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Production of Multilayered Nanostructure from Zingiber Officinale by Spray Pyrolysis Method

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S. Kalaiselvan [α] N Mathan Kumar [σ] & J Manivannan [ρ]

Abstract- This present work describes exploring cost-effective a natural renewable Environment-friendly green precursor for the synthesis of Multi-walled carbon nanotubes (MWCNTs) using methyl ester of *Zingiber officinale* oil over Fe-Co impregnated alumina support at 850°C under N_2 atmosphere. The characterization of the as-grown carbon materials was analyzed by Scanning electron microscopy (SEM), High-resolution transmission electron microscopy (HRTEM), XRD and Raman spectroscopic analysis. The bimetallic catalyst of Fe and Co supported on alumina gel particles improves the quality and uniformity in diameters of CNTs. The diameters of as-synthesized nanotubes are in the range of 22 nm to 24 nm.

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I. Introduction

Right from the discovery by Iijima [1], carbon nanotubes attracted significance attention. In general, CNTs are synthesized by arc discharge, laser ablation, and CVD or spray Pyrolysis. Among these method spray pyrolysis method is regarded as a promising method. There are few reports on the synthesis of CNTs from various natural precursors [2-11]. The advantages of the plant derived carbon precursor are that it is a very cheap, renewable biomaterial, green, and abundantly available hence, the resulting product could be commercialized at a lower cost. The present work aims to utilize renewable precursor *Zingiber officinale* oil for the synthesis of CNTs over Fe-Co impregnated Silica support.

II. Experimental Methods

The scheme of our home-made experimental spray-pyrolysis set-up and sprayer used for the synthesis of carbon nanotubes were represented in Figure 1 and 2.

a) *Preparation Catalyst Nanoparticles and Synthesis of Nanotubes*

The bimetallic catalyst of cobalt and iron supported on silica gel particles were used as a catalyst for CNT growth by spray pyrolysis. The mixture of $Co(NO_3)_3.\ 3H_2O$ and $Fe(NO_3)_3.\ 6H_2O$ were heated at 90 °C to liquefy the Co and Fe nitrates. A one-third weight of alumina gel concerning to the total weight of metal nitrates was added into the liquefied mixture of Co and Fe nitrates and stirred well for 30 min. The mixture of catalyst was cooled down to room temperature and kept in the oven for drying at 60 °C for 24 hours.[12] The complete dried catalyst ground into fine particles in an agate mortar. The fine powder were then calcined for 1 hour at 450 °C and then re-ground before loading into the reactor. The catalyst placed on the quartz boat. The boat placed in the heating furnace. The carrier gas nitrogen (100 ml/min) was flushed out before switch on the reaction furnace to remove air and create nitrogen atmosphere.

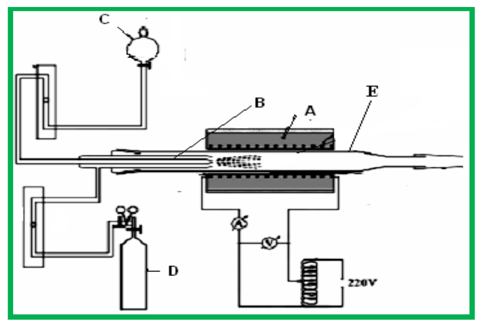

Figure 1: The schematic diagram of the spray pyrolysis set-up. (A) Heating source, (B) Spray nozzle, (C) Carbon feedstock inlet, (D) Nitrogen gas, (E) Quartz tube.

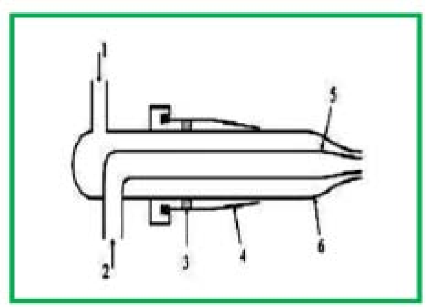

Figure 2: Schematic diagram of the Sprayer 1. Gas inlet; 2. Solution inlet; 3. Tightening; 4. Polished glass-to-glass connection; 5. Inner Pyrex tube; 6. Outer Pyrex tube

The temperature was raised up to the desired growing temperature. Subsequently, *Zingiber officinale* oil introduced into the quartz tube through the spray nozzle, the flow was maintained using the saline tube at the rate of 0.5 ml/min. The deposition time lasted for 45 minutes for each deposition at different temperatures from 550-850°C. The reactor was then allowed to cool to

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room temperature with nitrogen gas flowing. The carbon product on the silica support was collected and characterized by Raman Spectroscopy, X-ray diffraction (XRD), scanning electron microscope and High-resolution transmission electron microscope (HRTEM).

