

Synthesis of carbon nanoparticles from kerosene and their characterization by SEM/EDX, XRD and FTIR

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Abstract: Carbon nanoparticles (CNPs) were synthesized by a simple way of incomplete combustion of kerosene. Synthesized nanoparticles were characterized by Scanning electron microscope (SEM), Energy dispersive x-ray (EDX), Powder x-ray diffractometry (XRD) and Fourier transform infrared spectroscopy (FTIR). The SEM particle sizes of prepared CNPs were found to be non-uniform. The average size of particles is in the range of 20-100 nm. EDX analysis predicts the presence of pure carbon without any contamination. X-ray powder diffractometric analysis of prepared CNPs indicates the presence of large amounts of amorphous carbon material in association with hexagonal graphite lattice. FTIR spectroscopic analysis shows that the prepared CNPs are a mixture of elemental carbon and a trace amount of hydrocarbons.

Keywords: Kerosene, Carbon Nanoparticles, Characterization, SEM, XRD and FTIR

1. Introduction

Carbon nano particles/tubes/fibers are the most researched materials of the 21st century with an international intention of growing industrial quantities due to their unique properties such as good electrical/thermal conductivity, enhanced chemical/bio compatibility and excellent corrosion resistance [1-3] for wide range of applications which include polymer composites, electrochemical energy storage and conversion, filtration, hydrogen storage, catalysis and biotechnology [4-7]. Successful utilization of carbon nanoparticles in various applications is strongly dependent on the development of simple, efficient and inexpensive technology for its production.

Different methods such as thermal carbonization, sonication, laser irradiation and exfoliation have been used for synthesizing of different carbon nano particle/tubes/fibers [2, 8-10]. Raw material of carbon is one of the most important factors in controlling the morphology and the yield of the carbon nanoparticles. Various raw materials such as coal [11], scrap tyre rubber [12], ethanol [13], arc-discharge and thermal plasma jet [14], etc. have been used for the synthesis of carbon nanoparticles.

Chemical vapour deposition of coal [11] and scrap tyre rubber appears [12] to be promising methods but producing

a mixture of different carbon nanomaterials. Catalytic decomposition of ethanol [13] or hydrocarbon gases [15] over selected metal particles that include iron, cobalt, nickel, and some of their alloys at temperatures over the range 400 to 1000°C produce carbon nanoparticles. Both arc-discharge and thermal plasma jet produce high quality carbon nanoparticles but expensive, thus limit their use as large-scale synthesis [14].

However, there is a need for alternate carbon sources for the synthesis of pure and inexpensive carbon nanoparticles due to increasing demand for carbon-based nanomaterials in various emerging fields. Hence, kerosene as a carbon rich and cost effective raw material receives our great attention for the synthesis of carbon nanoparticles. Herein, we report the synthesis of carbon nanoparticles from the incomplete combustion of kerosene and their morphological structure was investigated by using SEM, EDX, XRD and FTIR studies.

2. Experimental

2.1. Synthesis of Carbon Nanomaterials

Kerosene was collected from local market in Dhaka city of Bangladesh. A glass round bottom flask, lamp and lamp-wick were finely cleaned with detergent and followed by acetone. Then these were dried in oven at 120 °C.

Kerosene was taken in the lamp to use it as fuel. At first the kerosene lamp was fired and a glass round bottom flask was placed over the flame of lamp to prevent the excess air oxygen. During the burning of kerosene, black colored materials were prepared which was deposited inside of the glass round bottom flask. Deposited materials were collected in a dry bottle and stored in a desiccator. Then the percent yield, and the production cost were calculated.

2.2. Characterization of Carbon Nanoparticles

The morphological structure of deposited black materials was studied by scanning electron microscope (SEM), energy-dispersive X-ray (EDX), X-ray diffraction (XRD) and Fourier transform infrared (FTIR) spectroscopy.

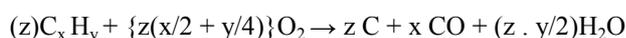
For SEM analysis, prepared samples were spread on glue-covered aluminum stubs. The stub mounted samples were imaged on a scanning electron microscope (JSM-6490, JEOL, Japan) operated at 20 kV and observed the surface morphology at different magnifications from 25,000 to 2,00,000 times. Elemental analysis of prepared sample in SEM was performed by taking a spectrum of energy dispersive x-ray (JED-2300 Analysis Station, JEOL, Japan).

