FUDMA Journal of Sciences (FJS)
ISSN online: 2616-1370
ISSN print: 2645 - 2944
Vol. 4 No. 3, September, 2020, pp 731 – 734
DOI: https://doi.org/10.33003/fjs-2020-0403-315

COMPARATIVE STUDY ON THE SYNTHESES OF CARBON NANOMATERIALS USING POLYETHYLENE AND RISK HUSK AS CARBON PRECURSOR.

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ABSTRACT

The study compares the syntheses of carbon nanostructures (CNS) using polyethylene (PE) and Rice husk (RH) as carbon precursor via commercial microwave oven at 2.45 GHz. The Microwave energy offers the requisite temperature for catalytic disintegration of the carbon precursors at 750 °C under atmospheric pressure. The CNS were grown on coated silicon dioxide. The as-synthesized CNS was analysed with Raman spectroscopy which shows carbon quality was found to be 0.92 and 1.01 in PE and RH respectively, indicating good graphitic nature with average diameter at (16.0 to 20.0) ± 0.5 nm. The high intensity ratio is attributed to the defect mode in the CNS. The Field Emission Scanning Microscope (FESEM) analysis shows a warped and randomly oriented structures with an interlayer spacing of about 0.35 nm in the internal structure of most CNS. Furthermore, the level of purity in the graphitic nature of the CNS were obtained with Thermogravimetric Analysis (TGA) technique with 90 % in PE and 50 % in RH. Hence, a fast and cheaper method of synthesizing CNS utilizing microwave energy was demonstrated at 750 °C under atmospheric pressure. Lastly, the presence of catalyst, carbon precursors and plasma are necessary for the microwave heating and synthesis process.

Keywords: Carbon nanostructures, Raman, Rice Husk, Polyethylene, Silicon Substrate.

INTRODUCTION

The emergence of nanotechnology has attracted much interest from scientific community in the area of carbon-based science due its exceptional carbon-carbon bonding assists carbon to form its allotropes, amongst such carbon nanostructures (CNS) are carbon nanofibers, graphites, graphenes, and carbon nanotubes (Kroto, 1991; O'Connell, 2006; Kure *et al*., 2015; Kure *et al*., 2017). Their exceptional properties such as electrical, thermal, chemical and mechanical properties attract interest from researchers (Kroto *et al*. 1991; Iijima, 1991). Extensive research has been carried out on how to synthesize this carbon-based nanomaterial. Among such methods are arc discharge (Iijima, 1991), laser ablation (Guo *et al*. 1995), chemical vapor deposition (Suriani *et al*. 2013) and Microwave-assisted chemical vapor deposition (Kure *et al*., 2017), with Carbon precursor playing a vital roles in the formation of CNS (Kure *et al*., 2015 and Kure *et al*., 2017). These techniques of synthesizing CNS are believed to be time consuming and expensive. In order to reduce cost and time wastage, an attempt was made to synthesize CNS using polyethylene (PE) and rice husk (RH) as carbon precursor via 600 W household microwave oven at 2.45 GHz (Kure *et al*., 2017 and Muhammad *et al.*, 2018). The PE and RH has dielectric loss tangent at 2.45 GHz, which makes them a good conducting polymer (Hotta *et al*. 2011; Kure *et al*., 2017). The synthesis utilizes microwave heating due to its advantages over the conventional methods such as rapid reaction time and volumetric heating which provides the temperature needed for catalytic decomposition of PE and RH (Bajpai *et al*., 2014; Kure *et al*., 2017 and Muhammad *et al.*, 2018).

MATERIALS AND METHODS

A tubular quartz tube was used as reaction chamber. The household microwave oven was adapted to introduce the tubular quartz duct across it. It was vacuum below 0.81 mbar of atmospheric pressure via rotary vacuum pump continually throughout the synthesis process. Polyethylene beads of 100 mg were used as carbon sources and silicon dioxide as substrate. Iron (III) nitrate was impregnated on the substrate which served as the trigger material for CNS growth. The catalyst and carbon source were sited within the reaction chamber. The temperature of the process was measured using thermocouple attach to Fluke multimeter to be 750 °C (Kure *et al*, 2017). This technique utilizes the advantage of microwave heating over conventional heating, to synthesize the CNS at much faster rate. The as-grown nanomaterial was allowed to reach ambient temperature before characterization. Raman spectroscopy was used to analyse the carbon quality using Witec alpha 300R with 532 nm laser excitation wavelength. Field emission scanning electron microscope (FESEM) images were produced via Joel JSM-7600F to know the structural morphology. A Mettler thermobalance TG-50 in a Mettler TA-4000 System with temperature ranging from 30 °C to 1000 °C with interval of 10 °C/min under oxygen gas with flow rate of 50 mL/min was used to carryout thermogravimetric analysis (TGA) in order to determine the level of purity in the CNS. The diameter distribution of the CNS was determine using Image J processing software.

