J. Environ. Nanotechnol.
Volume 1, No. 1 (2012) pp.46-49
ISSN (Print): 2279 – 0748
ISSN (Online): 2319 - 5541
doi: 10.13074/jent.2012.10.121016

Synthesis of One Dimensional Carbon Nanofibers from Tire Pyrolysis Oil

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Abstract

Tire pyrolysis oil derived from waste tire material has been used to synthesis carbon nanofiber on quartz substrate in Ar atmosphere by spray pyrolysis method. The structure and nature of carbon nano fiber were characterized by scanning electron microscopy, X-ray diffraction and Raman Spectroscopy. The diameters of the synthesized carbon nanofiber are 260 ± 40 nm.

KEY WORDS: Carbon nanofiber(CNFs); Spray pyrolysis; Tire pyrolysis oil and quartz substrate.

Received: 4.09.2012 Accepted: 26.10.12

INTORDUCTION:

Carbon nanofibers have attracted the tremendous attention because of their potential applications in the field of science and technology. The CNFs can be used in hydrogen storage materials (Park et al., 1999), field emission devices, sensors, fuel cells and super capacitors (Dai et al., 2002). CNFs are chemically stable for corrosive attack in acidic and basic environment. Therefore, extensive efforts have been made to develop CNFs by different methods using different metal catalyst (Fe, Co, Ni, Cu, etc) and different carbon feed stock. However, most of the work done with zero valence iron compounds, such as $Fe(C_5H_5)_2$ or $Fe(CO)_5$ in order to favor and control the catalyst particle size (Hoque et al., 2001) to grow selective nanofiber. To date, various petroleum by products, such as methane (Benissad- Aissani et al., 2004), xylene (Martin-Gullon et al., 2006), benzene (Endo et al., 2001) etc., are in practice to synthesize CNFs. However, in view of foreseen crisis of fossil fuels in the near future, it is desirable to look for alternative carbon feedstock to synthesize various kinds of nanomaterials. However, these are few reports on the synthesis of CNFs from natural precursor, such as turpentine oil (Ghosh et al., 2008), camphor (Pradhan et al., 2003) etc. Disposal of waste scrap tire is a major environmental and economical issue (Cunliffe et al., 1998). Tires have been recycled as grinding, crumbing, re-treading, reclaiming, combustion and pyrolysis (Kaminsky and Mannerich, 2001). Pyrolysis offers an environmentally attractive method to decompose wide varity of wastes, including scrape tires (Karthikeyan et al., 2012). Tire pyrolysis yield: solid char, 30-40; liquid residue, 40-60; and gases, 5-20 wt%. Solid carbon residue may be used as reinforcement in rubber industry or fuel. Gaseous fraction can be used as fuel in pyrolysis process. Tire pyrolysis oil fraction can be used as alternative fuel. A detailed study of the growth of structure carbon nanofiber from tire pyrolysis oil has not to the best of our knowledge, been published. The aim of the present study is to grow carbon nanofiber on quartz substrates by spray pyrolysis of inexpensive precursor tire pyrolysis oil. Designing of catalyst material and optimization of reaction parameters , which is suitable for specific morphological one dimensional carbon nano structures from tire pyrolysis oil is one of the future prospects in this area of research.

EXPERIMENTAL PROCEDURE:

The synthesis of CNFs was carried out using the spray pyrolysis method. In this spray pyrolysis method; pyrolysis of the carbon precursor with a catalyst take place followed by deposition of CNF occurs on quartz substrate. Tire pyrolysis oil was used as carbon source and ferrocene [Fe $(C_5H_5)_2$] (Sigma Aldrich, high purity 98 %) was used as a source of Fe, which acts as a catalyst for the growth of CNFs. Quartz substrate of size ($1x1cm^2$) was used as a substrate. The spray pyrolysis setup consisted of a nozzle (inner diameter~0.5mm), attached to a precursor solution supply used for spraying the solution into a quartz tube (500mm long with an inner diameter of 30 mm). The outer part of the quartz tube was attached with a water bubbler. Before used, substrate was cleaned properly in acetone by ultra-sonication followed by de-ionized water and finely dried using argon blower. The substrate was kept in a quartz boat which was then placed at the center of the quartz tube. In a typical experiment, the quartz tube was first flushed with argon (Ar) gas in order to eliminate air from the quartz tube and heated to a reaction temperature.

