Exp. Theo. NANOTECHNOLOGY 5 (2021) 33 – 38 https://doi.org/10.56053/5.3.33

Characterization of carbon nanotubes by chemical vapor deposition

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Received 4/2/2021, *Accepted* 15/6/2021, *Published* 15/9/2021

This study investigates novel multiwalled carbon nanotubes (MWCNTs) grown on agricultural waste, using loaded iron nanoparticles as catalyst templates and acetylene as carbon source through chemical vapor deposition under specific conditions, to wit: 550 °C reaction temperature, 47 min reaction time, and 1 gas ratio. The specifications of MWCNTs are analyzed and characterized with the use of field-emission scanning electron microscopy and energy-dispersive X-ray spectroscopy. The results reveal that MWCNTs have high quality and unique morphologies.

Keywords: CNTs; CVD; RSM.

1. INTRODUCTION

Relentless industrialization and infrastructure development has led to progressive environmental degeneration. Pollutants, including industrial waste, agricultural run-off and wastewater discharge, sewage, are primary contributors to environmental contamination and contain deadly substances, such as metallurgical toxins, and other organic and inorganic poisons. Specifically, metal-based pollution is generated as a consequence of industrial effluence being released into the environment via water sources. The resultant damage threatens mot merely human welfare and existence, but all life on Earth. Aluminum is particularly reactive to both oxygen and carbon. Moreover, it has the potential to cause significant damage to human health and has been implicated in the development of autistic spectrum disorders, Alzheimer's disease, and neurotoxicity of the central nervous system [1]. According to the Environmental Protection Agency, the concentration of aluminum in potable water must not be permitted to pass 0.05 to 0.20 mg/L [2]. This suggests that it is necessary to eliminate aluminum from waste prior to any discharge which might reach natural water sources.

There are multiple techniques which are currently utilized for the removal of metals from wastewater, including chemical precipitation with hydroxides, chelating precipitation with sulfides, ion exchange, bioadsorbents, adsorption using porous carbon materials, electrodialysis, coagulation and flocculation, membrane filtration, flotation, and electrochemical treatment [3]. The technique of choice is currently

deemed to be adsorption using porous carbon materials due to its relative inexpensiveness, its applicability for large-scale use, the ease with which it can be employed, and its inherent adaptability. Typically, adsorbents use comprised of elements such as sawdust [4], rice husks [5], maize cobs [6], granulated blast furnace slag [7], kaolinitic and giru clays [8], aquatic plants [9], powdered activated carbon (PAC) [10], and nanomaterials [11].

The use of nanomaterials has been widely investigated due to the excellent mechanical performance of these materials. Carbon nanotubes (CNTs) have been widely used and evaluated in water treatment as a consequence of their unique structure, small size, catalytic potential, high reactivity, easy separation, and large surface area, all of which render them an effective adsorbent for many metals. CNTs have been developed using several methods, including chemical vapor deposition (CVD), laser ablation, and arc discharge. CVD is superior thanks to its cost effectiveness and ability to produce high-purity materials under controlled growth conditions.

PAC is considered the ideal substrate for CVD because of its wide precursor accessibility, low-cost, and chemical alteration potentials. Selective growth is comparatively easy to achieve through synthesis using prepatterned catalyst nanoparticles, such as iron, nickel, molybdenum, aluminum, cobalt, and zirconium. Research by Xiang et al. [12] and Mustafa et al. [13] resulted in the successful synthesis of CNTs on PAC via CVD through the use of acetylene as a carbon source with several catalysts produced from iron, cobalt, aluminum, and nickel. Moreover, Haiyam et al. synthesized CNMs on commercial PAC loaded with nickel nanoparticles by using methane as a carbon source [14] and Arash et al. synthesized CNTs via CVD on silicon sheets loaded with iron by utilizing methane as a carbon source [15]. Research by Zaho et al. has demonstrated that high yields of CNTs can be synthesized in water through CVD by using iron and molybdenum catalysts supported on a magnesium oxide substrate [16]. An inverse correlation between catalyst lifetime and CNT growth rate was observed by Chen et al. [17]. Furthermore, Mamtm et al. have reported that PAC can function as a useful precursor for CNT growth [18]. Interestingly, when compared to other substrates, PAC does not need to be chemically or physically removed from the functional bulk material. Catalyst iron nanoparticles with acetylene as carbon source can provide CNTs with high quality, increased density, and enhanced purity. Having reviewed the relevant literature in this field, the researcher intends that the current study will offer insights into the growth of unique multi-walled carbon nanotubes (MWCNTs).

