

Preparation of activated carbon from empty fruit bunch for hydrogen storage



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ABSTRACT

There are many advantages associated with the use of hydrogen as clean fuels, thus there is a need to search for good hydrogen adsorbents. Activated carbon (AC) from various sources has been tested as hydrogen adsorbents, with the exception of AC produced from empty fruit bunch (EFB). The aim of this study is to produce AC from EFB obtained in Malaysia, followed by testing on its hydrogen storage capacity. The AC samples were activated using physical and chemical processes. The total surface areas (S_{BET}) and microporous structures of the ACs produced were in the range of 305–687 m²/g and up to 94%, respectively. The hydrogen storage capacity was studied at constant temperature, 77K and pressure from ambient pressure up to 100 bar. The hydrogen uptake increased occasionally from 0 bar to 20 bar and decreased above 20 bar. EFB activated with 2M KOH demonstrated maximum hydrogen adsorption capacity with 2.14 wt% hydrogen uptake at 20 bar. More research is needed to improve the hydrogen adsorption capacity on AC, and to determine its potential applications other than mobile applications.

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1. Introduction

Hydrogen is one of the renewable resources that can help in addressing the growth of energy demand without contributing to the global climate change [1], as it contains high energy density (on weight basis), and is readily available from renewable source. In addition, it is non-pollutant since water is the only by-product during the conversion of hydrogen to energy. Hydrogen has been used as an alternative for traditional sources of energy like oil and gas for more than a decade. Various efforts have been carried out to expand the application of hydrogen as an energy source, particularly in transportation sector.

US Department of Energy (DOE) has put minimum gravimetric and volumetric requirement for onboard hydrogen storage systems. However, the current market and technology scenario has led to some revisions and changes in targets. In 2010, the targeted storage system has a gravimetric density of 4.5 wt% (1.5 kWh/kg) and volumetric density of 28 g/L (0.9 kWh/L), while for 2015, the targeted gravimetric density was changed to 5.5 wt% (1.8 kWh/kg), and the volumetric density was revised to be 40 g H₂/L (1.3 kWh/L). However,

it is difficult to achieve these targets at room temperature as Fierro et al. [2] found that the maximum gravimetric adsorption of 5.3 wt% was reached at 40 bar and 77K, while Zhao et al. [3] obtained gravimetric adsorption of 6.6 wt% under the same operating conditions, both for activated carbons. Considering that the DOE targets are for system applications, the hydrogen adsorption for the materials has to be higher to be useful in practical applications.

Several technical solutions are anticipated to solve the aforementioned issue, including compression or liquefaction of hydrogen gas, storage in metallic hydrides, chemical storage and hydrogen storage by physisorption. However, liquefaction of hydrogen requires high amount of energy [4], while compression requires operation at very high pressure, which leads to high operation cost with safety issues [5]. On the other hand, metallic hydrides are suspected to be poisonous, while having incomplete reversibility and low kinetics behavior [2]. Thus, physisorption storage system on carbon materials is an interesting alternative, as it does not require high operation pressure, and is completely reversible with fast kinetics [2] and relatively offers higher hydrogen storage capacity. Among the carbon materials, those derived from biomass are highly potential alternatives for hydrogen storage that are safe, have high adsorption/desorption rates, as well as environmentally friendly and economically feasible [6,7]. Activated carbon (AC) is a microporous material

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which can store hydrogen at high capacity due to its well-developed porosity, high surface area and chemical stability. There are a number of studies that indicate the feasibility of AC to be used in practical application for hydrogen storage [2,6,8–10].

Empty fruit bunch (EFB) can be a potential source material for production of AC, as it has high surface area, well-developed porosity, good chemical stability and is widely available as waste from oil palm industry. Several potential applications of AC have been studied, mainly in removal of organic pollutants [11–13]. However, to the best of the authors' knowledge, there is no study on hydrogen storage by activated carbon produced from EFB, which may give several advantages on the storage system. Therefore this study was conducted to determine the hydrogen adsorption capacity of EFB AC prepared by physical and chemical activations with KOH.

2. Materials and methods

2.1. Synthesis of activated carbon (AC) from EFB

The empty fruit bunches (EFB) sample was obtained from Malaysia Palm Oil Board (MPOB). The sample was first dried and weighed prior to carbonization in a tube furnace (Barnstead, Thermolyne). The tube furnace was set up horizontally, and the EFB sample was heated to 500 °C at a heating rate of 10 °C/min, followed by heating at mentioned temperature for 80 min. Nitrogen gas was flown through the reactor at 1.5 L/min throughout the heating to ensure inert condition and prevent oxidation of the sample. The AC produced was then activated under CO₂ gas flow (900 °C, 0.1 L/min CO₂, 15 min activation time) according to method described by Alam et al. [14].

