



Fabrication of high pressure hydrogen adsorption/desorption unit—Adsorption study on flame synthesized carbon nanofibers

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ABSTRACT

An in-house custom made high pressure adsorption/desorption unit has been designed and fabricated to study reversible hydrogen (H₂) intake capacity, hysteresis, kinetics, plateau pressure of various nanomaterials, zeolites and metallic compounds, in the pressure range of $1 \leq P \leq 150$ atm. The unit has been used to estimate H₂ intake capacity of carbon nanofibers prepared by flame synthesis in the absence of catalyst. H₂ adsorption studies have been carried out in the pressure range of 25–100 atm at 297 K. The maximum H₂ intake capacity has been observed to be 3.7 wt% at 100 atm.

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1. Introduction

Hydrogen is considered as an eco friendly and an ideal fuel for future, which can replace the fossil fuels due to their by-product, water, has a large heat of combustion, 287 kJ/mol [1]. Practical application of molecular hydrogen as a fuel is feasible due to its low critical temperature of 32.97 K. The major challenging aspect in developing an efficient energetic application is the availability of lightweight material with high degree of physical and surface interactions [1–3]. These interactions ensure reversibility in hydrogen storage. In recent years, a rapid development has been noticed in hydrogen adsorbing materials such as carbon nanomaterials, metal hydrides and zeolites [4–7]. Previously, researchers have extensively carried out adsorption studies under ambient condition (297 K) on only carbon nano tubes produced by various processes like chemical vapor deposition [8–13], arc discharge [14–18], polymer blend technique [19] and catalytic decomposition [20–22]. Many have reported very low to high range of gravimetric adsorption rates of hydrogen in various carbon nanomaterials and majority of the adsorption experiments have been carried out on CVD based nanocarbons, but none has so far reported adsorption of hydrogen on flame synthesized carbon nanofibers. Flame synthesis process has been used to produce bulk quantities of nano metal oxides of greater crystallinity, morpholo-

gy and quality [23,24]. It has also been used to produce various carbon nanostructures [25,26].

In the present work we discuss in detail the design and development of hydrogen adsorption unit, Flame synthesis of carbon nanofibers (CNFs) in the absence of catalyst, its characterization, hydrogen adsorption calculation, procedure and results. It is to be noted that many researchers have classified CNFs as nanofilament, which differs greatly with its counterpart like nanotubes due to the arrangement of graphene layers in different directions along the fiber axis.

2. Materials and methods

2.1. Flame synthesis of carbon nanofiber

Carbon nanofibers have been synthesized in a flame reactor in the absence of catalyst. The design details and procedure of the flame reactor and synthesis have been discussed elsewhere in detail in our previous work [27,28]. Carbon nanofibers have been synthesized without catalyst in meth-oxy-acetylene diffusion flames under different flow rates of methane as 0.4, 0.8, 1.2 and 1.6 slpm respectively. Whereas, the flow rate of acetylene and oxygen have been kept constant at 0.02 slpm and 0.2 slpm respectively throughout the experiments. The experiment is allowed to run for thirty minutes and the soot obtained is then purified in an open air furnace at 723 K for 1 hr to remove an impurity like amorphous carbon. Later the same have been characterized for crystallinity, morphology and surface area. The

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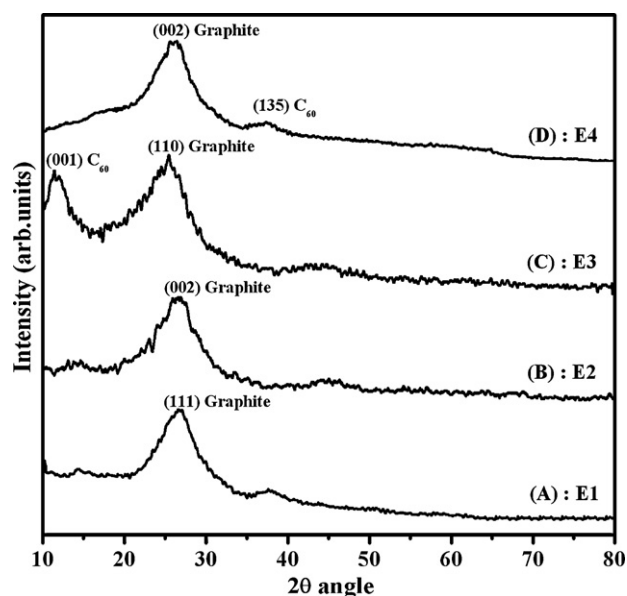


Fig. 1. XRD of flame synthesized carbon nanofibers produced at different flow rates of methane (0.4–1.6 slpm) and constant flow rate of acetylene (0.02 slpm) and oxygen (0.2 slpm) respectively. The samples have been coded as E-1 to E-4 in alphabetical chronology (a) E-1 (CH_4 : 0.4 slpm; C_2H_2 : 0.02 slpm; O_2 : 0.2 slpm); (b) E-2 (CH_4 : 0.8 slpm; C_2H_2 : 0.02 slpm; O_2 : 0.2 slpm); (c) E-3 (CH_4 : 1.2 slpm; C_2H_2 : 0.02 slpm; O_2 : 0.2 slpm); (d) E-4 (CH_4 : 1.6 slpm; C_2H_2 : 0.02 slpm; O_2 : 0.2 slpm).

samples are then subjected to hydrogen adsorption at high pressure using the in-house fabricated high pressure adsorption unit.