This research investigates the use of synthesized MWCNTs on PAC as an agrarian effluence precursor when loaded with iron nanoparticles using CVD. Several techniques are adopted in order to identify and evaluate the structural and morphological properties of MWCNTs, to wit: energy-dispersive X-ray spectroscopy (EDX) and field-emission scanning electron microscopy (FESEM). The results indicate that it is possible to divorce MWCNTs from water with comparative ease due to their hydrophobicity. This suggests that this procedure might comprise a scalable process with applicability to industrial waste management, both due to its ease of operation and its superior removal performance. Moreover, MWCNTs are also a relatively inexpensive adsorbent.

2. EXPERIMENTAL

Iron (III) nitrate nonohydrate Fe $(NO_3)_3.9H_2O$ and acetone are purchased from Friendemann Schemicit, Malaysia. Aluminum standard solution Al $(NO_3)_3$. $9H_2O$, hydrolytic acid (HCl), sodium hydroxide (NaOH) is obtained from Merck (Malaysia). C_2H_2, H_2 and N_2 are used for MWCNTs growth are purchased from Alpha gas solution (AGS), Malaysia. The agricultural wastes powder activated carbon (PAC) which has been fabricated by our group is utilized to synthesis MWCNTs [19]. Fe was utilized as a catalyst and added in 5 ml acetone, then mixed with (2g) PAC. However, mixture is sonicated at 60 ℃ for 99 min till acetone evaporated. Subsequently sample bio-PAC/ Fe is dried at 105 ℃ for 24 h. Calcinated PAC/Fe at 400 ℃ at 2 h under inert gas (purified N_2, 200ml/min) [20].

CNTs growth is carried out by placing (300 mg) PAC/Fe in ceramic boat with in CVD reaction tube. A typical growth accomplished by reduction under H_2 at 550 ℃ with flow (160 ml/min). Thereafter, C_2H_2 is used as a carbon source and mixed with H_2 at 1:4 ratio. The reaction is passed through heated reactor at 47 min. After MWCNTs growth sample is slowly cooling under purified N_2 flow rate (200ml/min) to room temperature, the MWCNTs sample is collected after the completion of the reaction. Surface morphology for MWCNTs is characterized by field emission scanning electron microscope (FESEM) with energy-dispersive X-ray spectroscopy (EDX), model ZEISS (Merlin, UK).

3. RESULTS AND DISCUSSION

The morphologies and structures of synthesized MWCNTs are determined through FESEM prior to and after fabrication. Fig. 1(a) and (b) demonstrate that FESEM images with different magnifications can be observed before growth (AC/Fe). The substrate surface is rough, and the catalyst is dispersed. Fig. 1 (c) and (d) reveal that there is a carpet-like deposit containing highly dense MWCNT arrays without evident catalyst impurities following growth. The diameter of the nanotubes ranged from 34.18 nm to 37.45 nm. TEM images confirmed that MWCNTs possess successful structure formation and high quality.

