

# Hydrogen storage on boron substituted carbon materials



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#### ABSTRACT

Hydrogen storage capacity of boron substituted carbon materials synthesized by the carbonization of resorcinol and triethylborate as carbon and boron sources respectively is reported. The effects of the boron-doping and carbonization temperature and the role of functional groups have been investigated. The hydrogen adsorption capacity has been studied with a high pressure volumetric analyzer (HPVA) and a maximum of 5.9 wt% hydrogen storage capacity was observed at 298 K and 100 bar pressure. At 77 K in both carbon samples (namely BC-600 or BC-800) show only fractional weight percent of hydrogen sorption (1.1 and 0.5 wt%). The nearly 5 weight % hydrogen storage was obtained by loading heteroatom (B) to carbon sample at 298 K and 100 bar.

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#### Introduction

Hydrogen storage is an important component for operation of transport sector in green energy mode. Hydrogen storage is particularly challenging as evolving a safe and efficient hydrogen storage medium with the required capacity appears to be difficult. Hence it has evolved as one of the foremost research topics of material research scientists. The US-DOE specifies a hydrogen storage capacity for on board automobile application as 9.0 wt% to be achieved by 2015. The question at this time is can one meet this target for the hydrogen energy storage capacity within the time frame available? [1–7]. There are three possible ways for hydrogen storage

namely compression, liquefaction and storage in solids. The first two options have many issues like demand on materials, high cost and logistics of the application [7]. In the case of solid-state hydrogen storage, carbon materials have been examined as one of the possible options. In view of the possible promising traits, hydrogen storage using nanostructured carbon materials has quickly gained as one of the hot areas of research in science and technology [8]. The carbon nanostructures also find potential applications in various other areas like super-capacitors [9], energy conversion systems [10] and fuel cell [11]. The scientific society in their anxiety has examined variety of carbon materials for sufficient hydrogen storage and also focused on realizing reproducible results for hydrogen storage. The minimum storage

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capacity for commercial application has been estimated to be 6.5 wt% as postulated by DOE. It is necessary to recall that hydrogen storage up to 67 weight % has been reported in exotic carbon materials, though the reproducibility of the result has not been established [8,12-15]. Various types of carbon-based adsorbents have been proposed for hydrogen storage, including carbon nanotubes (CNTs), graphite nanofibers (GNFs), activated carbons (ACs), templated carbons (TCs), and graphite [3,16-27]. The introduction of heteroatom like nitrogen, sulphur, boron and phosphorus in carbon materials has been shown to produce activation centres for dissociation of molecular hydrogen and thus possibly facilitates hydrogen storage. Even though reversible nature of the hydrogen absorption/desorption process on/in carbon-based materials and high surface area sorbents has been realized, they suffer from lower hydrogen storage capacity, especially under desirable operating conditions. However, irrespective of their high surface area, the presence of heteroatom on the surface of carbon materials has been regarded as an attractive feature to increase the hydrogen storage capacity [28-41].

In the present study, boron substituted carbon materials have been examined for hydrogen storage capacity. It may be recalled that boron substitution in adjacent and alternate positions in carbon framework may be the two possible centres and the preference is for alternate positions of substitution as favourable hydrogen storage. The boron substituted carbon material has been synthesized using resorcinol and triethylborate as carbon and boron sources respectively. The structure, composition of the material, morphological aspects and composition of the functional groups of the synthesized carbon material are investigated by Raman spectroscopy, XRD, electron microscopy and FT-IR. The hydrogen storage capacity of the boron substituted carbon material has also been determined by volumetric high pressure unit.

#### Materials and methods

Resorcinol 99% ( $C_6H_6O_2$ ), formaldehyde 37% (HCHO) solution, tri-ethylborate 97% (TEB) solution, were purchased from Sigma Aldrich and used without further purification.

