



ELSEVIER

Available online at www.sciencedirect.com

ScienceDirect

journal homepage: www.elsevier.com/locate/he

Synthesis, characterization and hydrogen storage characteristics of ambient pressure dried carbon aerogel

Sweta Singh^a, Ashish Bhatnagar^a, Viney Dixit^a, Vivek Shukla^a,
M.A. Shaz^a, A.S.K. Sinha^b, O.N. Srivastava^{a,*}, V. Sekkar^{c,**}

^a Hydrogen Energy Centre, Department of Physics, Banaras Hindu University, Varanasi 221005, India

^b Department of Chemical Engineering and Technology, Indian Institute of Technology, Banaras Hindu University, Varanasi 221005, India

^c Application Development Division, Vikram Sarabhai Space Centre, Indian Space Research Organization, Trivandrum, Kerala 695022, India

ARTICLE INFO

Article history:

Received 6 October 2015

Received in revised form

12 December 2015

Accepted 26 December 2015

Available online 21 January 2016

Keywords:

Carbon aerogel

Submicropore

Hydrogen storage

ABSTRACT

The present communication deals with the hydrogen storage performance of ambient pressure dried pristine as well as platinum doped carbon aerogel (CA-0.10 Pt). These carbon aerogels (CAs) have been prepared from resorcinol-formaldehyde (R-F) through sol-gel synthesis route with sodium carbonate as a catalyst (C). The synthesis parameters adapted led to the formation of CA having preponderance of submicropores. Structural and microstructural characteristics of these carbon aerogels have been investigated through XRD, SEM, TEM, nitrogen adsorption and Raman spectroscopic techniques. Nitrogen adsorption and TEM studies confirm the large density of micropores with the majority of pores having sizes between 0.30 and 1.46 nm (submicropores). The hydrogen storage characteristics of as synthesized carbon aerogels have been investigated by monitoring the hydrogen ad/desorption curves. At room temperature and at pressure upto 22 atm the CA and CA-0.1 Pt have hydrogen storage capacity of 0.40 wt.% and 0.33 wt.% respectively. However, under the same pressure but at liquid nitrogen temperature CA and CA-0.10 Pt have hydrogen storage capacity of 5.65 wt.% and 5.15 wt.%. Feasible reasons for the high hydrogen storage capacities at liquid nitrogen temperature for the present CAs have been put forward.

Copyright © 2016, Hydrogen Energy Publications, LLC. Published by Elsevier Ltd. All rights reserved.

Introduction

Carbon aerogels are relatively new class of materials associated with a wide variety of applications, including hydrogen

storage [1–4]. Hydrogen is considered to be the promising upcoming green fuel. It has the potential to replace the conventional fossil energy sources like coal and petroleum. Hydrogen takes care of the two detrimental effects associated

* Corresponding author. Tel.: +91 0542 2368468.

** Corresponding author.

E-mail addresses: heponsphy@gmail.com (O.N. Srivastava), vsekkar@gmail.com (V. Sekkar).

<http://dx.doi.org/10.1016/j.ijhydene.2015.12.174>

0360-3199/Copyright © 2016, Hydrogen Energy Publications, LLC. Published by Elsevier Ltd. All rights reserved.

