Research Article

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Synthesis, characterization, and electrochemical properties of carbon nanotubes used as cathode materials for Al–air batteries from a renewable source of water hyacinth

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Abstract: Water hyacinth (WH) is a noxious weed. Its rapid growth can clog waterways, causing widespread ecological and environmental threats as well as power generation and irrigation issues. In the present study, water hyacinth was utilized as a precursor to synthesize multiwall carbon nanotubes (MWCNTs) using a simple chemical vapor deposition method. FESEM-EDS revealed that the synthesized MWCNTs had noodle-like, dense, and rough surfaces. TEM confirmed that MWCNTs had a bamboo-like structure with the diameter of 30–40 nm, and the wall thickness of approximately 7 nm. The interlayer distance was found to be approximately 0.34 nm by XRD. Raman spectra displayed three major bands of MWCNTs: D-band at $1,340\text{ cm}^{-1}$, G-band at $1,596\text{ cm}^{-1}$, and (D + G)-band between 2,783 and $2,953\text{ cm}^{-1}$. The ratio of the D-to-G band intensity was 0.94 ± 0.03 , indicating that the synthesized MWCNTs had well degree of graphitization. Electrochemical measurement results of the prepared MWCNTs cathode for aluminum–air battery showed that MWCNTs exhibited higher energy capacity than commercial graphite. Collectively, this study shows that water hyacinth could be used effectively as a precursor for the production of MWCNTs, thus successfully converting the noxious weed into high value-added materials.

Keywords: carbon nanotubes, water hyacinth, aluminum–air battery

1 Introduction

Since it was discovered by Iijima in 1991 [1], carbon nanotubes (CNTs) have shown remarkable properties and have been widely applied in many research fields to produce a variety of products such as electronic devices, field emitters, hydrogen storages, composite materials, biosensors, fuel cells, capacitors, and asphalt reinforcement [2]. Traditional methods for CNT synthesis are arc discharge, laser ablation, and chemical vapor deposition (CVD). Compared to arc discharge and laser ablation, CVD is more attractive because it is a relatively low-cost and less hazardous chemical synthesis technique that requires a low synthesis temperature, allowing the use of renewable feedstocks and enabling an easy industrial scale-up. In contrast, arc discharge and laser ablation methods allow less control over the processing parameters, consume higher energy, and give a lower percent yield of the product [3].

In CVD, a carbon source gas, such as ethylene, methane, or acetylene, is cracked into atoms and decomposed over a catalyst on a support, which is placed in the middle of the furnace under an optimal reactive temperature and gas environment (e.g., nitrogen, hydrogen, argon) [4]. Several parameters including the types of the carbon sources, catalyst, support, the temperature, and the reaction time are controlled in the CVD process to synthesize CNTs [5]. Mainly, a transition metal (e.g., Fe, Ni, Co) is used in the CVD process as a catalyst [6]. As a precursor, several inorganic materials, for example, quartz, silica, alumina, silicon, silicon carbide, and zeolite, are commonly employed. However, these precursors are expensive and unsustainable [7]. Based on the rapid global demand of CNTs for various applications, a new approach for green sustainable and eco-friendly production must be considered [8].

Biomass, an organic material derived from plants and animals, is an abundant resource that is also cost-effective, renewable, and eco-friendly [9]. Thus, it has been extensively utilized to produce bio-fuel, biogas, charcoal, and biochar. Biochar is a carbon-rich product, made by pyrolysis of biomass [10,11]. Several recent studies have reported the simplification of CNT synthesis using biochar.

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Fathy [12] reported the CNT synthesis through carbonization of hydrothermally treated rice straw using either a single catalyst of ferrocene or mixed catalysts of ferrocene and nickel nitrate. The carbon source was derived from heating camphor under flowing nitrogen gas at 800°C. They proposed that the presence of nickel nitrate with ferrocene can enhance the yield, thickness, and graphitization of CNTs. Although biomass has been widely utilized to produce CNTs, noxious aquatic weeds have not yet been employed for CNTs synthesis [13].