Figure 1 depicts the experimental setup used for the derivation of carbon nanomaterials from the carbon precursors. The synthesizing pressure plays a vital in synthesis process, hence, the utilization of microwave energy. The PE pellets and RH

powders were irradiated for 4 minutes and 38 minutes respectively (Kure *et al*., 2017 and Muhammad *et al*., 2018). The temperature and pressure were maintained at 750 °C and 3×10^0 mbar respectively for the both carbon precursors (Kure *et al*., 2017 and Muhammad *et al*., 2018).

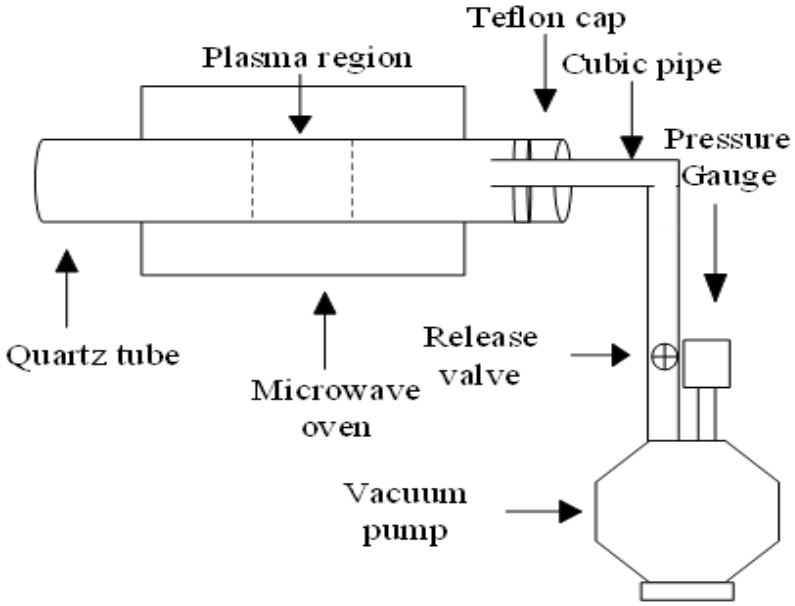

Figure 1. The experimental setup

RESULTS AND DISCUSSIONS

Results obtained are as depicted in Figure 2 (a) and (b) which shows the Raman spectrum of the carbon nanostructures from carbon precursors (PE and RH) with two protruding D and G peaks respectively. Figure 2 (a) shows two protruding peaks at 1336.77 cm^{-1} and 1588.50 cm^{-1} indicating defect (D) and graphite (G) mode respectively. The defect mode indicates the disordered carbon atom and graphite mode indicates the ordered carbon atoms on the CNS (Kure *et al*., 2017). The ratio of the defect intensity (ID) and graphite intensity (IG) is to determine the CNS carbon quality and it was calculated to be 0.97. The defect mode increases as a result of amorphous carbon indicating impurity (Kure *et al*., 2015). From Figure 2 (b), D peak was observed at 1335 cm^{-1}. The induced peak shows defective graphitic structures associated with sp^3 honeycomb carbon atom network. The G peak observed at 1601 cm^{-1} indicates the level of order in sp^2 honeycomb carbon atom network (Muhammad *et al*., 2018). The ratio of D band to G band (*I* D/*I* G) was used to estimate the degree of defects present on the carbon nanostructures. The ratio of (*I* D/*I* G) for the as-synthesized nanostructures was calculated to be 1.01 (Muhammad *et al*., 2018). The peaks intensities (*I* D/*I* G) shows lower quality of sp^2 carbon structures due to amorphous carbon and structural defects in carbon nanomaterials (Muhammad *et al*., 2018).