Powder X-ray diffraction patterns of prepared sample were taken by a two-circle (2θ - θ) X-ray powder diffractometer (X'Pert PRO XRD PW 3040 system) using Copper $k\alpha_1$ of wavelength 1.54056×10^{-10} m, and $k\alpha_2$ of wavelength 1.544426×10^{-10} m. The scan was taken between 2θ of 10° and 2θ of 45° at increments of 0.04° with a count time of 4 seconds for each step. Fourier transform infrared spectroscopy (FTIR) spectroscopic study of the sample was performed by IR Prestige 21, Shimadzu, Japan, using KBr as a reference. 0.5 g dried carbon sample was mixed with KBr (sample/KBr ratio was 1/100) and were pressed into transparent thin pellet. A FTIR spectrum of carbon materials was obtained in the range on 4000 to 400 cm^{-1} . Spectral output was recorded by the transmittance as a function of wave number.

3. Results and Discussions

3.1. Preparation of Carbon Nanoparticles

Kerosene is a mixture of hydrocarbons of the formula C_nH_{2n+2} , but it has no definite molecular formula. It consists of a variety of different hydrocarbons of a molecular weight greater than that of gasoline, and less than that of oil. When hydrocarbon is oxidized with high temperature, different types of combustion reactions i.e. complete and incomplete combustion takes place [16]. Incomplete combustion is occurs when there is not enough oxygen to allow the fuel to react completely to produce carbon dioxide and water. It also happens when the combustion is quenched by a heat sink such as a solid surface or flame trap. The stoichiometric chemical equation for incomplete combustion of hydrocarbon in oxygen is as follows [16]:



For example, incomplete combustion of dodecane in oxygen is as follows:



Complete combustion is almost impossible to achieve. In reality, as actual combustion reactions come to equilibrium, a wide variety of major and minor species will be present such as carbon monoxide and pure carbon. Additionally, any combustion in atmospheric air, which is 78% nitrogen present, will also create several forms of nitrogen oxides [17]. In our experimental set up, only carbon materials can be deposited on the surface of glass plate which was subsequently characterized by SEM/EDX, XRD and FTIR analysis. Percent yield of deposited carbon material was calculated (3%) which was equivalent to the cost of US\$ 27 per kg.

3.2. SEM-EDX Analysis

Prepared carbon materials deposited on the glass surface were investigated by scanning electron microscope at different magnifications to characterize the surface morphology of prepared material. Fig. 1 presents the SEM micrographs of carbon particles at 25,000 to 2,00,000 times magnification which clearly shows the non-uniform size spherical particles. The prepared spherical particles are in a size range between 20 and 100 nm in diameter but most of the particles are about 40 nm in diameter.

The elemental analysis of prepared spherical nanoparticles was performed using the energy-dispersive X-ray (EDX) spectroscopy arranged on the SEM. Fig. 2 shows the EDX spectrum of the prepared materials which indicated the presence of pure carbon without any contaminated substances in detectable limit ($\pm 0.01\%$).

3.3. XRD Analysis

X-Ray powder diffractometric analysis of carbon nanoparticles was carried out to identify their crystal structure. The XRD spectrum of carbon nanoparticles in Figure 3 shows that there are two Bragg diffraction peaks at near $2\theta = 23.68^\circ$ and 44.01° . It has been reported that the XRD peak at near $2\theta = 23.68^\circ$ indexed as (002) is an indication of the presence of large amounts of amorphous material in association with multi-walled carbon nanotubes [18-19, 23-24], and the peak at near $2\theta = 42.33^\circ$ indexed as (101) plane is an indication of the low quality of carbon nanomaterials [19-24]. It has also been reported that the peak at near $2\theta = 43.7^\circ$ indexed as (101) plane is an indication of the presence of hexagonal graphite lattice [25]. In the present study, the peaks at near $2\theta = 23.68^\circ$ and 44.01° were indexed as (002) and (101) planes which correspond to the presence of large amounts of amorphous carbon nano-materials in association with hexagonal graphite lattice.