Water hyacinth (*Eichhornia crassipes*) is an abundant noxious weed and has fast, uncontrolled, and crowded growth. Its high growth density of around 60 kg/m² can lead to serious problems in irrigation, navigation, and power generation and the depletion of nutrients and oxygen from water, deteriorating an ecosystem of aquatic plants and fish [14,15]. Sindhu et al. [16] suggested that water hyacinth would be a suitable candidate for bioconversion to create various value-added products. Marimuthu and Atmakuru [13] obtained cellulose nanofibers from water hyacinth by chemical treatments, including bleaching, alkaline, and sodium chlorite reactions. The liquid nitrogen was released to cryocrush the micron-sized fibers from stem. The diameter of the produced nanofibers was 20–100 nm. Finally, Soenjiya et al. [17] prepared carbon fibers from liquid tar of water hyacinth, which was a resin that composed of high phenolic compounds. Water hyacinth resin was carbonized at 900°C, and the yield of carbon fiber obtained was 29%, which was relatively low. Obviously, water hyacinth is an abundant renewable biomass and carbon source with low cost. It is also an absorbent of heavy metals (e.g., Fe), which was used as a promising catalyst for the formation of CNTs [7,12]. Although the carbon fibers were successfully prepared from the liquid tar of water hyacinth, the synthesis of CNTs from this material has not yet been reported.

Therefore, this study explores the production process of CNT synthesis from water hyacinth as a precursor at 650°C by the CVD, which is simple and convenient for mass production. Moreover, the potential application of the as-prepared CNTs was demonstrated for the aluminum–air battery. Surface morphology and microstructure of the synthesized CNTs were characterized via field emission scanning electron microscopy (FESEM) and transmission electron microscope (TEM). Also, the degree of graphitization and the D-spicing value were determined by Raman spectroscopy and X-ray diffraction (XRD). Besides, the electrochemical property of CNTs used as cathode materials for aluminum–air battery was examined by cathodic polarization and galvanostatic technique.

2 Materials and methods

2.1 Materials and preparation of water hyacinth

Water hyacinth (WH) was obtained from a pond at Khon Kaen University, Khon Kaen, Thailand. Ferrocene 98% (C₁₀H₁₀Fe) was purchased from Sigma Aldrich. The fresh water hyacinth was first cleaned by tap water to remove the mud and grime. Then, it was washed with deionized water and dried in a hot air oven for 48 h at 105°C. The dried water hyacinth was milled and sieved into smaller sizes in the range of 2–2.36 mm. Finally, it was introduced into a furnace for carbonization at 450°C for 1 h under a nitrogen atmosphere represented as biochar of water hyacinth (denote as BWH) for increasing the porosity and the surface area [17]. The BWH was mixed with acetone as a solvent and ferrocene as a catalyst. Incipient wetness impregnation method was utilized for catalyst coating. First, the BWH and 2 wt% ferrocene was mixed together in a 30 mL well-suspended acetone solution. Second, sonication of the mixture was conducted in the ultrasonic bath at 60°C until the solvent was evaporated completely. Then, the dried cake denoted as WH/Fe was obtained.

2.2 Synthesis of CNTs

Chemical vapor deposition (CVD) was performed to synthesize CNTs. The CVD reactor consisted of a horizontal ceramic tube (diameter of 4.5 cm and length of 62 cm) located in an electric furnace. WH/Fe was left on a ceramic boat, placed in the middle of the furnace, and sealed. Nitrogen gas (N₂) was supplied in the CVD reactor with a flow rate of 200 mL/min for 30 min to prevent the oxidation of the catalytic metal and to remove the atmospheric air inside the ceramic tube. Following nitrogen purging, a heater was switched on to raise the temperature to 350°C. The temperature was maintained at 350°C for 1 h to eliminate moisture. Afterward, hydrogen gas (H₂) was released into the reactor with a flow rate of 100 mL/min, while the temperature was raised to 650°C and maintained at this temperature for 30 min. After achieving the target temperature and time, acetylene gas (C₂H₂) as a carbon source was passed through the tube for CNT synthesis, with a flow rate of 50 mL/min for 20 min. Then, acetylene gas was stopped, and the furnace was cooled down to room temperature. Hydrogen and nitrogen supplies were turned off when the temperature was lowered down to 500 and 200°C, respectively.

