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The synthesized nickel-doped multi-walled carbon nanotubes for hydrogen storage under moderate pressures

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ARTICLE INFO

Article history:

Received 15 October 2017

Received in revised form

30 December 2017

Accepted 15 January 2018

Available online xxx

Keywords:

Hydrogen storage

Nickel-decorated multi-walled

carbon nanotubes

Surface area

Adsorption

ABSTRACT

Hydrogen adsorption capacity of Multiwalled carbon nanotubes (MWCNTs) decorated with Nickel (Ni) nanoparticles has been presented at room temperature and under moderate pressures of 4–20 bar. The functionalization of carbon nanotubes was carried by H₂SO₄-HNO₃ reducing agents and the Ni supported MWCNTs (Ni-MWCNTs) were prepared by wet chemical method. The structure and morphology characterization of samples were performed by XRD, TEM, EDX and SEM analyses. These nanotubes then subjected to hydrogenation step by using Sievert's-like apparatus. The hydrogenation of the Ni-MWCNTs was performed at 298 K and moderate hydrogen pressures of 4–20 bar. The obtained results show that there is a correlation between hydrogen storage capacity and hydrogen pressure that; as the pressure was increased, hydrogen uptake capacity enhanced due to physisorption. In addition, maximum hydrogen storage capacity of Ni-MWCNTs was found to be 0.298 wt % at room temperature and under pressure of 20 bar.

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Introduction

Renewable energy sources are becoming increasingly widespread because of increasing energy consumption, decreasing fossil-based energy reserves, and intensifying environmental problems due to the use of fossil-based energy resources [1]. Production of hydrogen energy has been increased in recent years because hydrogen energy does not harm the environment and can be produced in a sustainable way [2]. The most important characteristics of hydrogen energy, namely the characteristics that distinguish it from other alternative energy sources, are its capacity to be widely produced, its low weight and energy density. Although many production methods exist

for hydrogen energy, storage methods are still limited and problematic [3]. Adsorbent materials have frequently been used in the storage of hydrogen energy in recent years to overcome these problems and the best performing of these adsorbent materials are carbon nanotubes [4,5]. Carbon nanotubes store hydrogen up to 14 wt % due to their mechanical and electrical properties, large surface areas and abundant pore volume [6]. There are single walled and multi walled carbon nanotubes produced in general, however producing a high volume of single walled carbon nanotubes is difficult, multi-walled carbon nanotubes (MWCNTs) are preferred to single-walled carbon nanotubes in hydrogen storage [7–9].

Dillon et al. claimed that Single walled carbon nanotubes (SWCNTs) adsorb hydrogen between 5 and 10 wt % inside the

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<https://doi.org/10.1016/j.ijhydene.2018.01.084>

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pores of molecular dimensions [10]. This result led to intense interest in carbon nanotubes for hydrogen adsorption. However, Hirscher et al. was measured SWCNTs hydrogen adsorption capacity up to 0.01 wt % by thermal desorption spectroscopy at ambient pressures and temperature. They also noted that carbon nanotubes must be used in high quantities when storing hydrogen, otherwise the amount of stored hydrogen cannot be accurately measured [11]. Moreover, Zhu et al. found hydrogen adsorption capacity to be 5 wt % in MWCNTs at 300 K under 10 MPa pressure [12]. However, Tibbetts et al. found that SWCNTs and MWCNTs uptake hydrogen less than 0.1 wt % under a pressure of 3.5 MPa and at room temperature [13]. The differences in the results of the studies of the carbon nanotubes storage may be due to the weakness of the Van der Waals bonds which is established between hydrogen carbon nanotubes.

The adsorbent capacity, and hence the hydrogen storage capacity of MWCNTs can be increased by adding various metals. Adding various metals and chemical compounds to the nanotubes increases the porosity, the dipole–dipole interaction, and thereby enables higher hydrogen storage capacity [14]. Numerous studies have demonstrated that hydrogen uptake capacity is enhanced by doping metals and chemical compounds such as Ni, Pd, Pt, Li, TiO₂, Al₂O₃, ZnO, BN [15–17]. The doped nanotubes are used also for many highly demanding applications such as lead removal, heavy metal removal, energy storage, biosensors, agriculture, field-emitting devices etc. [18–21].

