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Morphological Characterization of Soot From the Combustion of Candle Wax

David Ntaote Shooto, Ezekiel Dixon Dikio[*]

Department of Chemistry, Vaal University of Technology, P. O. Box X021, Vanderbijlpark, South Africa.
[*]E-mail: ezekield@vut.ac.za.

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Candle wax has been used as a precursor for the production of carbon nanomaterial without a catalyst precursor. Nanomaterials formed in the process were analysed by Raman spectroscopy, scanning electron microscope (SEM), energy dispersive spectroscopy (EDS), high resolution transmission electron microscope (HR-TEM) and X-ray diffraction (XRD). Carbon nanomaterial produced from candle wax flame soot show the morphology of carbon nanospheres. The results obtained are presented.

Keywords: Carbon nanospheres, candle, wax, soot

1. INTRODUCTION

Candle is generally composed of paraffin which is made of heavy straight-chain hydrocarbons obtained from crude petroleum oil [1,2]. Candles have been used since ancient times as a source of light and in various indoor environments [3].

Michael Faraday in 1860 [3,4] described the mechanism of the combustion taking place in a candle flame as a diffusion flame with wax serving as fuel and the wick as transport mode of the fuel by capillary forces. Crude oil based crystalline paraffin waxes are the most important waxes for industrial application. They are solid and crystalline mixtures of hydrocarbons consisting of linear *n*-alkane and branched *iso*- and *cyclo*-alkanes with carbon lengths ranging from C_{16} – C_{30} and higher [5-7]. Candle flames present a simple example of a diffusion flame, its burning is still complicated by the nature of its finite cylindrical wick [4].

A health problem related to candle burning is the occurrence and release of metal additives from the wick. Studies of particle emission from a steady burning single paraffin wax candle by Li and

Hopke [8] and other researchers, found the size of particles produced to be around 30 nm in diameter [3,8]. Afshari *et al* [9] measured the concentrations of particles emitted from a pure wax candle and a scented candle in a chamber and found the concentrations of particles to be 240 000 particles cm^{-3} and 69 000 particles cm^{-3} respectively [3,10]. The particles of matter emitted from candle wax flames, which are hydrocarbons in origin, are soot particles. These soot particles result from competition between soot formation and oxidation. Soot particles smaller than 300 nm are known to have a negative impact on health due to their physico-chemical properties. Studies aimed at providing information on soot characteristics, soot volume fraction and soot morphology have been carried out by several researchers [11-13].

The objective of this study is to explore the nature and morphology, i.e. primary particle diameter and aggregate size, of carbonaceous soot produced from the combustion of candle wax flame from a chemistry view-point using modern analytical techniques such as scanning electron microscope, (SEM), X-ray diffraction, (XRD), and Raman spectroscopy, Energy dispersive spectroscopy (EDS) and transmission electron microscopy (TEM).

2. EXPERIMENTAL

2.1. Materials

Candle wax used in this study were purchased from open shop in Vanderbijlpark, Republic of South Africa and used without further purification.