2.3 Characterization of CNTs

The synthesized CNTs were characterized by transmission electron microscopy (TEM) in a FEI TECNAI G2 20 electron microscope to study their morphology, crystallization, and size. Field emission scanning electron microscopy (FESEM) was used to assess the surface structure of the CNTs. The configuration of chemical elements was measured by energy-dispersive X-ray spectrometer (EDS) attached to the FESEM. The X-ray diffraction (XRD) patterns were recorded on a Rigaku Dmax-rc diffractometer using Ni-filtered CuK radiation ($V = 50$ kV, $I = 80$ mA). The degrees of defect and purity of the synthesized CNTs were evaluated by Raman spectroscopy, utilizing a Horiba Xplora confocal Raman microscope spectrometer (Horiba Jobin Yvon, Northampton, UK) with an excitation laser of 532 nm and an acquisition time of 10 s. The purification and the thermal stability of the CNTs were examined by the thermal gravimetric analysis (TGA). After CNTs were loaded into a TGA chamber, they were heated from room temperature to 110°C in a N_2 atmosphere with a heating rate of 10°C min⁻¹ and were maintained at this temperature for 2 min. Then, the N_2 atmosphere was changed to air, and the temperature was increased to 850°C with a heating rate of 10°C min⁻¹ and maintained for 1 min. Finally, the TGA chamber was cooled down to room temperature under the air atmosphere.

2.4 Electrochemical measurements

The potentiostat (Gamry Reference 600) was used to investigate the electrochemical performance of CNTs in 4 M NaCl solution. The electrochemical cell, a three-electrode system composed of platinum (Pt) as a counter electrode, standard Ag/AgCl electrode (SSE) as a reference electrode, and the CNT coated on nickel foam as a working electrode, was used in this investigation. CNT-coated nickel foam of 1 cm² was prepared by pressing CNT powder on nickel foam, which was not peeled off in NaCl solution throughout the experiment. Cathodic polarization curves were plotted at a scan rate of 5 mV/s. CNT-coated nickel foam was applied as the cathode and Al foil was used as the anode, and galvanostatic technique was used to investigate the performance of aluminum–air battery as a cathode in 4 M NaCl electrolyte at room temperature and a constant current of 0.5 mA/cm². The electrochemical performance of CNTs was compared with the commercial graphite cathode electrode. In addition, all tests were repeated for at least three times to obtain the average values.

3 Results and discussion

3.1 Morphology characterization

Figure 1a shows FESEM images of the rough surface with the porosity of WH/Fe before the CVD process, which was biochar derived from water hyacinth carbonization. Figure 1c and d displays FESEM images of as-growth CNTs after the CVD process at 650°C. Figure 1c shows the overall surface morphology of the as-growth CNTs, which appeared dense, entangled, and noodle like. As shown in Figure 1d, the CNTs had a rough tube surface. The open ends tubes were observed in some of the CNTs, suggesting that the growth mechanism of the CNTs was a tip growing model [18]. The result indicates that the BWH served as an effective pore and active site for anchor catalyst particles for synthesizing CNTs. Table 1 presents the element composition of WH, BWH, WH/Fe, and MWCNTs before and after the CVD process. As shown earlier, carbon is the main content of WH, and it increased nearly 59.2% after the CVD process due to the formation of CNTs. The Fe content of BWH had 0.1 wt% after impregnation of ferrocene catalyst, increasing to 1.8 wt%, which indicated that the BWH has slightly high porosity and surface area to support catalyst. The oxygen content was decreased to approximately 59.1%. Conversely, the Fe content after the CVD process decreased to approximately 0.9 wt%, indicating that the Fe played as a nucleation role for the formation of CNTs. The synthesized CNTs contained a high amount of carbon (85.2 wt%) with some other elements, including O (13.9 wt%) and Fe (0.9 wt%), which corresponded with the results reported by Zhu *et al.*, Araga and Sharma, and Liao *et al.* [19–21]. Furthermore, the EDS mapping (Figure 1b) shows that the Fe catalyst was well distributed over the biochar surface.