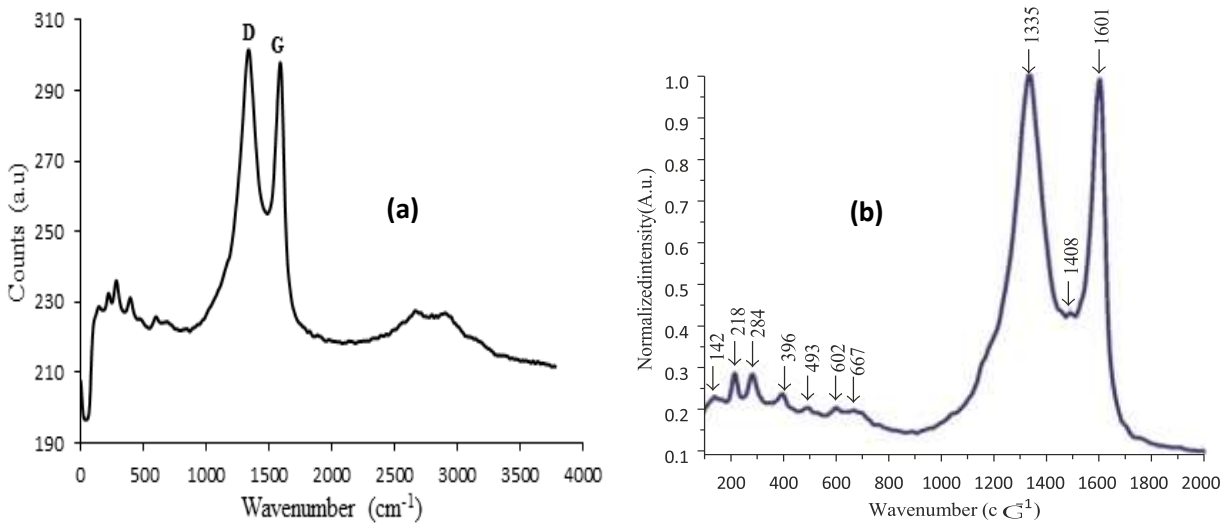

Figure 2(a) Raman spectrum of PE (b) Raman Spectrum of RH

Figure 3(a) and (b) shows the surface morphology of the derived CNS. These images from FESEM shows the formation of a twisted and flower-like CNS with several micrometers longdue to catalytic decomposition of the carbon precursors. The CNS size variations may be attributed to defect mode as depicted in Figure 3 with diameter range between (16.0 to 20.0) ±0.5 nm. From Figure 3, it shows a bright-tip color on the CNS indicating the presence of the catalyst remained (Kure *et al*., 2015; Kure *et al*., 2017 and Muhammad *et al*., 2018).