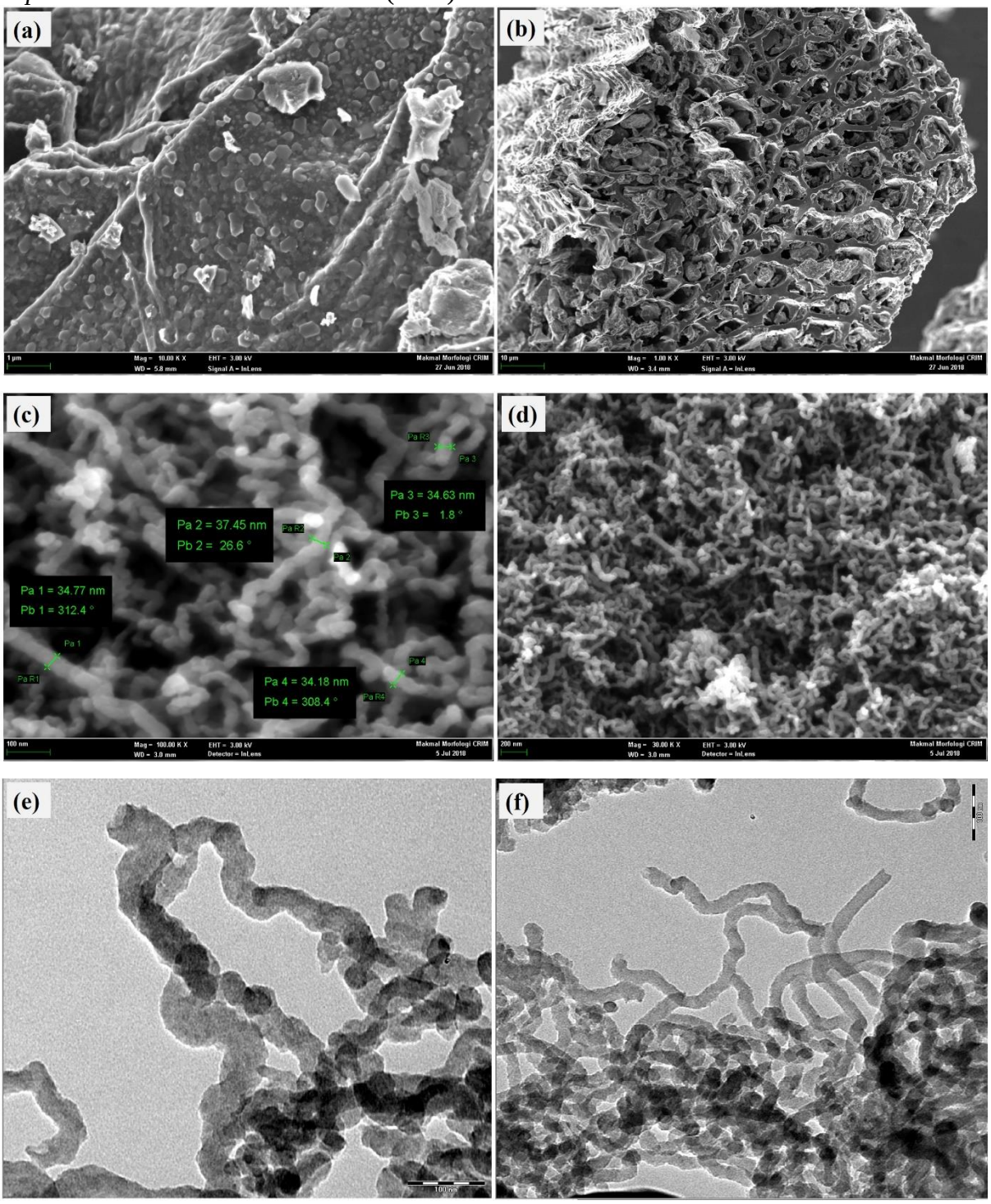

Figure 1 FESEM images (a and b) before growth, (c and d) MWCNTs and TEM images (e and f) of MWCNTs.

EDX analysis determined the substrate elements prior to and following growth. Fig. 2 (a) shows that the EDX spectra has indicated that the catalyst is successfully embedded on the AC/Fe surface. The surface of MWCNTs contained 97.8% carbon after growth with other impurities either decreasing or disappearing, as illustrated in Fig. 2 (b).

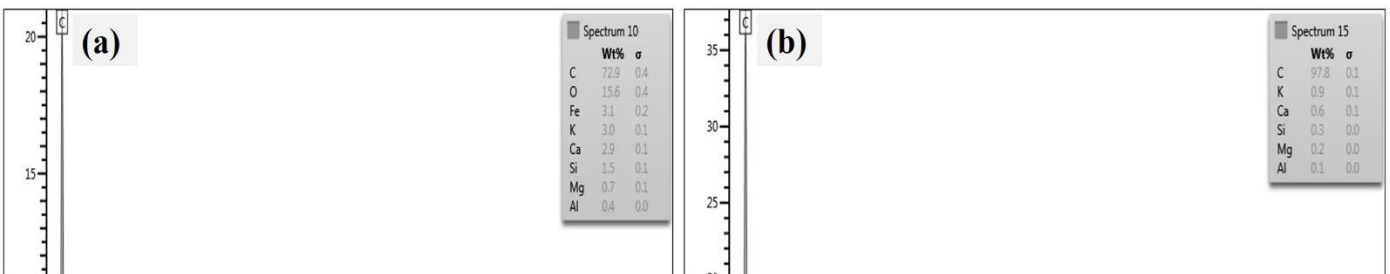

Fig .2. EDX spectra (a) AC/ Fe before growth and (b) after growth.

4. CONCLUSIONS

Figure 2 EDX spectra (a) AC/ Fe before growth and (b) after growth.

The objective of the current research has been to examine the synthesized MWCNTs grown on PAC impregnated with Fe catalyst (PAC/Fe). This is achieved through the use of acetylene as a carbon source under conditions of 550 °C for 47 min. The growth of synthesized MWCNTs was characterized via EDX and FESEM. The high-quality structure of MWCNTs can be attributed to the supported catalyst (Fe) on the PAC substrate. The MWCNTs on the PAC/Fe, which are synthesized from agricultural waste, can be used as a low-cost adsorbent and can be expanded in the treatment of other pollutants in wastewater and for several other purposes.

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