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Studies on The Synthesis of Carbon Nanomaterial using Pyrolysis oil as Feed Stock from Printred Circuit Board (PCB) of Waste Electrical and Electronic Equipments (WEEE)

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Abstract: The aim of the present study is to synthesis suitable carbon nano material using pyrolysis oil as feed stock from printred circuit board (pcb) of waste electrical and electronic equipments (weee). According to the American Journal of Environmental Engineering, electronic waste (E-waste) is the fastest growing stream in the industrialized world. It is estimated that 3-5% of e-waste consists of PCBs/connectors that need environmentally friendly recycling techniques to manage. The original research focuses on catalytic pyrolysis of waste PCBs to generate energy rich e-oil and by using spray pyrolysis method carbon nano material (CNMs) are synthesized on a crystal surface under inert environmental conditions. For the synthesis of carbon nano material naturally occurring organic material are used as a feed stock initially and hence the study further reveals that the structure of the resulting CNM is determined by the feedstock type. Thus liquid condensate obtained from the pyrolysis of printed circuit board is used as a raw material for the synthesis of carbon nano material by using spray pyrolysis technique under inert Nitrogen environment on the surface of a quartz crystal and the study demonstrates an efficient method to synthesize CNMs. The future study deals with application of synthesized Carbon Nano Materials (CNMs) as a catalyst to improve the yield of pyrolysis oil.
Key words: Pcbs, CNMs, Pyrolytic oil, Quartz crystal.

1. Introduction:

Due to the advancement in science and technology and change in life style of an individual, the electronic equipments are getting outdated before reaching the end of life. This has resulted in mass generation of e-waste which requires proper disposal. According to the American Journal of Environmental Engineering, electronic waste (E-waste) is the fastest growing stream in the industrialized world. It is estimated that 3-5% of e-waste consists of PCBs/connectors that need environmentally friendly recycling techniques to manage.

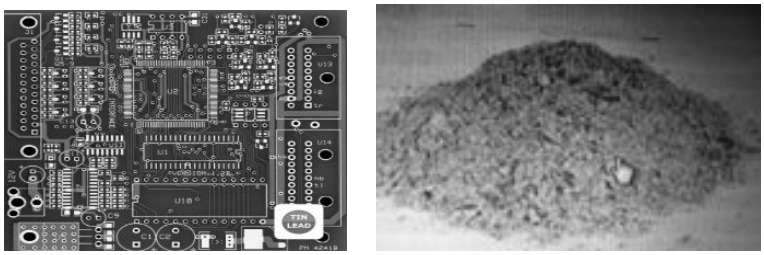
The printed circuit boards are an integral part of all electrical and electronic equipments. The aim of the present study is to efficiently utilize the waste printed circuit board for the synthesis of carbon nanomaterials. The past five years have witnessed relentless growth in the research, development and technological understanding of CNMs. The global CNMs market's revenue was estimated to be worth $926.3 million in 2012, which is expected to reach $2,398.4 million by 2018, growing at a CAGR of 15.5% from 2013 to 2018[1-2].

Industrial-scale production of CNMs is typically achieved through chemical vapor deposition (CVD) methods or by flame synthesis techniques. The spray pyrolysis deposition technique is gaining traction in the scientific community due to its cost effectiveness and ease of integration in to a standard CMOS process. The technique is used to grow crystal powders.

The study deals with the synthesis of Carbon Nano Materials (CNMs) using pyrolysis oil as feed stock from printed circuit board (pcb) of waste electrical and electronic equipments (weee) and by using spray prolysis method, on a quartz crystal surface, under inert Nitrogen environment.

2. Experimental Procedure

PCBs are collected from various electrical and electronic equipments. Collected Pcbs are pulverized by using different size reducing equipments like ballmill, jaw crusher and drop weight crusher. Mixture of equal volumes of equal normal HCl, H_2SO_4 and HNO_3 called "Mixed Acid" is prepared in which powdered PCB's are added and it is found to be effective in removal of most of the metals by leaching process[3]. The resultant mass is then dried in a Tray drier at 100°C for 15-20 minutes. The drier is inspected for every 5 minutes to prevent any burning.



