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Hydrogen storage on pyrolyzed chicken feather fibers

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ABSTRACT

Pyrolyzed chicken feather fibers (PCFF) that were prepared by two-step process (215 °C/15 h + 400–450 °C/1 h) demonstrated a significant H₂ adsorption uptake due to their microporous nature. Considering their large availability, cost and H₂ storage capability, PCFF can be a significant, environmentally friendly and bio-renewable candidate to address the H₂ storage problem. A wide range of microporosities was obtained when the second step pyrolysis temperature was maintained between 400 and 450 °C for 0.5–2 h. The optimal H₂ storage was obtained using 1 h pyrolysis in this temperature range. The maximum excess H₂ storage capacity was 1.5 wt% at 77 K and at pressures below 2 MPa. The notable H₂ adsorption of PCFF below 1 MPa can be justified by the abundance of microporosity, and the nanopores available for H₂ penetration. The estimated adsorption energy for PCFFs, 5–6 kJ/mol was in the range of typical physisorption materials indicating the easy recovery of H₂.

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1. Introduction

The adverse effects of fossil fuels on the earth and their limited availability require the utilization of renewable energy sources. In the near future, it is expected that an H₂ economy based on renewable energy will have a significant impact on the energy distribution. If the transition to a H₂ economy is a success, it is forecasted that H₂ powered automobiles can occupy ~30% of the traffic by 2050 [1]. However, because H₂ is not available in pure form on the earth and it is more than 8000 times lower in physical density than gasoline at ambient conditions, production and storage remain a challenge. Even liquefied H₂ or pressurized H₂ (above 35 MPa) densities are 10 and 30 times lower than ambient gasoline density. The low efficiency of these conventional storage methods in terms of energy requirement, cost and weight outlines the obvious disadvantages. The physisorption mechanism on high surface area materials provides an alternate H₂ storage route which offers fast loading and easy recovery of H₂. Particularly

microporous carbon materials such as carbon nanotubes [2–5], activated carbon [2,3,5–7], graphite, carbon nanofibers [3,4], and metal organic frameworks [8,9] (MOF) provide significant H₂ storage capacities at lower pressures (0.1–4 MPa) and considerably higher temperatures above that of liquid H₂ temperatures. The aromatic and cyclic monomer based polymer frameworks [10,11] have been developed recently, showing high microporosity along with appreciable H₂ uptake capacities. Their carbon–nitrogen and oxygen containing frameworks provided an alternative to the carbon based materials. Furthermore, they lacked heavy elements in the structure which gives them low intrinsic density [10].

When one evaluates the problem overall, it is clear that a low-cost hydrogen storage material with fast adsorption kinetics, high gravimetric and volumetric H₂ storage capacities is required. For any kind of vehicle fuel system it is desired to have at least a 500 km (300 miles) driving range [12]. The design of a recent high pressure, cryosorption based storage system [13] for a 500 km driving range demonstrates

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4.5 wt% system storage and requires approximately 30 kg of adsorbent material (AX-21) with the 29 wt% storage medium (compared to the system weight) estimation. Storing H₂ closer to ambient conditions will require even higher than 30 kg adsorbent due to the lack of extra storage resulting from the pressurization of the void space. Therefore, the environmental impact and the cost of materials must be investigated from the design stage and it is prudent to direct the research to cheap and highly available materials as discussed herein. Furthermore, note that the majority of adsorbent materials introduced above were produced from petroleum-based chemicals which question their sustainability in terms of future availability and cost.

Recently we produced a new adsorbent from chicken feather fibers (PCFF) [14], which is a renewable agricultural waste. This material exhibited promising attributes such as microporosity and high surface area, which are promising toward resolving the H₂ storage problem. When keratin-based chicken feather fibers were pyrolyzed in a N₂ atmosphere by a two-step temperature profile [14], it was observed that the fibrous structure of the keratin fibers did not change and turned into carbon–nitrogen abundant fibers [14]. The low pore size distribution of PCFF [14] encouraged us to explore adsorption and storage of small molecules on these fibers. In this article the main objective is to investigate the H₂ adsorption on PCFF and evaluate the possibility to use these fibers as a new H₂ storage material.

