogen storage of RHC by KOH activation. Then, it was interesting note to that the best hydrogen storage capacity of 2.85 wt.% was observed in the RHC1 sample (KOH ratio of 1). The hydrogen storage capacity was strongly influenced by the nanometered size distribution and micropore volume than the specific surface area or total pore volume.

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Introduction

Recently, hydrogen has emerged as one of the most promising alternative energy source to replace fossil hydrocarbon resources, due to its high abundance, high combustion heat, non-pollutant emission while combustion. Many studies have been performed on the effective and safe use for hydrogen energy. Research has been carried out targeting 5.5 wt.% (revised in 2009) of a hydrogen storage system to use hydrogen as transportation energy as suggested by the U.S. Department of Energy, in order to utilize hydrogen storage on certain materials. Many methods for hydrogen storing have been suggested such as liquid, solid-state storage, metal hydride and microporous materials. Among others, microporous materials are attractive hydrogen energy carrier, due to its high specific surface area, owning small pore for storing gas molecule. Recently, researchers have been actively researching materials for hydrogen storage using microporous materials, such as metal–organic frameworks (MOFs), zeolite and carbon materials [1–4].

The hydrogen molecule interacts weakly with the storing materials surface. So, interaction mechanism between hydrogen molecule and storage materials surface highly depends on physisorption mechanism, then chemisorption mechanism. The hydrogen molecule can easily be desorbed with adsorbent, due to its weak van der Waals interactions between hydrogen molecule

and surface of adsorbent materials. Eventually, the physisorption mechanism on porous materials is an attractive method for hydrogen storage, due to its rapid adsorption and desorption speed [2,3].

Researchers are paying attention to the possibility of MOFs, the microporous crystal compounds formed after organic bridging ligand connects metal ions or metal clusters through coordination bond, for heterogeneous catalysts, separation, sensors, electrons and gas storage. Researchers are actively investigating the potential use of MOFs as materials for storing hydrogen. As MOFs shows properties such as framework interpenetration, framework flexibility and chirality. It also have a high specific surface area and are microporous. Further, MOFs have nanoscaled cavities and open channels to small molecules can give access with inherent conditions. However, MOFs are vulnerable to water, go through a complex synthesis process and use an expensive precursor. In addition, it is difficult for them to control the adsorption and desorption speed [1,5,6].

Zeolite is a general term for aluminosilicates with high crystallinity. Zeolite has an ample cage and channel structure, high heat stability and ion exchange capacity, and huge potential as a reservoir of non-polar gas. Gas Reversible closure of zeolite, which is a widely known symptom, is very effective about gas storage [7]. As zeolite is vulnerable to water and hard to reproduce, there has been a great deal of research into ways to complement those aspects [8,9].

There are various carbon materials, and they can be classified based on their synthesis types into carbon black, carbon nanotubes, carbon fibers and activated carbons, and their

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application range is very wide. Carbon materials are chemically very stable and have high thermal conductivity, electro conductivity, high strength and high modulus, so they are good in terms of structural stability as well. As it is easy to activate activated carbons and to treat its surface, in particular, revelation of the specific surface area and porosity is possible, and activated carbons have various structural and surface properties depending on the synthesis methods [10–15]. As a result, activated carbons have been the most actively researched materials for gas adsorption and storage. Researchers can freely choose the materials of activated carbons, and the working processes are simple, so activated carbons are highly practical and have additional physical and chemical properties from chemical treatment. As such, scientists are actively researching the application of activated carbons in the fields of gas storage and harmful gas removal [16–20].

Each year, Korea produces about 700,000 tons of rice husks, as an agricultural by-product. Most of these are used simply as fertilizer, or for warming and water treatment. As amorphous matters containing 30–40% carbons and 20–30% SiO₂, rice husks are composed of silicates and carbons. Rice husks have a complex structure of carbons and silicates after organic substances are removed after carbonization, so they are appropriate materials for activated carbons. KOH, widely known as an activation reagent, reacts to silicon that is an atom of the same group [21], as well as carbons of rice husks, and when it can be activated, higher activation efficiency can be expected from KOH than existing carbon materials [22,23].