Waste PCB **Pulverized PCB**

Fig.1 Feed stock used for obtaining pyrolytic oil.

The pulverized and dried PCB is pyrolysed in a fixed bed reactor [3], shown in Fig.2

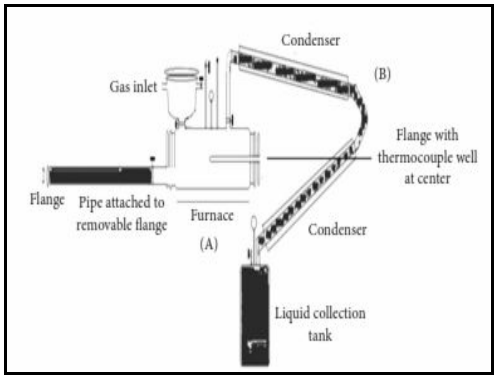


Fig.2. Schematic representation of fixed bed pyrolytic reactor

The experiment is carried out at a temperature of 650°C in an inert nitrogen environment to yield pyrolytic oil [4-7]. The properties of the oil obtained are shown in Table 1.

Table 1. Properties of pyrolytic oil

Properties	Oil Characteristics
Colour	Reddish brown to dark green
Odor	an acid smoky smell
Density	1.3 kg/liter
Viscosity	Can vary from 25-1000 centistokes
Heating value	lower than fossil oil

The synthesis of CNMs was carried out in a spray pyrolysis unit by using liquid condensate as a feed stock. The carbon precursor in the presence of a catalyst is subjected to pyrolysis which resulted in deposition of CNMs on a quartz substrate [8-10]. .

PCB pyrolysis oil was used as carbon source and ferrocene [Fe (C5H5)2] (Sigma Aldrich, high purity 98 %) was used as a source of Fe, which acts as a catalyst for the growth of CNMs. Quartz crystal of size ($1x1cm^2$) was used as a substrate[11]. .

The spray pyrolysis set up consisted of a nozzle (inner diameter~0.4mm), attached to a precursor-solution-supply used for spraying the solution into a quartz tube (~300 mm long with an inner diameter of ~25 mm). The outer part of the quartz tube was attached with a water bubbler[12]. Prior to use, the substrate was cleaned properly with acetone by ultra-sonication followed by de-ionized water and finely dried using Nitrogen blower. The substrate was kept in a quartz boat which was then placed at the center of the quartz tube. The quartz tube was first flushed with Nitrogen gas in order to eliminate air and heated to a reaction temperature.

The precursor solution (PCB pyrolysis oil and ferrocene mixtures) was sprayed into the quartz tube, using Nitrogen gas. The concentration of ferrocene in PCB pyrolysis oil was ~15 mg/ml. The solution was sonicated for 10 min to prepare the homogeneous mixture. The flow rate of Nitrogen was 250 sccm/min. The experiments were conducted at 650 °C and 1 atmospheric pressure, with reaction time of 1 hr. was maintained for each deposition. After deposition, the furnace was switched off and allowed to cool down to 30°C under Nitrogen gas flow. A uniform black deposition on the quartz substrate at the reaction zone was observed. Finely, the substrate containing entangled CNMs was removed from the quartz tube for characterization [13]. .

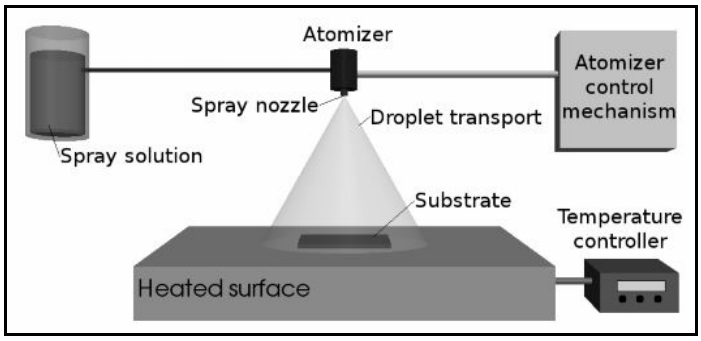
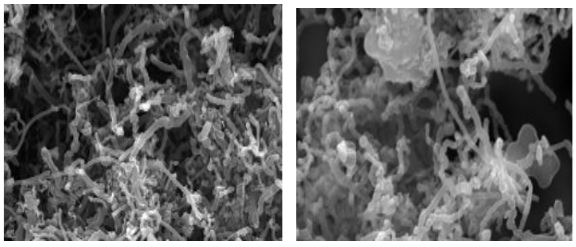


Fig.3. Schematic representation of spray pyrolysis unit

The N_2-grown CNMs 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.