#### Experimental section

The typical synthesis procedure for boron substituted carbon material is as follows: 11.2 g of resorcinol was taken in a beaker, 30 ml of formaldehyde solution was added stirred at room temperature for 1 h, 20 ml of triethylborate (TEB) solution was added stirred well, kept in an oven at 373 K for 24 h. The as-synthesized materials were placed in an alumina crucible and subsequently transferred to a tube furnace for carbonization. The furnace was purged with nitrogen gas at room temperature for 1 h. Then the temperature was increased at a rate of 5 °C/min up to 200 °C and maintained at 200 °C for 5 min to remove trace adsorbed contaminants. After 5 min, the temperature was increased at a rate of 10 °C/min to the target temperature (6000 and 800 °C). Upon reaching the target temperature, the temperature was maintained an additional 6 h. Then the furnace was cooled to room temperature in the presence of nitrogen. Throughout this

procedure, the furnace was continually purged with nitrogen gas. The resulting carbon material was ground into a fine powder using a mortar and pestle. The material was then transferred into a scintillation vial for storage. Finally obtained boron containing carbon materials denoted as BC-600 and BC-800 was subjected to further characterization. A similar synthesis procedure was adopted to prepare pure carbon material without boron for comparison purpose.

#### Physical measurements

Wide angle Powder XRD patterns of the calcined carbon materials were recorded using a Rigaku Miniflex II diffractometer with Cu K $\alpha$  as the radiation source at a wavelength of 0.154 nm with  $2\theta$  angle ranging from  $10^{\circ}$  to  $80^{\circ}$  with a 0.02 step size. Fourier Transform Infrared Spectra (Perkin-Elmer FT-IR spectrophotometer) were collected at room temperature by using the KBr pellet technique in the range  $4000-400 \text{ cm}^{-1}$ . The vibrational characteristics of the samples were analyzed via Raman spectroscopy using 532 nm laser (Witec Alpha 300) as the excitation source in the range from 1000 to 2000  $\text{cm}^{-1}$ . BET N<sub>2</sub> adsorption and desorption isotherms were measured with surface area and porosity analyzer (Micromeritics Accelerated Surface Area and Porosimetry System (ASAP 2020)). Prior to the adsorption measurements, the sample was degassed at 473 K for 6 h. FEI Quanta FEG 200-High Resolution Scanning Electron Microscope (HRSEM) was employed for obtaining the micrographs. JEOL JEM-2100 High Resolution Transmission Electron Microscope (HRTEM) was employed for obtaining the micrographs, Elemental mapping and SAED patterns. X-ray photoelectron spectroscopy (XPS) measurements were performed with an Omicron Nanotechnology spectrometer with hemispherical analyzer. The monochromatized Mg K $\alpha$  X-source (E = 1253.6 eV) was operated at 15 kV and 20 mA. For the narrow scans, the analyzer pass energy of 25 eV was applied. The base pressure in the analysis chamber is  $5 \times 10^{-10}$  Torr. The hydrogen adsorption isotherms were carried out on high pressure volumetric analyzer (HPVA-100) from micromeritics particulate systems. The HPVA product operating pressure ranges from high vacuum to 100 bar. The sample temperature can be during analysis from cryogenic to 500 °C. Analysis and data collection are fully automated to assure quality and high reproducibility of data.

#### **Results and discussion**

#### X-ray diffraction pattern

The X-ray diffraction patterns of the synthesized boron containing carbon materials are shown in Fig. 1. The carbon material BC-600 shows two broad diffraction reflections centred at 20 values of  $23.9^{\circ}$  and  $43.6^{\circ}$  corresponding to reflections from (002) and (100) planes of carbon. However for the carbon material BC-800 showed reflections at 20 values at  $24.1^{\circ}$  and  $44.2^{\circ}$  thus showing the synthesized materials possess carbon lattice, the broad reflections indicate the crystalline nature of the synthesized material. The pure carbon material shows broad diffraction at  $23^{\circ}$  and  $42.5^{\circ}$ . As the calcination temperature is increased, the intensity of the diffraction peaks Download English Version:

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