2. Experimental

2.1. Sample preparation

Chicken feather fibers (CFF) were received from the Featherfiber Corporation (Nixa, MO). The pyrolysis of CFF was achieved by a Thermolyne Type F6000 box furnace with a volume of 0.014 m³. N₂ gas at a flow rate of 100 cm³/min circulated through the furnace. Approximately 6 g of CFF were placed at the bottom of flat bottom crucibles and the furnace was purged at room temperature by N₂ gas for 3 h prior to the pyrolysis.

The preparation of the PCFF was similar to the two-step pyrolysis temperature profile described in Ref. [14]. The first step of the pyrolysis increased the temperature up to 215 °C with a 3 °C/min heating rate and the CFF were heat treated at this temperature for 15 h. The first step was immediately followed by a second step of the pyrolysis during which the fibers were heated to various isothermal temperatures of 350, 400, 420, 450 or 500 °C at a heating rate of 3 °C/min. The CFF were heated at the second step temperature for 0.5, 1, 1.5 or 2 h. The product was taken out after letting the furnace cool below 100 °C in an N₂ flow. The fibers were then washed by 100 cm³ toluene and 300 cm³ water subsequently in order to clean the material from impurities. Finally, the PCFF were dried overnight under a vacuum at 60 °C. The residue mass fractions of the samples after the preparation are presented in Fig. 1.

2.2. Porosity characterization by nitrogen adsorption

A Micromeritics ASAP 2010 physisorption equipment was used to determine the pore structure of the pyrolyzed chicken

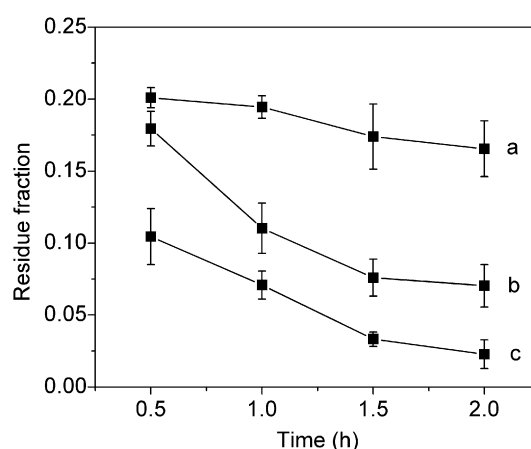


Fig. 1 – Residue mass fractions of PCFF samples prepared at 400 °C (a), 420 °C (b) and 450 °C (c) versus the pyrolysis time at the 2nd step.

feather fibers with N₂ as the analysis gas. In order to clean the surface of the PCFF from the physisorbed impurities, the samples were degassed for at least 10 h at 250 °C prior to the N₂ adsorption measurements. The BET method was utilized in order to obtain the specific surface areas (SSA). The relative pressure range for BET was 0.01–0.05 [15] due to the microporous structure of the material [14]. The micropore volumes of the samples were determined by the t-plot method [16]. The Harkins and Jura [17] equation was used to calculate the statistical adsorbate thickness. The median pore diameters were calculated by using Horvath–Kawazoe method with the slit pore assumption [18]. The existence of mesopores was determined by the Barrett, Joyner, Halenda (BJH) pore size distribution method.

2.3. Hydrogen storage

The hydrogen adsorption measurements were conducted by a custom built volumetric technique based stainless steel Sievert's apparatus. A reservoir is simply connected to a manifold with various valves to control the gas flow, vacuum and venting operations. The other end of the manifold is attached to a vertical sample holder. VCR fittings supported by copper gaskets were used in the joints within the sealed part of the system in order to minimize leaks. The copper gaskets for the sample holder were replaced whenever a new sample was loaded to avoid undesired leaks. The entire system was also leak-tested by pressurized helium gas and a Swagelok Snoop[®] leak detector before each analysis. A K-type and a T-type thermocouple with 0.2 °C accuracy were used in the temperature of the reservoir and sample holder, respectively. The pressure signals were collected by a pressure transducer capable of measuring up to 17 MPa with less than 0.1% FS accuracy.

The sample holder was loaded with approximately 1 g sample. The samples were flushed with pure helium gas multiple times at various pressures between 1 and 6 MPa at room temperature. Each sample was degassed at 300 °C overnight below 1.3×10^{-2} Pa to evacuate the micropores.

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