with using the fossil fuel, the pollution and the climate-change effects. For specific usage of hydrogen, particularly for fuel cells, its storage is the main concern and has become a challenging topic of contemporary research [5,6]. Several techniques like (a) compression of gaseous hydrogen (b) liquefaction (c) storage in the form of solid hydrides and porous materials have been investigated [7–9]. The porous materials are an attractive option for hydrogen storage as they are light weight, exhibit nearly complete reversibility, possess faster kinetics and manageable pressure of hydrogen ad/desorption [10,11]. A wide range of porous carbon materials have been investigated, including graphene, SWCNT, CA and many others [11]. The hydrogen storage capacity of these materials typically lies between 1.70 wt.% and 5.70 wt.% at liquid nitrogen temperature [12–16] which satisfy the DOE limits of storage (2017: 5.50 wt.%, 40 kgH₂/L) [17]. Out of various porous solids, CA shows promising hydrogen storage properties particularly at liquid nitrogen temperature, owing to their continuous porosity, ultrafine pore size and high surface area [3]. Apart from hydrogen storage applications, CAs also have several other attractive applications, such as an active electrode material for supercapacitors, batteries, fuel cells, gas filtration membranes, reinforcing agents for natural rubber, light weight structural composites for space explorations, etc. [18–23]. All these applications strongly depend on structural and physicochemical properties of the CAs [24,25]. It is very important that the tuning of these properties is achieved by suitable adjustment of the synthesis conditions i.e. precursor composition, type of catalysts, their concentrations and also on drying procedure and pyrolysis temperatures. Several methods for the synthesis of carbon aerogel have been developed [19,24,26–28]. Conventionally, mesoporous RF aerogels are obtained by evaporating the liquid embedded in the RF-gels under supercritical conditions. Supercritical drying ensures negligible shrinkage during the drying process [29]. However, supercritical drying involves a very high pressure which renders the drying process unsafe and uneconomical. On the other hand, subcritical (ambient pressure) drying process offers a more economical and industrially scalable route to obtain high quality carbon aerogels [29]. Also, the eventual microstructure of the CAs is known to depend upon the concentration of the reacting species and also on the concentration of the catalyst with respect to resorcinol [15]. Therefore, in the present study, we have adopted ambient pressure drying with optimum resorcinol (R): formaldehyde (F) and R: catalyst (C) ratios followed by carbonization for the preparation of carbon aerogel. The as synthesized CAs has been characterized by XRD, SEM, TEM, nitrogen adsorption and Raman spectroscopic techniques. It has been found that the aerogel synthesized in the present study has high density of micropores with pore size predominantly between 0.30 and 1.46 nm which may be termed as submicropores. This is very useful for hydrogen storage with high capacity [1]. The hydrogen storage characteristics have been monitored by recording hydrogen ad/desorption curves. The present studies are unique in the sense that near ambient condition, dried carbon aerogels with high density of micropores have been deployed for hydrogen storage. Only scant studies on the synthesis of CAs with near ambient condition drying and hydrogen storage

characteristics thereafter have been reported [3,4,28]. An important aspect emerging from the present studies is that CAs prepared through ambient drying, possess high hydrogen storage capacity (~5.13–5.65 wt.%) at liquid nitrogen temperature. The work described here suggests that, further research is required to tailor synthesis of CAs, so that they possess high density of submicropores. Also investigations need to be done to understand the details of hydrogen storage in CAs through micropore filling which corresponds to condensation like characteristics.

Experimental

Synthesis of carbon aerogel and platinum doped carbon aerogel through ambient pressure drying method

In the present investigation our objectives were to obtain CAs with negligible shrinkage which was achieved by ambient pressure drying. Moreover, it was envisaged to synthesize such CAs which has a preponderance of micropores with negligible density of mesopores and macropores. The CAs were synthesized from resorcinol (99%) (Merk speciality chemicals India Ltd), formaldehyde (37% in water, Poly-formalein Pvt Ltd; Cochin, India) and sodium carbonate (NICE India Ltd.). Several variations of the concentrations of reactant and catalyst were tried. For each of them, microstructure was determined through TEM and SEM. The optimum concentration for achieving highest density of micropores was found to be 1.0 mol of resorcinol to 2.0 mol of formaldehyde i.e. R:F is 1:2 in distilled water with total solids being 30%. Sodium carbonate was used as catalyst and optimum R:C molar ratio was 1500:1 (for the formation of micropores). The precursors were mixed by magnetic stirring for 3 h in a sealed container. After that the mixture was gelled at room temperature for 24 h. The aquagel was then aged in an oven at 50 °C for 24 h and 90 °C for 72 h. In order to promote additional crosslinking of the gel, 2% acetic acid was added to the solvent during the initial water to solvent (acetone) exchange for 24 h. Then the RF wet gel was subjected to acetone exchange at 50 °C for 72 h, where acetone was replaced with fresh acetone once in 24 h. Finally, we have carried out ambient pressure drying of the RF wet gel at three different temperatures i.e. 25 °C, 50 °C and 80 °C. It was found that the optimum microstructure was obtained when drying was done at 50 °C. Therefore, the RF wet gel was allowed to dry at 50 °C (optimum temperature) for 72 h in the acetone medium. The dried RF aerogel samples were then carbonized by heating it under argon atmosphere (Ar flow rate = 2 l/min) by adopting the following heating programme: room temperature to 1050 °C at a heating rate of 1 °C/min and hold the sample at 1050 °C for three hours. Afterwards the samples were cooled from 1050 °C to 300 °C at a heating rate of 0.5 °C/min and then from 300 °C to room temperature by ambient cooling. Fig. 1 shows the schematic protocol for synthesis of carbon aerogel. Besides the above described pristine CAs, platinum doped CAs (CA-0.10 Pt) were synthesized by gelling the resorcinol-formaldehyde system in the presence of hexachloroplatinic acid solution (0.10 M) followed by the same procedure as adopted for the synthesis of pristine CA.

Download English Version:

<https://daneshyari.com/en/article/1270258>

Download Persian Version:

<https://daneshyari.com/article/1270258>

[Daneshyari.com](https://daneshyari.com)