III. Results And Discussions

Fig. 3a and 3b show the scanning electron microscopy image of the as-grown nanostructures over Fe-Co bimetallic catalyst, impregnated in alumina at 850 °C under the flow of nitrogen by CVD assisted spray pyrolysis method. SEM image reveals that MWNTs grew nicely on the surface of the Alumina particles.

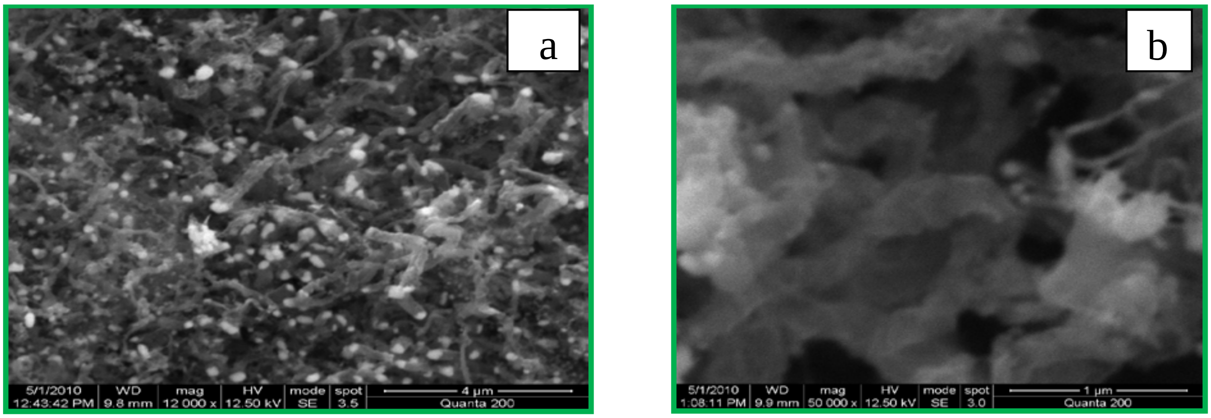

Fig. 3: a and b: SEM images of CNTs grown Fe-Co supported on Alumina at 850 °C

In Fig. 5a and 5b we have presented the high-resolution HRTEM images of MWNTs, grown over Fe-Co bimetallic catalyst impregnated on Alumina support at 850 °C with a flow rate of the methyl ester of *Zingiber officinale* oil at 0.5 mL per minute. Fig. 4a & 4b show that the inner diameters of the nanotubes were in the range of 10-12nm and the outer diameters of the grown nanotubes 22-24nm. The inner core visible (Fig. 4b).