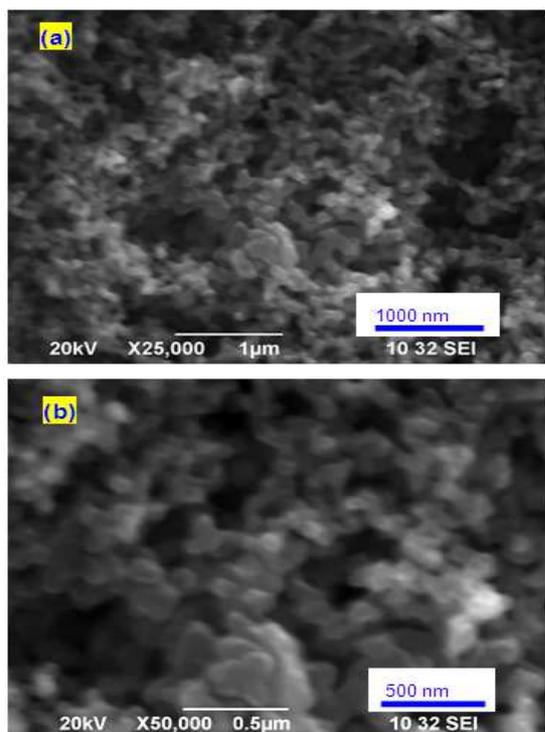


Figure 1(a & b). SEM micrographs of carbon nanoparticles prepared from kerosene at low magnifications.

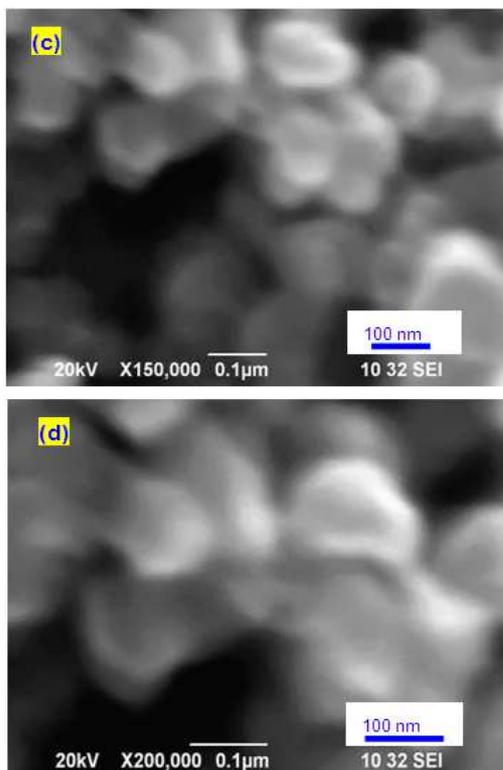


Figure 1(c & d). SEM micrographs of carbon nanoparticles prepared from kerosene at high magnifications.

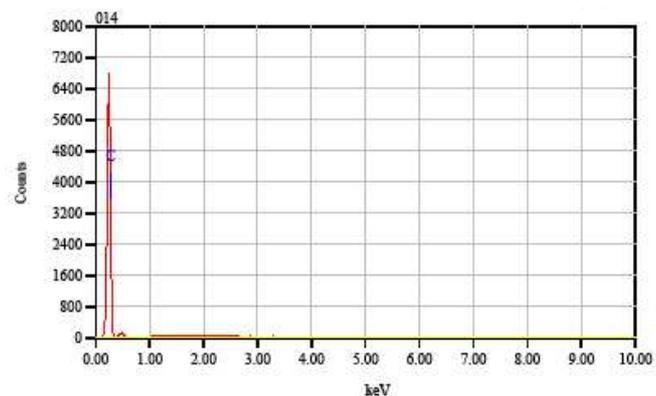


Figure 2. EDX spectrum of carbon nanoparticles prepared from kerosene.

