Preparation of Carbon Nanoparticles from Candle and Their Characterization by Advanced Spectroscopic Methods

Mohammad Abul Hossain* and Shahidul Islam

Department of Chemistry, Faculty of Science, University of Dhaka, Dhaka-1000, Bangladesh

Abstract

This study presents the preparation of carbon nanoparticles (CNp) from incomplete combustion of the candle under a controlled air supply. The shape, size, and purity of the prepared carbon particles were investigated by using a scanning electron microscope (SEM), energy dispersive x-ray (EDX), X-ray diffractometer (XRD), Fourier transformed infrared spectroscopy (FTIR), and laser-induced breakdown spectroscopy (LIBS). Non-uniformed size of carbon particles was observed by SEM studies. Most of the particles are spherical and the diameter is in the range between 10 and 70 nm. The formation of carbon particles was affirmed by EDX analysis. Mostly amorphous carbon material, in addition to some hexagonal crystal lattice of graphite, was identified by XRD analysis. FTIR spectroscopic analysis indicated the possible presence of oxygen with carbon in the prepared nanoparticles. LIBS analysis confirmed the existence of trace amount (< 0.01 wt%) of oxygen as an impurity with carbon in the prepared nanoparticles.

Keywords: Candle, incomplete combustion, spherical carbon nanoparticles, amorphous and hexagonal graphite lattice of carbon.

I. Introduction

Recent understanding of the unique properties of carbon nanoparticles and their broad application has led to a great interest in research worldwide[1]. An extensive variety of carbon nanostructures such as carbon nanotubes[2], nanoparticles, and nanofibers[3], carbon nanoshells onions[4], and cubic nanocages[5-8] are observed. Carbon nanoparticles are used in water treatment[9], filler in rubber[10] enhancing mechanical and electrical properties of epoxy composites, synthesis of polar vapor sensor[11] and hydrogen sensor[12]. Carbon nanoparticles are used to synthesize another metal nanoparticle like palladium which is an electro catalyst[13].

Carbon nanoparticles and nanofibers are being prepared from synthetic polymers by carbonization processes such as polyvinyl alcohol (PVA) and different hydrocarbons, and the pyrolysis of methane, ethanol, benzene, acetylene. Sometimes a mixture of different types of particles is found, and electrostatic technique[14] is used to separate carbon nanoparticles. Fluorescent carbon nanoparticle[15], photoluminescent (PL)[16-17], zero fullerene onion-like and two-dimensional[18], magnetic[19-20], and other different properties carbon nanoparticles are possible to synthesize.

The unique properties of high porosity, low density, increased surface area, and relatively high thermal and chemical stability of carbon nanospheres (CNS) are governed by their semi-crystalline structure[21]. CNS can be prepared by a series of methods such as the arc-discharge[22-25], laser ablation[26], and chemical vapor deposition[27], $Ar/H_2/C_2H_2$ plasmas[28], high current pulsed arc[29] practices with inorganic metals. Organometallic complexes are also used as catalysts in some cases of these processes. Metals or their salts are generally used for the preparation of the catalyst. Catalyst is used to form nanoparticles and nanotubes in the vapor deposition method[30]. In the preparation of carbon nanospheres and carbon nanotubes, hydrocarbons are the most widespread precursors sources of carbon.

Most of these processes are expensive and not suitable for the large-scale industrial production of carbon nanotubes or nanospheres. Therefore, it is needed to find a cost-effective suitable method to prepare carbon nanomaterials. In this study, we have selected a commercial candle for the production of carbon nanoparticles as a low-cost process. A candle is a solid form of fuel that is commonly prepared from wax. Recently, most of the candles are prepared from paraffin. The solid forms of paraffin, called *paraffin wax*, contain molecules from $C_{20}H_{42}$ to $C_{40}H_{82}$. Soy, other plant waxes, beeswax, and tallow as a by-product of beef-fat rendering are also used for the production of different candles. Here, we used candles as a source of carbon to prepare carbon nanoparticles from their incomplete combustion and the prepared particles were characterized by using different advanced spectroscopic methods.

II. Experimental

Materials

A commercial-grade candle, available in the local market, was used as a raw material for the preparation of carbon nanoparticles without further treatment.