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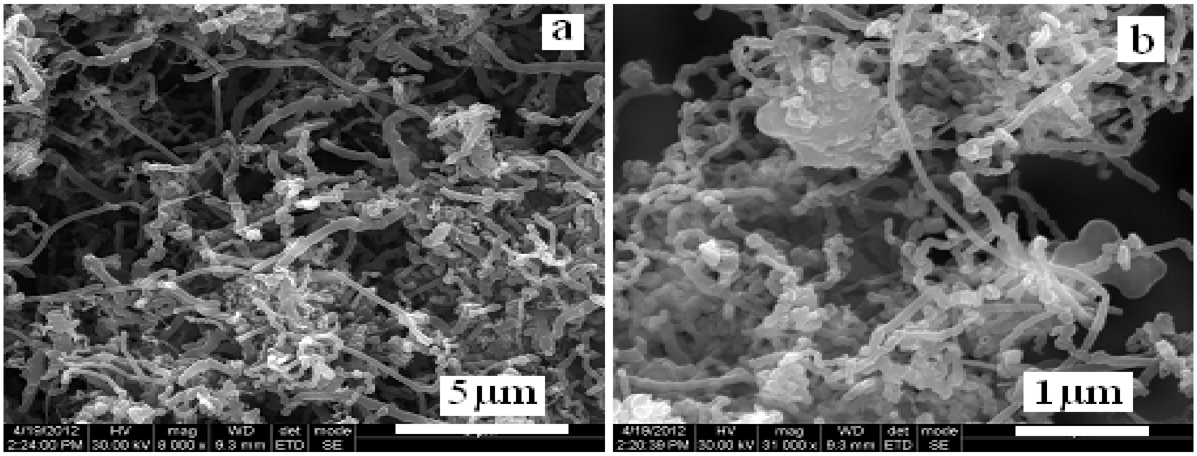

Fig. 1 a & b : HRSEM images of the as-grown CNFs at 850

The precursor solution (Tire pyrolysis oil and ferrocene mixtures) was sprayed into the quartz tube, using Ar gas. The concentration of ferrocene in Tire pyrolysis oil was ~25 mg/ml. The solution was sonicated for 5 min to prepare the homogeneous mixture. The flow rate of Ar was 200sccm/min. The experiments were conducted at 850 ºC and 1 atmospheric pressure, with reaction time of 45 min was maintained for each deposition. After deposition, the furnace was switched off and allowed to cool down to room temperature under Ar gas flow. A uniform black deposition on the quartz substrate at the reaction zone was observed. Finely, the substrate containing entangled CNFs was removed from the quartz tube for characterization.

The as-grown CNFs materials were characterized using scanning electron microscope (SEM was performed by Hitachi-3000 H). Raman spectroscopy of samples was performed by JASCO NRS-1500 w, green laser with excited on wave length 532 nm.

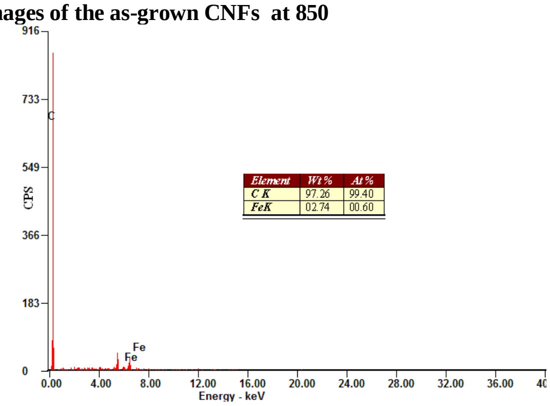

Fig. 2: EDX of the as-grown CNFs at 850.