2.2. Characterization

The CNFs have been subjected to X-ray diffraction (PW1830 Phillips X-ray Diffractometer) studies (Fig. 1) operated at 40 kV and

30 mA current using a graphite monochromator and $\text{Cu K}\alpha$ radiation. The diffractograms have been obtained by increasing step size at the rate of $2^\circ/\text{min}$, in the scanning angle (2θ) range of 10 – 80° . The peaks (signals) have been analyzed using PDF-2 (ICDD) database 2003. The sample has been further analyzed using SEM (Fig. 2) for surface morphology using Phillips XL 30 Series field emission scanning electron microscope operating at an accession voltage between 20 and 25 kV at 10^{-5} mbar vacuum. The sample has been analyzed by sticking the sample powder on a double sticking conducting carbon tape affixed on an aluminum stub. Transmission electron microscope (Hitachi H7500) operated at 200 kV has been used for TEM analysis (Fig. 3). A very small amount of the sample has been sonicated in 5 ml of ethanol for 5 min at 75% power with a sonicating frequency of 33 kHz. Later a drop of the sonicated solution was added on to the copper grid and analyzed. The nitrogen (N_2) BET surface area measurement have been carried out for carbon nanofibers using surface area analyzer (SMART SORB 93) by pre-treating the samples at 473 K for 2 h under N_2 purging. N_2 adsorption and desorption isotherms obtained at 77 K have been used to calculate BET surface area. The results have been discussed in Section 3.

2.3. Hydrogen adsorption unit

The high-pressure hydrogen absorption/desorption, Piping & Instrumentation Diagram (P & ID) and experimental setup are shown in Figs. 4 and 5 respectively. It comprises of two units (unit-1 and unit-2), each having its own adsorber assembly and a measuring section. In this experimental setup, the stainless tubes, tees, elbow joints and needle valves procured from Swagelok (USA) have been used. They can withstand up to a maximum pressure of 150 atm. The pressure transducers procured from Keller (USA) have been used to measure pressure in the range of 1–150 atm.

2.3.1. Description of unit-1

The hydrogen adsorption studies of CNFs can be carried out in two units (unit-1 and unit-2). Their description is as given below.

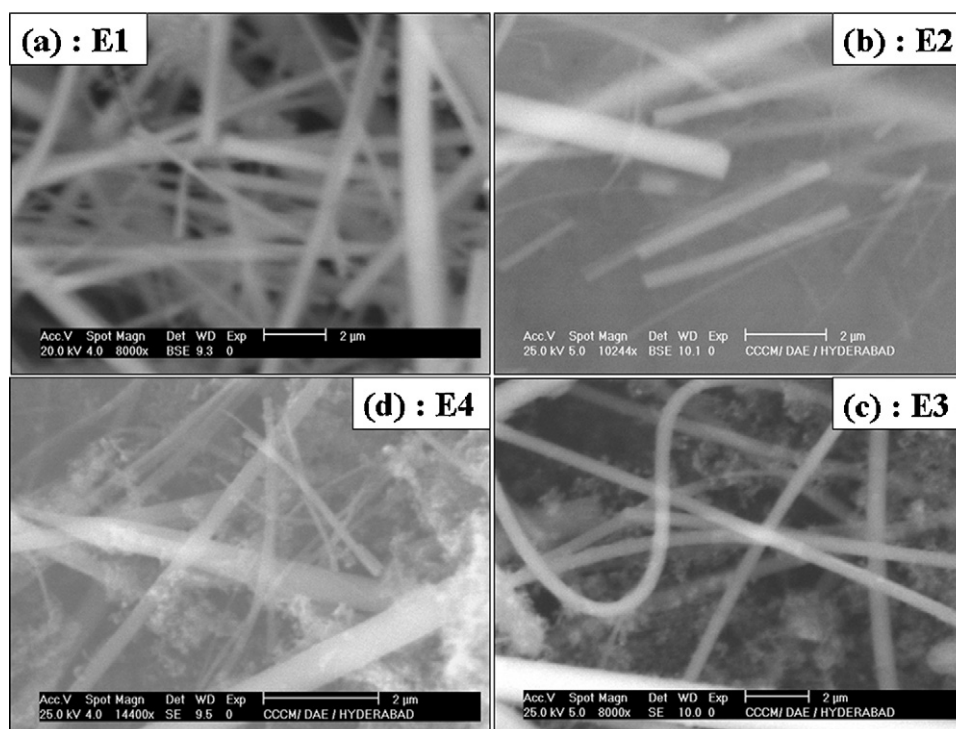


Fig. 2. SEM image of flame synthesized carbon nanofibers.

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