Preparation of Carbon Nanoparticles

A candle was used as a lamp. A round bottom glass plate was put over a burning candle lamp with control of air supply[31-32]. Black particles of carbon were produced during the burning of the candle which was accumulated on the inner surface of the glass plate. After 30-40 minutes, produced particles were collected from the glass plate in a container and were kept in a desiccator.

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*Author for Correspondence. e-mail: hossainabul@yahoo.com

Calculation of Percent Yield

A round glass plate was washed with detergent and then acetone so that any impurities can be removed from the surface of the plate. The weight of a candle was taken by an electric balance. The dried round glass plate was carefully placed over the burning candle. Time was monitored with a stopwatch. Stoped the burning after 40 minutes, and the glass was cooled at room temperature. The deposited particles were carefully collected from a round glass plate in an airtight bottle and weighted. The final weight of the residual candle after burning was taken. This gave the burned candle weight. Then the percent of carbon particles yield was calculated.

Characterization of Prepared Particles

Different spectroscopic methods such as scanning electron microscope (SEM), energy-dispersive X-ray (EDX), X-ray diffraction (XRD), Fourier transform infrared (FTIR), and laser-induced breakdown spectroscopy (LIBS) were used to characterize the prepared particles from the candle. The SEM microgram of the prepared sample was obtained by using JSM-6490, JEOL, Japan. The SEM was operated at 20 kV under a vacuum of 10^{-4}-10^{-6} Pa., without any coating. Surface morphology was observed at different magnifications of SEM micrographs. An energy-dispersive x-ray (JED-2300 Analysis Station, JEOL, Japan) spectrum of the prepared sample in SEM was carried out to perform the elemental analysis. The XRD spectrum of the prepared sample was taken by using copper $k_{\alpha 1}$ and $k_{\alpha 2}$ with the wavelength of 1.54056×10^{-10} m and 1.544426×0^{-10} m, respectively, and the scan was performed between 2θ of $10°$ to $45°$ at increments of $0.04°$ with 4 seconds per step. The FT-IR spectrum of the KBr plate of sample material was taken by IR (Prestige 21, Shimadzu, Japan), and recorded transmittance as a function of wavenumber in the range of 4000 to 400 cm^{-1}.

Laser-induced breakdown spectroscopy (LIBS) was used for the more precise elemental analysis of the prepared particles. The small pellets of sample particles were prepared by using a hand press with 80 bars of pressure. A convex lens (100 mm focal length) was used for focusing the fundamental beam at 1064 nm from a Q-switched Nd: YAG laser (Spectra-Physics LAB-170-10) on the sample pellet to produce an intense, transient plasma. A fused quartz lens (50 mm focal length) was used to focus the light emitted from the plasma and collected to the entrance slit (750 mm focal length) computerized Czerny-turner spectrograph (Acton Model SP-2758). An ICCD camera (Unigen II coated Princeton PI-MAX camera with 1024×1024 pixels) was used to detect the spectrum and was transferred to a personal computer, controlled by WinSpec/32 software. The energy of 40 mJ was used as laser pulse. LIBS spectrum experimented in ambient air. The experimental setup and more details of LIBS are schematically presented elsewhere[33-34].

III. Results and Discussion

Preparation of Carbon Nanoparticles

A process of air-controlled incomplete combustion of candle, *paraffin wax*, was used to prepare carbon nanoparticles[32]. In the present work, a candle was directly used for the synthesis of carbon nanoparticles as a very easy method, compared with others methods[22-29].