TEM, HRTEM, and selected area diffraction (SAED) were used for characterization of the morphological structures of the synthesized CNTs. As shown in the TEM image of MWCNTs in Figure 2a, the as-growth CNTs bundles appeared crooked and tangled, having the average diameter and length in the range of 30–40 nm and several micrometers, respectively. In addition, an open tube was seen at the tip of the CNTs (as shown in the dotted white square in Figure 2a), being consistent with the FESEM observation. CNTs grown with the ferrocene catalyst had multiwall carbon nanotube structure (MWCNTs), consisting of graphene layers of concentric nanostructure as shown in Figure 2b and c, consistent with the structures reported by Wang *et al.* [22–26]. The wall thickness was approximately 7 nm. Using of ferrocene as a catalyst in the CVD process led to the formation of MWCNTs, agreeing with previous

Figure 1: FESEM images of different magnifications: (a) WH/Fe surface before CVD process, (b) the EDS mapping showing the consistency distribution of the Fe element, (c) low magnification of dense and entangled as-growth CNTs after CVD process at 650°C, and (d) high magnification of the surface structure of as-growth CNTs.

Table 1: Element composition of WH, BWH, and WH/Fe before CVD and MWCNTs before and after the CVD process

Elemental composition (wt%)	C	O	Fe
WH	26	73	0.1
BWH	64.3	35.5	0.1
WH/Fe*	64.4	33.8	1.8
MWCNTs	85.2	13.9	0.9

* WH/Fe obtained from Section 2.2.

studies by Khan et al. and Li et al. also proposed that ferrocene act as a catalyst can largely formation of MWCNTs [7,21,27]. Figure 2c demonstrates that the carbon nanotubes possessed a bamboo-like structure, as shown by the wrinkles, the compartments, and the curvatures of the tube walls. Basically, the growth mechanism of the CNTs synthesized by the CVD method consists of the following three steps: (i) the decomposition of the carbon source (i.e., acetylene) into free carbon atoms, (ii) the diffusion of the free carbon atoms to the surface of catalyst particles, and

(iii) the precipitation of the carbon atoms over the catalyst particles resulting in the CNT synthesis [28]. The initial state free carbon atom dissolves into the surface of the Fe catalyst until it is supersaturated in the catalyst particle. The carbon atom starts to precipitate on the Fe catalyst particle and increases the dissolution of carbon atom over time, resulting in the formation of a C–C bond with close atoms. Furthermore, the hexagonal carbon was formed over the Fe catalysts particles like a graphitic cap and provided the nucleation site of other carbon precipitation. Finally, the interaction between the carbon atom and the surface of the Fe catalyst is weak due to the stress of kinetic energy, leading to the graphitic cap lift off from the catalyst. Continuing of these graphitic caps lift off to procedure not only the compartment layer but also a form of the multiwall of carbon nanotubes. Formation of the CNTs was due to the differences between the temperature gradients of the top and bottom surfaces of the catalyst particles as well as the concentration gradient of the fluid flow. The CNT structure depends on the size and the shape of the catalyst. Small catalyst particles are formed as hollow

Figure 2: (a) TEM image of MWCNTs, (b and c) HRTEM images of the concentric multiwall CNTs structure and the bamboo-like structure, and (d) SAED pattern of MWCNTs.

structures, whereas large catalyst particles lead to the growth of a bamboo-like structure. In this case, the bamboo-like structure of MWCNTs was formed as shown in Figure 2c, which could be explained by the following steps. After the acetylene decomposition to carbon atom process, it diffused into the surface of the catalyst metal, producing graphitic sheets as a cap on the catalyst particles. The dense graphitic sheets created driving force, which pushed the cap lifts off the catalytic particle along the growth direction owing to the stress accumulation of graphitic sheets. It can form the compartment, leading to create the wrinkles as a curvature compartment (see Figure 2c), and a closed tip with inside hollow is produced. This phenomenon occurred repeatedly, leading to the formation of the bamboo-like structure. Similar explanation was reported by Jia *et al.* [29]. The arrangement crystallinity of the CNTs was confirmed by selected area electron diffraction pattern (SAED) (Figure 2d). As shown, the plane (002) is indicated in the SAED pattern of obtained CNTs, which is consistent with the XRD peak at $2\theta = 26.44^\circ$ plane

(002; Figure 4). In this pattern, the bright spot and broad continuous rings could be attributed to small crystals.