3. Result and Discussion:

Fig.4 shows Scanning electron microscopic image of as-grown CNMs deposited on quartz substrate at 650 ° C in Nitrogen atmosphere by spray pyrolysis[14]. .



5 µm **1 µm**
Fig.4. HRSEM images of CNMs at 650 ºC

HRSEM images (Fig.4) illustrate a high density of fibers with diameter of 250 ± 50 nm laid down on quartz substrate, with long randomly oriented spaghetti-like structures.

Energy Dispersive X-ray (Fig. 5) 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.

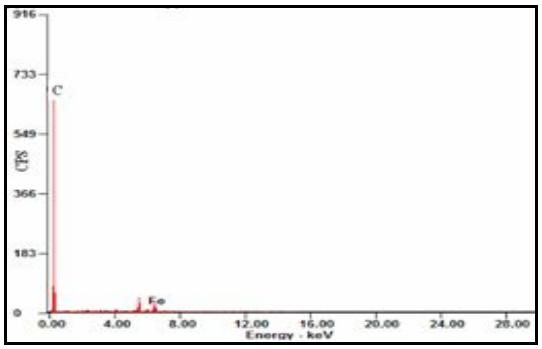


Fig.5. EDX of the CNMs at 650ºC

The CNMs crystal structure was studied by X-ray diffraction. In Fig. 6 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 CNMs.

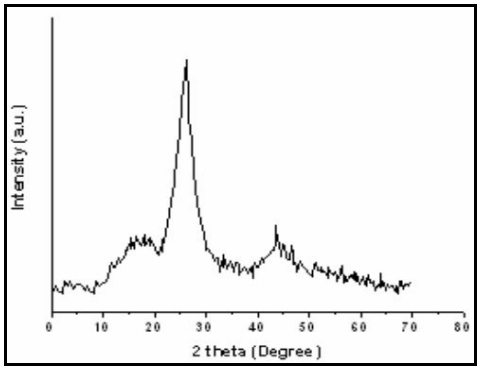


Fig.6. XRD spectra of the CNMs at 650 ºC

Raman spectroscopy is wildly used to characterize the structural and phase disorder information in carbon related material. Fig. 7 shows Raman spectra of as-grown CNMs on quartz substrate indicating two characteristic peaks at 1346 cm 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 CNMs have graphitic structure.

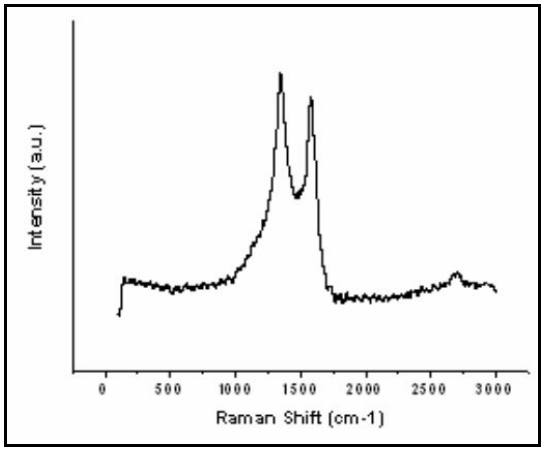


Fig.7. Raman spectra of the CNMs at 650 ºC

The main conclusion of this work is the growth of CNMs on quartz substrate by spray pyrolysis using PCB pyrolysis oil which is derived from waste PCB. From the above experimental results, we conclude that the carbon nano-fiber diameter of 250 ± 50 nm was observed with HRSEM.

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