All waxes are essentially hydrocarbons, (a byproduct of petroleum refining) which means that they are largely composed of hydrogen (H) and carbon (C) atom one. The hydrocarbon composition of the waxes is varying and is a mixture of C_{20} to C_{40} so an exact equation for combustion cannot be given. However, the standard combustion reactions are described elsewhere[31-32]. Thus, the solid carbon particles were prepared by the incomplete combustion of candles in presence of insufficient oxygen from the air and deposited on the glass plate. Other products of carbon dioxide (CO_2), carbon monoxide (CO), and water (H_2O) were entered into the air. The percent yield of carbon nanoparticles prepared from the candle is 4.0.
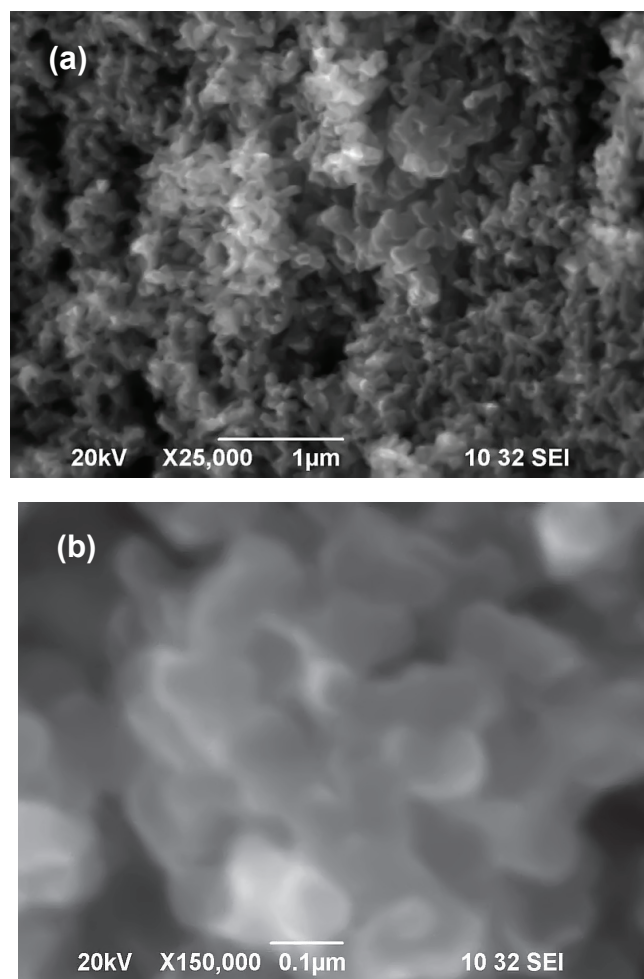

Fig. 1. Scanning electron microscopy micrographs of carbon nanoparticles prepared from the candle at different magnifications and 20 kV voltage.

SEM Characterization

The shape and size of prepared carbon particles from the candle were identified by using a scanning electron microscope (SEM). SEM micrograms of carbon particles at different magnifications of 25,000 and 1,50,000 times are shown in Fig. 1. These figures show that the surface morphology of the carbon particles is different in size. There are several grains of almost sphere particles in a diameter of 10 to 70 nm. But the diameter of most particles are about 20 nm and are mostly nanosphere-like particles.

EDX Analysis

An energy-dispersive X-ray (EDX) spectroscopy was used for the elemental analysis of prepared nanospheres. Fig. 2 shows a strong peak at 0.277 kV, which corresponds to the elemental carbon in the EDX spectrum of the prepared nanospheres. No, another peak was found in the detectable limit (± 0.01 wt%) of the EDX[35]. That meant the prepared nanospheres are carbon particles.