Moreover, the interlayer distance as shown in Figure 2c was approximately 0.34 nm, which was similar to the interlayer distanced calculated from the XRD result (Figure 4). From these results, it could be suggested that the obtained CNTs were graphitic in nature and well degree of reorganization of crystallinity.

3.2 Structure analysis

Raman spectroscopy, a powerful tool for characterization of CNTs, has been widely used to investigate the order of crystallites, the defect of the graphene sheet, and the vibration of sp^2 -hybridized carbon atoms [30]. Raman spectra of MWCNTs are illustrated in Figure 3. Two sharp peaks were observed around $1,340\text{ cm}^{-1}$ (D band) and $1,590\text{ cm}^{-1}$ (G band). The D band is a characteristic band representing the defect in the graphene sheets, while the

Figure 3: Raman spectra of MWCNTs.

G band is one of the vibrations of the sp^2 -hybridized carbon atoms in the graphene sheets [31,32]. The intensity ratio of the D to G bands (I_D/I_G) was used to assess the crystalline order of a carbon material; the ratio of less than 1 ($I_D < I_G$) indicates well crystalline CNTs. For the CNTs synthesized in this study, the D-band and G-band intensity ratio was found to be 0.94 ± 0.03 , indicating well degree of graphitization. Besides there was a broad beak between 2,783 and 2,953 cm^{-1} , which represents an overtone of the D and G bands, denoted as (D + G) band.

It indicates the presence of the long-range order of crystalline structure in MWCNTs, corresponding with a report by Araga and Sharma [33]. Figure 4 shows the XRD pattern of the MWCNTs synthesized by the CVD method. The strongest peak observed at $2\theta = 26.44^\circ$ represented the plane (002) of the hexagonal graphite carbon of CNTs, agreeing with the previously published results by Chai and coworkers, Cui and coworkers, and Qu et al. [32,34,35]. This extremely high diffraction peak at $2\theta = 26.44^\circ$ suggested a high amount of the graphitic carbon. The other peaks around $2\theta = 38.66^\circ$, 48.30° , and 56.87° could be attributed to Fe_3C , Fe and $FeCO_3$ were abundant at peak $2\theta = 64.71^\circ$ and 71.94° , respectively, due to the ferrocene catalyst. The interlayer distance was obtained using Bragg's law (Eq. 1):

$$n\lambda = 2d \sin \theta \quad (1)$$

where n is a positive integer (1), λ is wavelength of the incident X-ray, and θ is the position of the peak. The interlayer distance between the (002) plane was approximately 0.34 nm for CNTs, which is close to the interlayer

distance of pure graphite around 0.3354 nm, corresponding well with the inter layer distance value from the TEM and SAED pattern images, and the plane (002) of CNTs is also represented in SAED pattern (Figure 2d).

The purity and the thermal stability of the synthesized CNTs were assessed by using the thermogravimetric analysis (TGA). Figure 5 shows a TGA plot of the weight loss of the MWCNTs, WH/Fe, and BWH versus temperature. As shown, all of these were degraded at the temperature of 50–150°C due to the moisture evaporation, resulting in weight loss of BWH and WH/Fe about 10%. However, the weight loss of MWCNTs was about 4%. For BWH, there was three main stage thermal degradations: 150–480, 480–650, and 650–950°C. At these stages, the weight losses were discovered to be 35%, 9%, and 39%, respectively. It could be attributed to the degradation of hemicelluloses, cellulose, and lignin, which remain in BWH that are the main composition in biomass. As the residue of BWH was roughly 8%. Likewise, for WH/Fe, there were also three stages of thermal degradations, but in the last stage, the weight losses were around 16%. The residue of WH/Fe was approximately 32%, which is higher than that of the last stage of BWH. It indicated that the residue of WH/Fe was attributed to the Fe catalysts.