Furthermore, Zacharia et al. claimed that metal nanoparticles may increase hydrogen uptake approximately by 30% on carbon nanotubes via spill over mechanism. It was cited that the capacity of hydrogen adsorption on Pd-V doped MWCNTs was measured 0.69 wt % at 2 MPa by using Sievert's-like apparatus [22]. Rather et al. indicated that TiO₂ doped carbon nanotubes adsorbed 0.40 wt % of hydrogen under pressure of 18 bar, also this result exhibited this nanotubes hydrogen adsorption capacity five times higher than purified CNTs. Studies conducted so far have shown that the surface modification of carbon nanotubes is done via metals and chemical compounds which is responsible for high amount of hydrogen storage [22–24].

The hydrogen uptake capacity of Ni-MWCNTs was studied by Kim et al. and founded that Ni nanoparticles are spilled over on MWCNTs by incipient wetness impregnation method and the spreading of Ni nanoparticles over MWCNTs allows the hydrogen to make chemical bonds on the surface of MWCNTs. Additionally, they showed that 2.8% of Ni-MWCNTs deposited hydrogen approximately 1.2 wt % at ambient temperature [24].

Reyhani et al. observed that Ni nanoparticles display the maximum hydrogen adsorption capacity of 1.54 wt % at room temperature and atmospheric pressure. It was concluded that hydrogen molecules dissociate on metal particles and hydrogen is stored between the walled of nanotubes due to diffusion [25]. However, because of the inconsistency of the level of hydrogen storage in these studies, we were eager to work on this field.

In this present study, multi-walled carbon nanotubes were coated with nickel nanoparticles to investigate hydrogen storage capacity under different pressures. Wet chemical

oxidation process was used for surface functionalization of the MWCNTs, then Ni nanoparticles were impregnated to the nanotubes by wet chemical technique. The scanning electron microscopy (SEM), transition electron microscopy (TEM), Energy dispersive X-Ray (EDX) analysis and X-ray powder diffraction (XRD) have been used to investigate the morphology of the synthesized Ni-MWCNTs. The hydrogenation of the Ni-MWCNTs was performed by Sieverts-like apparatus at room temperature and moderate hydrogen pressures of 4–12 bar.

Experimental

Materials

MWCNTs of 5–8 nm in width and less than 6 mm in length (98% purity) were purchased from Nanografi. N, N-Dimethylformamide (DMF) (99% purity), sulphuric acid (H₂SO₄) (99% purity), and nitric acid (HNO₃) (65% purity) were purchased from Sigma Aldrich.

Purification and functionalization of MWCNTs

Functionalization of carbon nanotubes generally is done by using oxidation agents i.e. H₂SO₄, HNO₃, H₂O₂, K₂Cr₂O₇, KMnO₄, H₂SO₄-K₂Cr₂O₇ and KMnO₄-H₂SO₄. The surface of MWCNTs was functionalized by carboxylic (–COOH), carbonyl (–C=O), and hydroxyl (–OH) groups for improving dispersability of hydrogen on the MWCNTs [26–30]. In this present study, acid treatment of MWCNTs was prepared with 96 wt % sulphuric acid (H₂SO₄) and 65 wt % nitric acid (HNO₃). The raw MWCNTs were first oven dried and calcined at 450 °C for 1 h in O₂ at ambient pressure. When the MWCNTs cooled down to room temperature, 0.4 g of the MWCNTs suspended in 45 ml solution of sulphuric acid 96 wt % and nitric acid 65 wt % in ratio 70:30 in a 100-ml bottom round flask and it was refluxed with magnetic stirrer for 6 h. The solution was then filtered, and distilled water was used to rinse the solution from the nanotubes until the pH was neutral. The nanotubes were finally dried for one day in an oven at 150 °C.

Preparation of Ni-doped MWCNTs

150 mg MWCNTs were placed in a solution of 1 M of Ni(NO₃)₂·6H₂O and subjected to ultrasonication to prepare Ni-MWCNTs nanocomposites. The nanotubes were treated with Ni nanoparticles by an ultrasonicator for 1 h. Subsequently, the mixture was dispersed by using DMF using ultrasonicator for 3 h. Thereafter, the nanotubes were kept in an oven at a temperature of 200 °C for 2 h to remove any impurities.

Sample characterization

The Zeiss Ultra Plus field emission gun scanning electron microscopy (FESEM), FEI 200S transition electron microscopy (TEM), Rigaku Ultima IV X-ray diffraction (XRD) using Cu K α radiation have been used to investigate the morphology of the synthesized Ni-MWCNTs. Ni-MWCNTs elemental

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