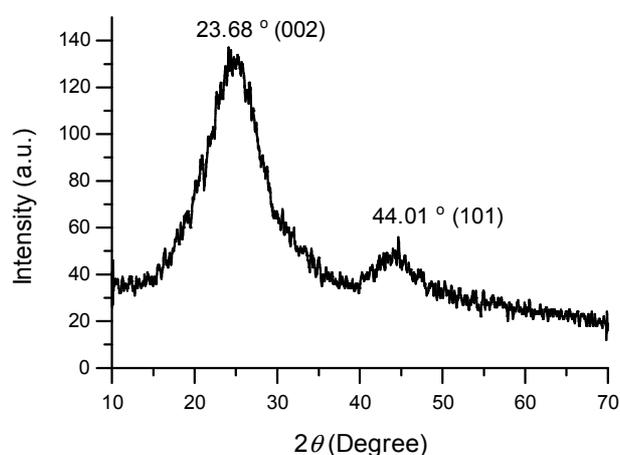


Figure 3. XRD spectrum of carbon nanomaterials prepared from kerosene.

3.4. FTIR Study

FTIR spectroscopic analysis was carried out to identify the chemical structure of carbon particles as well as the presence of any functional group in carbon nanoparticles. Thermal decomposition of kerosene could be produced other substances along with carbon particles [22]. It has been reported that the atmospheric combustion of kerosene produced a complex mixture of elemental carbon, a variety of hydrocarbons and other species [22]. Figure 4 shows the FTIR spectrum of prepared dried carbon nanoparticles, which indicated the presences of some functional groups of hydrocarbon and oxygen. In FTIR spectrum, peak at 3436 cm^{-1} (m) is for O-H stretch, peak at 2927 cm^{-1} is for C-H stretch in alkane or in aromatic compounds. A very weak peak at 1630 is for C=C aromatic stretch. Peaks at 1465 , 1383 and 1327 cm^{-1} are for C-H bend in CH_3 and the peak at 1118 is for C-O stretch [26]. Since EDX is more sensitive method than FTIR method, the signals for O-H and C-O groups might be due to the contamination of moisture during the taking of FTIR spectrum or the presence of very small amount of oxygen which is less than the detection limit of EDX (0.01%). Thus the prepared carbon nanoparticles may contain very small amount of hydrocarbons with trace level

of oxygen (< 0.01%). Further study with more sensitive instruments is required to know the actual composition of prepared carbon nanoparticles from kerosene due to the inconsistency of FTIR data with EDX analysis. But the EDX analytical data showed that 72.63% (elemental weight) of oxygen present in CNPs prepared from kerosene using other reported methods [19, 23]. It is clear that the purity of CNPs prepared in the study is comparatively high than the reported methods [19, 23].

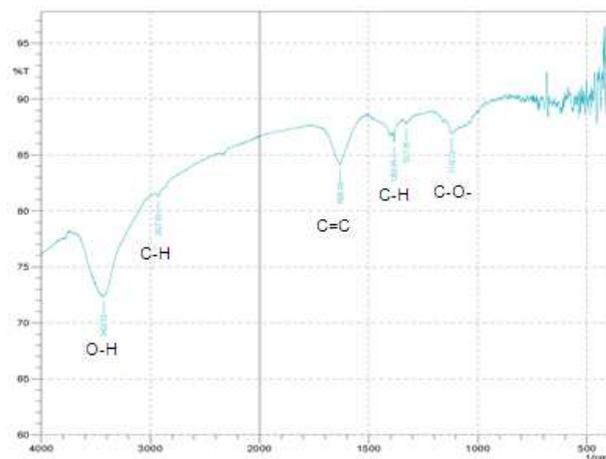


Figure 4. FT-IR spectrum of carbon nanoparticles synthesized from kerosene.

4. Conclusions

Nanostructure carbon was prepared from incomplete combustion kerosene fuel which is a very cheap and easy method. Non-uniform size of spherical carbon nanoparticles were observed by SEM analysis and purity of carbon was confirmed by EDX analysis. The average particles size of CNPs is in the range of 20-100 nm. The presence of a large amount of amorphous carbon nanoparticles in addition with a small amount of hexagonal graphite lattice were confirmed by X-ray diffraction study.

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