In this research, activated carbon materials were synthesized using rice husks and chemically activated by KOH. Chemical activation using KOH was effective in developing optimal micropores for hydrogen storage. The aim of this experiment was to examine the effect of KOH amount and pore size on the hydrogen storage behaviors of the synthesized activated carbon derived from rice husks. The textural properties of activated carbons were correlated with the hydrogen storage to figure out the factor that could enhance the property of rice husks based hydrogen storage materials.

Experimental

Materials and sample preparation

For this research, rice husks grown in Goheung, South Jeolla Province, were used as a carbon precursor. Prior to the experiment, rice husks in natural conditions were washed with distilled water to remove impurities. The moisture was removed from the washed rice husks in an 80 °C oven for 24 h. The rice husks were placed in a solution with a fixed weight ratios of KOH/rice husks = 0.5, 1, 2, and 3, respectively. The mixtures were dried at 80 °C oven for 24 h, and then transferred to an alumina boat in a furnace under an N₂ gas flow of 200 mL min⁻¹. The samples were heated to 900 °C for 1 h, with a heating rate of 2 °C/min. Rice husks were washed with distilled water until their pH reached around 7. The samples were finally dried at 80 °C oven for 24 h. The chemically further activated carbon samples are labeled RHC0.5, RHC1, RHC2 and RHC3 according to their KOH ratios.

Characterization

Thermo gravimetric analyzers (TGA, SDT-Q600, NETSCH) was used to figure out carbonization temperature of rice husks. Chemical properties of the surface of rice husk-based activated carbons was analyzed using X-ray photoelectron spectroscopy (XPS, K-Alpha, Thermo Scientific). AlKa was used as a light source for XPS measurement, and pressure within the chamber was controlled at 10⁻⁸–10⁻⁹ torr. X-ray Diffraction (XRD, D2 PHASER,

Bruker) investigated the transition of the activated carbons derived from rice husks that occurred in the crystalline phase and in their lattice distortions. The surface morphology was observed by Scanning Electron Microscope (SEM, S-4300SE, Hitachi Co.). The textural property was measured using N₂/77 K isothermal adsorption equipment (BELSORP, BEL Co.). The specific surface area was calculated using the BET equation (Brunauer–Emmett–Teller equation). The amounts of N₂ adsorbed at relative pressures ($P/P_0 = 0.99$) were used to investigate the total pore volumes. The mesopore volume was calculated through the BJH equation (Barret–Joyner–Halenda equation) and the micropore volume was calculated after deducting the mesopore volume from the total pore volume.

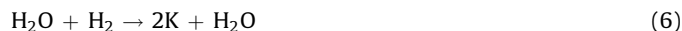
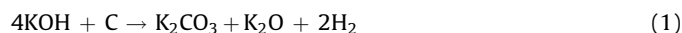
Hydrogen storage capacity

The hydrogen storage capacity experiment was conducted under H₂/77 K at 1 bar. For pretreatment measurement, the system was completely exhausted at 473 K for 6 h. After the system was cooled to room temperature, hydrogen was injected until pressure reached at 1 bar. The influence of moisture or other impurities on measurement was minimized using ultra-high pure hydrogen (99.9999%). Finally, the hydrogen storage capacity was measured using a method of volume measurement method at 77 K/1 bar.

Results and discussion

Activation mechanism and carbonization process

The activation mechanism between KOH and rice husks is as follows [21]. Considering the decomposition of KOH and the removal of carbons and silicon, the following reactions take place at the same time.



It has been reported that KOH activation actively occurs at temperatures of 600 °C or higher in general [3], and it has been confirmed that the carbonization of rice husks was almost complete at around 500 °C through the TG curve of Fig. 1. Through this, carbonization of rice husks and KOH activation took place at the same time to synthesize activated carbon derived from rice husks.

Characterization

XPS was analyzed to confirm the effects of KOH activation on the chemical composition of the RHC surface, and the results are shown in Fig. 2. As can be seen, three characteristic peaks of 102–105 eV (Si_{2p}), 283–286 eV (C_{1s}) and 529–534 eV (O_{1s}) appear on the surface of RHC. A weak Si_{2p} peak is observed in RHC0.5, and it

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