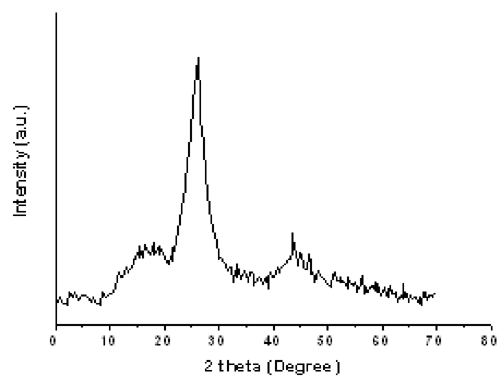

Fig. 3 : XRD spectra of the as-grown CNFs at 850.

RESULT AND DISCUSSION:

Fig.1 shows Scanning electron microscopic image of as-grown CNFs deposited on quartz substrate at 850 ° C in Ar atmosphere using TPO by spray pyrolysis. HRSEM images (Fig.1 a & b) illustrate a high density of fibers with diameter of 260 ± 40 nm laid down on quartz substrate, with long randomly oriented spaghetti-like structures. Small bright catalyst particle were seen at the tip of the fibers in fig (1.a). These results are confirmed by other workers (Sengupta et al., 2009; Sengupta and Jacob 2010) and they suggested that this is due to tip growth mechanism.

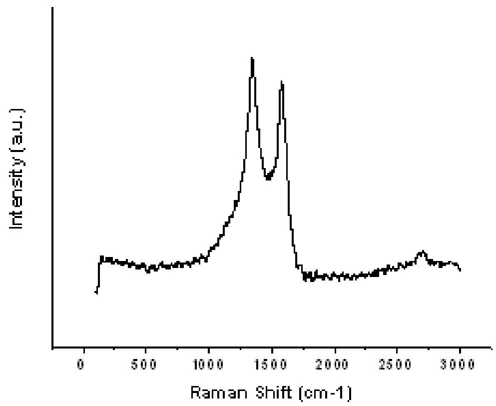

Fig. 4 : Raman spectra of the as-grown CNFs at 850.

Energy dispersive X-ray (Fig. 2) analysis shows the amount of carbon and iron. Carbon is the major element in the deposited products with 97.26 wt % followed by iron content of 02.74 wt %. Which is in agreement with our SEM observation that metal particle in our sample is negligibly small The CNFs crystal structure was studied by X-ray diffraction. In Fig. 3 the intense peak at ca. 26.35° can be indexed to the (002) diffraction plane of hexagonal graphite. The (002) peak indicates the graphitic structure of the CNFs.

Raman spectroscopy is wildly used to characterize the structural and phase disorder information in carbon related material. Fig. 4 shows Raman spectra of as-grown CNFs on quartz substrate indicating two characteristic peaks at 1346 cm^{-1} and 1576 cm^{-1} corresponding to D and G bands, respectively. The G bands are related to stretching vibration in the basal plane of graphite crystal, which have been normalized to the same intensity. D bands are associated with disorder (or) detective planner graphite structure. The D peaks at 1346 cm^{-1} has been known to be attributed to the defects in the curved graphene sheet. Therefore, the Raman spectrum provides definite evidence that the CNFs have graphitic structure.

CONCLUSION:

The main conclusion of this work is the growth of CNFs on quartz substrate by spray pyrolysis using tire pyrolysis oil which is derived from waste tire material. From the above experimental results, we conclude that the carbon nanofiber diameter of 260 ± 40 nm was observed with HRSEM. The scientific and commercial interest has increased immensely, disclosing this field as a most important technology of the future.

ACKNOWLEDGEMENTS

The authors acknowledge the Institute for Environmental Nanotechnology for technical support and IITM for access to Electron Microscopes.

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