Conversely, the TGA curve of MWCNTs differed compared to that of BWH and WH/Fe. There were three regions of the weight loss observed in MWCNTs. At the initial region, a slight drop in the weight loss at the temperature between 50 and 150°C, which was due to the loss of moisture by approximately 5%. The second region at the temperature around 150–550°C represented the weight loss of about 27%,

Figure 4: The XRD pattern of MWCNTs.

which was caused by the decomposition of light amorphous [35]. The last region at the temperature above 500°C was due to the destruction of the carbon structure, causing the weight loss of about 36%, which was due to the carbon oxidation of MWCNTs [36]. This finding suggests that the yield of obtained MWCNTs was approximately 36%, which is higher than the previous study by Soenjaya *et al.* [17]. Therefore, the MWCNTs have higher thermal stability than BWH and WH/Fe because they have a higher content of graphitic carbon and the well-organized crystallinity.

3.3 Electrochemical behavior

To evaluate the quality of obtained MWCNTs, which were used as cathode material for the aluminum–air

battery. Electrochemical and performance of MWCNTs were investigated by cathodic polarization techniques and galvanostatic techniques. The cathodic polarization responses of MWCNTs coated on nickel foam and uncoated nickel foam as well as commercial graphite cathodes were shown in Figure 6a. For MWCNTs, a sharp increase in the current density with the driving force of approximately 0.1 V was observed. It should be attributed to the rate of high oxygen reduction reaction and hydrogen evolution reaction on the MWCNTs surface. Then, the increase in the rate of the current density slowed down. Finally, the current density was constant even though the driving force potential was increased, which can be attributed to the formation of sodium carbonate in the MWCNTs layers, which obstructed reaction at the electrode surface. The cathodic behavior of the MWCNTs and commercial graphite was quite similar. The cathodic polarization responses of MWCNTs showed higher activity comparing to that of commercial graphite cathodes. At the potential of –0.6 V that was low over potential to drive high current density, the current density difference (~29 mA) between MWCNTs and commercial graphite cathodes was indicated in Figure 6a. This confirmed that the increase of the cathodic reaction behavior was due to the coating of MWCNTs on nickel foam substrates. Galvanostatic (as shown in Figure 6b) of MWCNTs as a cathode at the consistent current of 0.5 mA/cm² test revealed that the electric discharge was approximately 50 h, and the voltage was consistent in all experiments, that is, nearly 0.6438 V. Form these results, the energy density and the

Figure 5: The TGA curve of the percent weight loss versus temperature of MWCNTs, WH/Fe, and BWH.

Figure 6: (a) Cathodic polarization behavior of MWCNTs coated on nickel foam, uncoated nickel foam, and commercial graphite and (b) the galvanostatic of an Al-air battery, and MWCNTs were used as a cathode at a consistent current of 0.5 mA/cm^2 .

energy capacity of MWCNTs coated on nickel were 1363.63 Wh/kg and 2272.72 mAh/g , respectively, which results higher than that in the previous work by Supachai and Mathuros [37]. It could be concluded that the obtained MWCNTs have a potential to an application as an Al-air battery.

4 Conclusions

We reported for the first time the successful synthesis of MWCNTs from water hyacinth as a precursor, using a low amount of ferrocene catalyst through the chemical vapor deposition method. Acetylene was used as a carbon source. This method provided yield of MWCNTs approximately 36%, as verified by TGA. SEM images showed abundance of as-growth MWCNTs with dense and entangle structures, corresponding with the HRTEM results. The carbon nanotubes had multiwall types and the bamboo-like structure with the interlayer distance of approximately 0.34 nm , as determined by both the HRTEM and XRD results. The strongest peak of XRD at $2\theta = 26.44^\circ$ represented the graphitic carbon. Moreover, Raman spectroscopy revealed well degree of graphitization with the I_D/I_G ratio of 0.94 ± 0.03 . To test the quality of prepared MWCNTs, we studied the application of MWCNTs for energy storage material by using MWCNTs as a cathode material for aluminum-air battery. The results showed that MWCNTs cathode exhibited higher current density and energy capacity. This study demonstrated the successful conversion of a threat weed into value-added nanomaterial products and provided an

effective way of the easy process, promoting the green eco-friendly and sustainable environment.

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