The activation process was followed by KOH impregnation based on method used by Sun et al. [15] with some modifications. Four different concentrations of KOH solutions were used in this study (0.5M, 1.0M and 2.0M), and each sample of AC (4 g) was soaked and stirred with 100 mL of potassium hydroxide (KOH). The mixtures were left for 2 h at room temperature. Next, all the samples were washed with hot distilled water until the washing effluent reached pH 7.0. The samples were then dried in the oven at 105 °C overnight before being analyzed and characterized. The ACs activated by CO₂ gas and impregnated by 0.5M, 1.0M, and 2.0M KOH are denoted as AC1, AC2 and AC3 respectively.

2.2. Characterization

The surface chemistry of all ACs was identified using a Fourier transform infrared spectroscopy (Perkin-Elmer). The spectra was recorded in the range of 650–4000 cm⁻¹. The total surface area and pore volume of samples were identified using nitrogen adsorption at 77K with an accelerated surface area and porosimetry system (ASAP-2020, Micromeritics). Brunauer-Emmett-Teller (BET) model was applied in calculation of the specific surface areas, S_{BET} (m²g⁻¹). The pore volume, V_{pore} (cm³g⁻¹) and pore diameter, ϕ_{pore} (nm) of the samples were calculated using result from Barrett-Joyner-Halenda (BJH) and t-plot analysis respectively. The surface morphology of the samples was identified using Scanning Electron Microscopy (SEM) (Hitachi, S-3400N model).

2.3. Hydrogen adsorption capacity

The measurement of hydrogen adsorption capacity at 77K and up to 0.1 MPa was carried out on the samples by automated gas adsorption analyzer system (ASAP 2020). The volumetric analyzer was used to determine the adsorption and desorption isotherms at every pressure step to be studied. The samples were degassed at 300 °C for 12 h in the preparation port prior to the analysis to

remove moisture and any stray gases. The samples were then degassed thoroughly in the sample port. A standard ASAP 2020 tube (1/2 in. stem) with a seal frit was used to hold the sample. An isothermal jacket was used during analysis at cryogenic temperature.

The identification of hydrogen storage at 77K and up to 10 MPa was carried out using high pressure volumetric analyzer (HPVA-Micromeritics). High pressure hydrogen adsorption isotherm was performed using static volumetric method. The sample was degassed at 100 °C for 20 h. Hydrogen gas was introduced into the chamber containing about 0.3 g of AC sample. The final equilibrium pressure was recorded once the sample reached equilibrium with the hydrogen gases. The data were used to identify the amount of gas that had been adsorbed by the AC. The process was repeated at constant pressure range up to 100 bar.

3. Results and discussion

3.1. Sample characterization

3.1.1. Functional groups

The FTIR spectrum of AC prepared from EFB with CO₂ activation and treatment with different concentrations of KOH is shown in Fig. 1. In general, all samples displayed the same bands: 2700–3700 cm⁻¹ (O–H and N–H stretches); 2100–2250 cm⁻¹ (C–N stretches); 1900–2000 cm⁻¹ (C=C asymmetric stretching); 1750–1650 cm⁻¹ (C=O stretching); 1675–1575 cm⁻¹ (C=C stretching); 1300–1950 cm⁻¹ (C–O stretching); 900–650 cm⁻¹ (C–H bending) [16]. The results show that the impregnation with KOH with concentration of 0.5–2.0 M did not produce any significant change on the surface chemistry of AC.

3.1.2. Nitrogen adsorption isotherm analysis

According to IUPAC classification, the AC nitrogen adsorption-desorption isotherm for AC1, AC2 and AC3 in Fig. 2 were identified as Type-I isotherm [17]. Such classification indicates that predominantly microporous carbon materials were obtained by activation with CO₂ gas and KOH.

According to Fig. 2, the increase in KOH concentration during the treatment increased the volume of N₂ adsorption. AC3 had the largest BET surface area compared to the other ACs. KOH behaved as a dehydration agent which led to the pore opening, hence leading to formation of porous structure and increased sample surface area. Thus, the greater amount of KOH concentration of AC3 had resulted in a higher surface area and more pore structures [18]. Total surface area, micropore volume and micropore percentage of the prepared activated carbons are shown in Table 1. The total surface area of samples was in the range of 305 m²/g–687 m²/g.

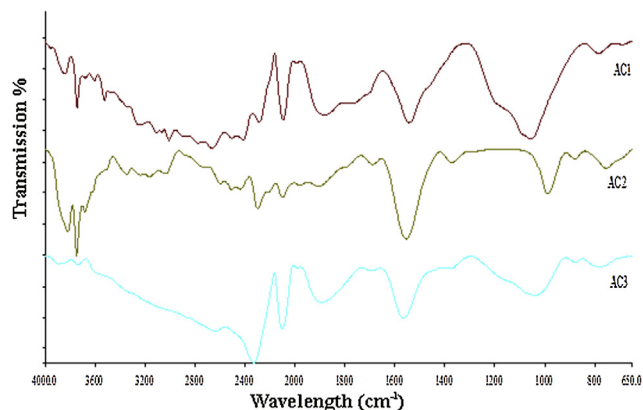


Fig. 1. IR-spectrum of samples AC1, AC2 and AC3.

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