XRD Study

X-ray powder diffractometric (XRD) analysis was performed to identify the crystal structure of carbon nanospheres. Fig. 3 shows the XRD spectrum of carbon nanospheres made from candles in which two intense Bragg diffraction peaks at $2\theta = 25.05°$ and $44.01°$ are corresponding to hexagonal graphite lattice of multi-walled carbon nanoparticles[36]. The peak at $2\theta = 25.05°$ is indexed as (002) plane, which is a relatively high-intensity broad peak which indicates the existence of large amounts of amorphous material[36-39]. The low intense peak at $2\theta = 44.01°$ indexed as (101) plane, is the evidence of the presence of low-quality carbon nanomaterials in the prepared particles[40].
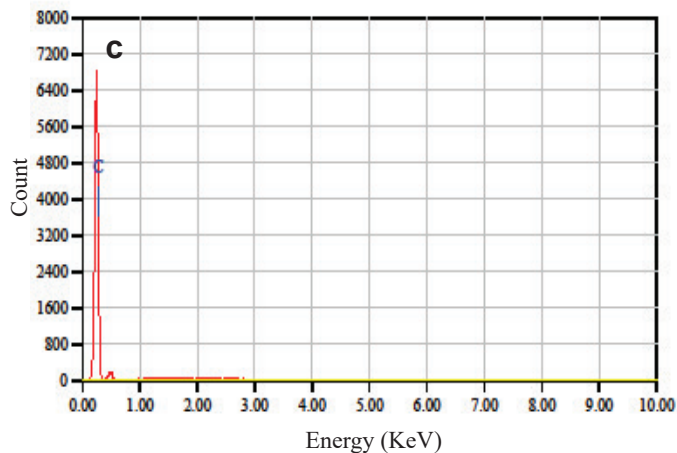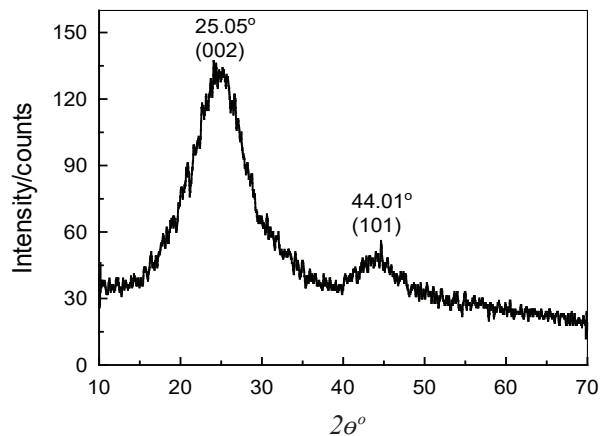
Fig. 3. X-ray powder diffractometric spectrum of carbon nanoparticles prepared from the candle.
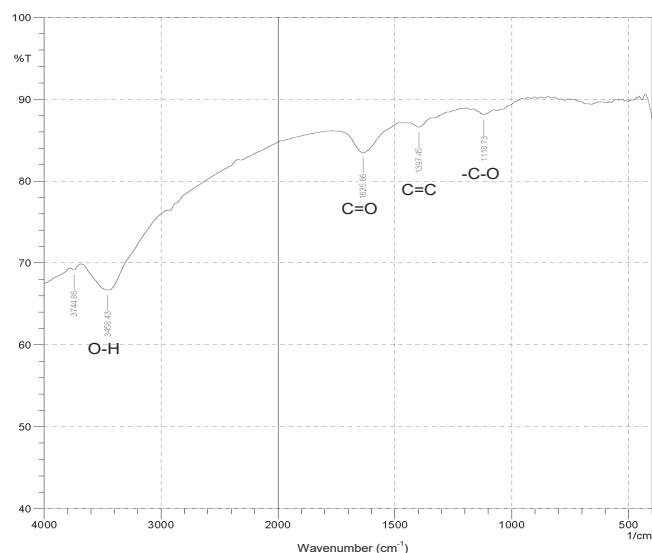
Fig. 4. FT-IR spectrum of carbon nanoparticles prepared from the candle.

FTIR Spectral Analysis

Fourier transform infrared (FTIR) spectroscopic analysis of prepared carbon nanospheres was performed to predict the presence of any hydrocarbon or functional group with elemental carbon during the atmospheric combustion of the candle. The FTIR spectrum of carbon nanospheres is presented in Fig. 4. The broad peak at 3458 cm^{-1} is for the hydrogen-bonded O-H stretching. The small peak at 1635 cm^{-1} is for C=O stretching mode and the peak at 1397 cm^{-1} is for aromatic C=C stretching. Another peak at 1158 cm^{-1} is for -C-O stretching. Such information indicated the presence of trace amounts of oxygen-containing hydrocarbons and aromatic compounds in addition to the elemental carbon of prepared nanospheres. According to the detection limit of FTIR spectroscopy, 35 µg·g^{-1} (0.0035 wt %), this result is more reliable than the EDX one[41].

Fig. 2. Energy dispersive x-ray spectrum of carbon nanoparticles prepared from the candle.

LIBS Elemental Analysis

To overcome the confusion of oxygen is present or not in the prepared nanosphere, laser-induced breakdown (or plasma) spectroscopy (LIBS or LIPS) as a sophisticated advanced elemental analytical technique, was used to analyze the constituents of the prepared nanoparticle from the candle. In the case of LIBS, a high-power laser interacts with a sample (solid, liquid, or gas,) to produce plasma leading to the optical emission spectra[42]. The use of high-quality laser sources and sophisticated optical detectors are the main reason for the powerful analytical performance of LIBS. The unique performance of LIBS is due to the capability of simultaneous multi-elemental analysis over a wide range of wavelengths[43-44].