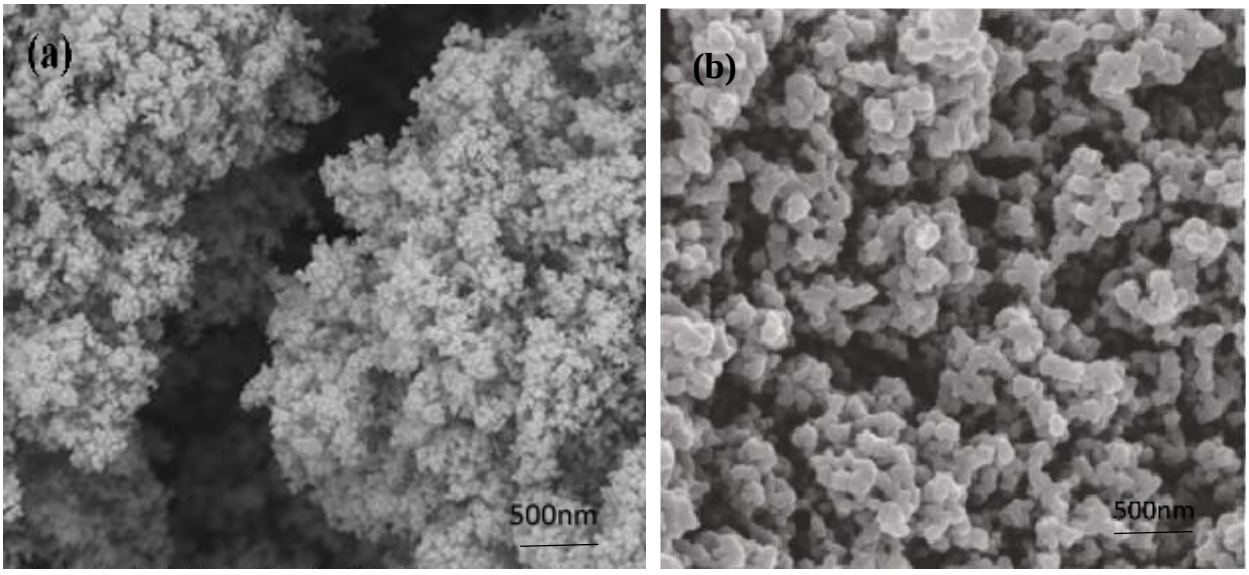

Figure 3 (a) FESEM image of PE CNS (b) FESEM image of RH CNS

The TGA results were shown in Figure 4 (a) and (b) below. From Figure 4 (a), it shows a quick oxidation of CNS from 540 °C to 640 °C with 90 % sample weight loss; the sharp peak observed from DTGA result corresponds to this region. At 640 °C the sample contains 2 % residue which continues to drop gradually to 1.3 % at 1000 °C (Kure *et al*., 2017). The TGA result shown in Figure 4 (b) shows 10 wt % weight reduction at 343 °C due to the presence of amorphous carbons and structural defects which further confirms the Raman analysis as depicted in Figure 2(a) and (b); a rapid decomposition of the sample was observed from 90 wt % to 4.6 wt % within the temperature range of 343 °C to 490 °C respectively; thus, this suggests the presence of carbon nanomaterials (Muhammad *et al*., 2018). The DTGA analysis showed a major protruding peak at 421.5 °C with 50 wt % loss which falls below what is required for CNTs when compared with previous results obtained by other research groups. However, this result is justifiable since the presence of iron oxide in CNS sample could result in lower decomposition temperature (Muhammad *et al*., 2018).

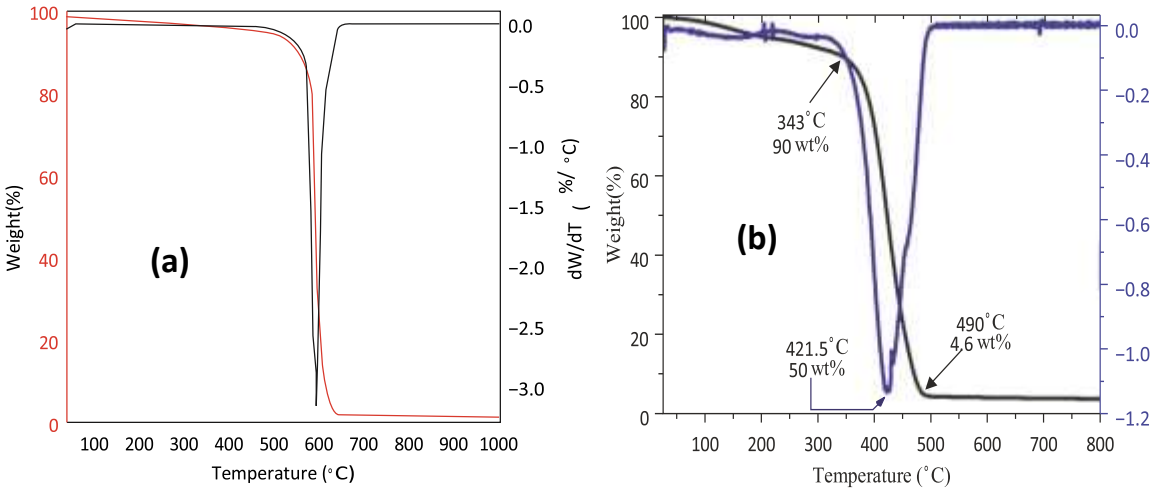

Figure 4 (a) TGA result of PE CNS (b) TGA result of RH CNS

CONCLUSION

This study shows that, the alternative technique of synthesizing CNS via Microwave-Assisted PECVD at the temperature of 750 °C, is more economical and faster which depend on the nature of the carbon precursors susceptibility to microwave irradiation. Hence, the time duration of CNS derived from PE is at 4 minutes and RH at 38 minutes. The CNS carbon quality was found to be 0.97 and 1.01 for PE and RH respectively, FESEM shows flower-like structures of average diameter size to be (16.0 to 20.0) ±0.5 nm for the both carbon precursors. The carbon purity was 90% to 50% for PE and RH respectively. CNS may be produced with high yield and purity if parameters are properly control.

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