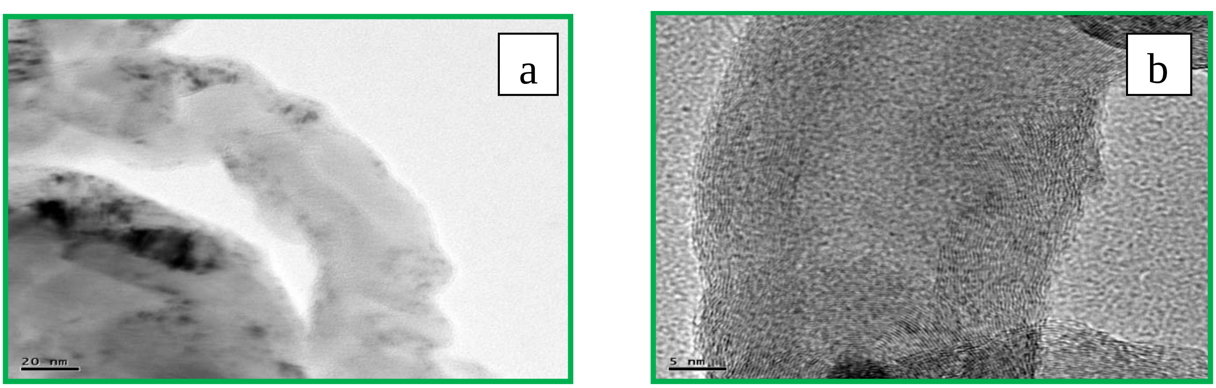

Fig. 4: a and b: HRTEM images of CNTs grown Fe-Co supported on Alumina at 850 °C

Raman Spectroscopy and XRD Analysis

The results of Raman spectroscopy of MWNTs grown on the catalyst surface at 850 °C (Fig-5), indicating two characteristic peaks at 1344.52 cm^{-1} and 1591.20 cm^{-1} which correspond to D and G bands, respectively. The G bands are related to stretching vibration in the basal plane of graphite crystal. The D peaks at 1344.52 cm^{-1} attributed to the defects in the curved graphene sheets. For high-quality samples without defects and amorphous carbon, the I_D/I_G ratio is often less than 3%[13,14]. I_D/I_G ratio was found to be 0.97 indicates lower defects which is in good agreement with theoretical evidence.Fig-6 shows the X-Ray Diffraction pattern of MWNTs. The intense peaks at 26.1° and 44.2° can be as indexed (002) and (101) reflections of hexagonal graphite. Appearance of characteristic peak of graphite shows the presence of Multi-walled carbon nanotubes in the sample [15].

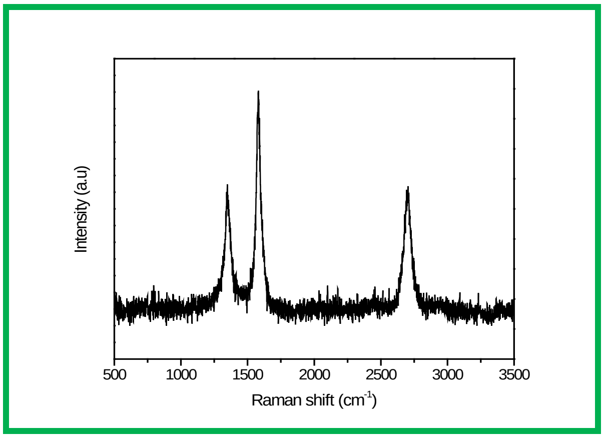

Fig. 5: Raman Spectrum of grown CNTs at 850°C

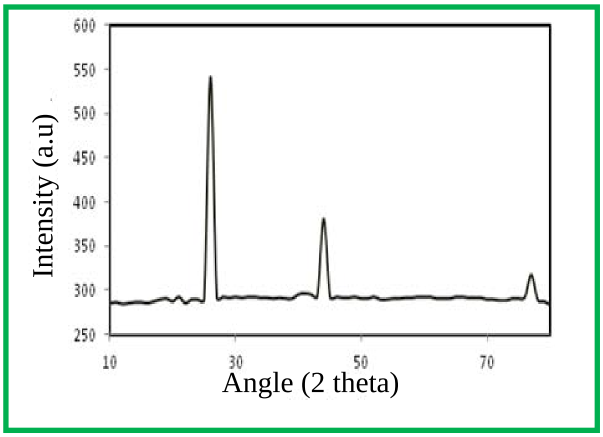

Fig. 6: XRD spectrum of grown CNTs at 850°C

IV. Conclusion

We accomplished the synthesis of MWCNTs at a temperature of 850°C using methyl esters of *Zingiber officinale* oil a renewable natural precursor, as a carbon source. CNTs morphology and structure were investigated by SEM, HRTEM, XRD and Raman spectroscopy. It was found that temperature was enough to transform hydrocarbon source into carbon nanotubes with heterogeneous diameter, good quality and yield at this temperature. The experiment has also succeeded in minimizing I_D/I_G ratio of about 0.97. The highest yield of 68% was obtained at the same temperature.

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