In the present study, LIBS spectra of prepared nanospheres were taken in the spectral ranges of 188 to 198 nm and 755 to 795 nm for specific identification of carbon and oxygen, respectively. Fig. 5 shows a strong peak at 192.993 nm from the ionic emission line of carbon with respect to relative references peaks (CI 192.77 and C 247.725 nm[45], CI 193.091 and C 247.86 nm)[46-49]. The emission line at 247.8 nm for principal neutral carbon (C) is absent in our spectrum,[32] which is out of our experimental range of wavelength. Fig. 6 shows strong peaks at 777.575 and 795.276 nm from the ionic and atomic emission lines of oxygen with respect of relative references peaks (OI 777.194 and OIt 777.416 nm[50], OI 777.190 and OIt 777.4 nm[46, 51], OI 777.194 and OIt 777.417 nm[52, 53], OI 777.212 and OIt 777.492 nm[45], and atomic oxygen O 794.76 nm[46], where OI is oxygen singlet and OIt is oxygen triplet). No, another peak was observed in the LIBS spectra. From the above observations of the highly sensitive instruments, LEBS having detection limits below 10 μg·g⁻¹ (0.001 wt %)[42] for different elements, revealed that the prepared nanoparticles are carbon material (> 99.99) with tress amount (< 0.01 wt %) of oxygen.
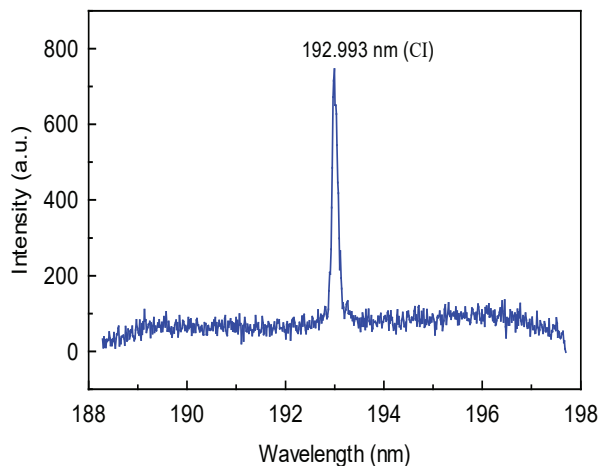

Fig. 5. LIBS spectrum of prepared nanoparticles between 188 and 198 nm with peak for C identified.
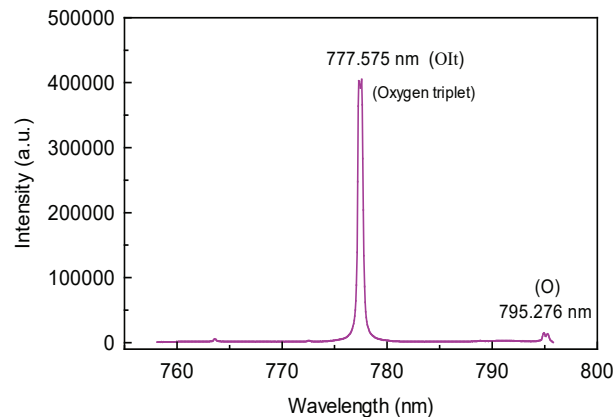

Fig. 6. LIBS spectrum of prepared nanoparticles between 755 and 795 nm with peak for O identified.

IV. Conclusion

Preparation of carbon nanoparticles is performed successfully from incomplete combustion of the commercial candle as a very cheap and easy method. The SEM study confirmed the formation of spherical carbon nanoparticles in the diameter of 10 to 70 nm. But most of the carbon nanospheres' size is about 20 nm in diameter. The X-ray diffraction pattern suggested that the prepared carbon nanospheres are mostly amorphous carbon material associated with a trace amount of hexagonal crystal lattice of graphite. Comparing the detection limits of EDX, FTIR, and LIBS, and their spectra of prepared nanosphere, it could be concluded that the composition of prepared nanoparticles is > 99.99 % carbon and < 0.01 % oxygen by weight.

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