2.2. Preparation of nanomaterials

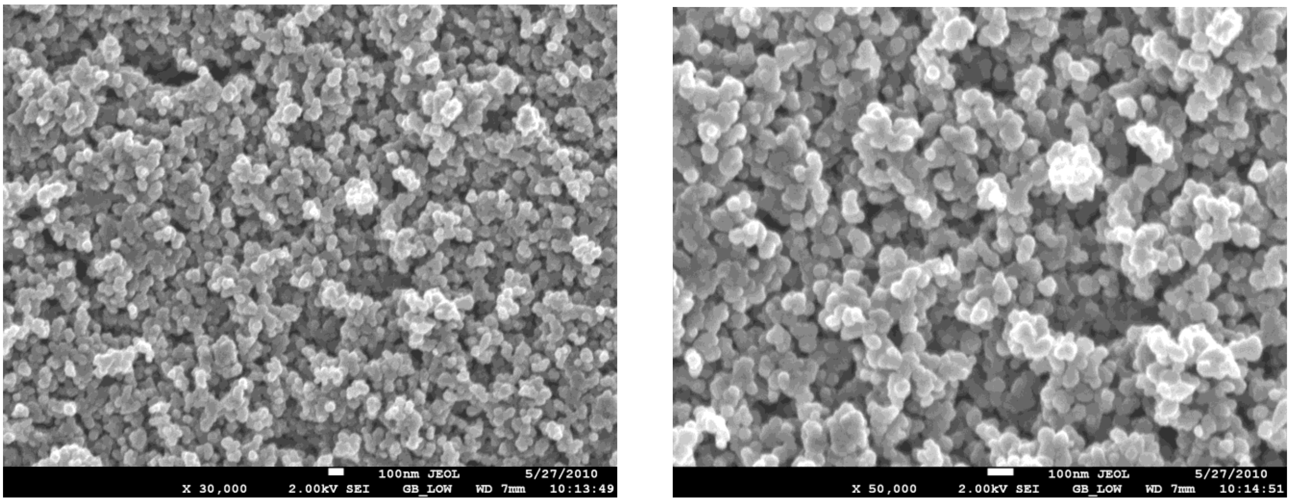
Candle wax was placed in a simple laboratory fume cupboard. The candle was lighted with a match and allowed to burn. A flat ceramic tile plate was placed above the flame of the candle to collect soot emitted from the candle flame. When an amount of soot approximately equal to 5 - 10 g was collected, the experiment was terminated.

2.3. Characterization

The morphological features of nanomaterials were analyzed by Raman spectroscopy, FE-SEM, HR-TEM, EDS, and XRD. The Raman spectra were obtained by a Raman spectroscope, Jobin-Yvon HR800 UV-VIS-NIR Raman spectrometer equipped with an Olympus BX 40 attachment. The excitation wavelength was 514.5 nm with an energy setting of 1.2 mV from a coherent Innova model 308 argon-ion laser. The Raman spectra were collected by means of back scattering geometry with an acquisition time of 50 seconds. The surface morphology and EDS measurements were recorded with a JEOL 7500F Field Emission scanning electron microscope. The HR-TEM images of the sample were obtained by a CM 200 electron microscope operated at 100 kV. Powder X-ray diffraction (PXRD) patterns were collected with a Bruker AXS D8 Advanced diffractometer operated at 45 kV and 40 mA with monochromated copper Kα1 radiation of wavelength (λ = 1.540598) and Kα2 radiation of wavelength (λ = 1.544426). Scan speed of 1 s/step and a step size of 0.03^{o}.

3. RESULTS AND DISCUSSION

Burning of candle wax flame to obtain soot is a thermal decomposition process in which the candle wax flame breaks up to form other substances. The air borne thermolytic particles are extremely small and occur individually. Some particles are seen to form aggregates on the ceramic collector plate. Particles obtained from the atmospheric combustion of candle wax flame are a complex mixture of elemental carbon and a variety of hydrocarbons.



a) **b)**

Figure 1. Scanning electron micrograph (FE-SEM) of soot obtained from the combustion of candle wax.

The FE-SEM micrograph of candle wax flame thermolytic carbon nanomaterial is presented in figure 1(a–b). The surface morphology of the carbon deposit obtained is seen to be non-uniform. There are several grains of nanomaterial in the micrograph. The candle wax flame soot particles are extremely small with a majority of the particles about 0.3 μm in diameter. The FE-SEM image of soot particles at 100 nm and 1 μm show particles of carbon which are agglomerations of particles as shown in figure 1a. A higher magnification of the FE-SEM image, figure 1(b), show the particles of matter as irregular particles all joined together. The FE-SEM image does not show any carbon nanotubes and the soot particles could not be classified as nanospheres.

Energy dispersive spectroscopy (EDS) of candle wax flame soot is presented in figure 2. The spectra show the presence of carbon and oxygen as the combustion product of candle wax flame. The composition of the soot aggregates from the EDS analysis indicates the soot to consist of about 27.29% weight carbon and 72.71% weight of oxygen. The result shows the product of the thermal decomposition of candle wax flame to be composed mainly of carbon and oxygen, figure 2 and table 1. Candle wax are produced with metal additives such as copper, tin, or the heavy metal lead, [14] which are released during combustion. These metal additives from the wick are known to cause health problems. The Energy Dispersive Spectroscopy (EDS) of the soot particles indicate the presence of no other metal except carbon and oxygen as recorded in the spectra.

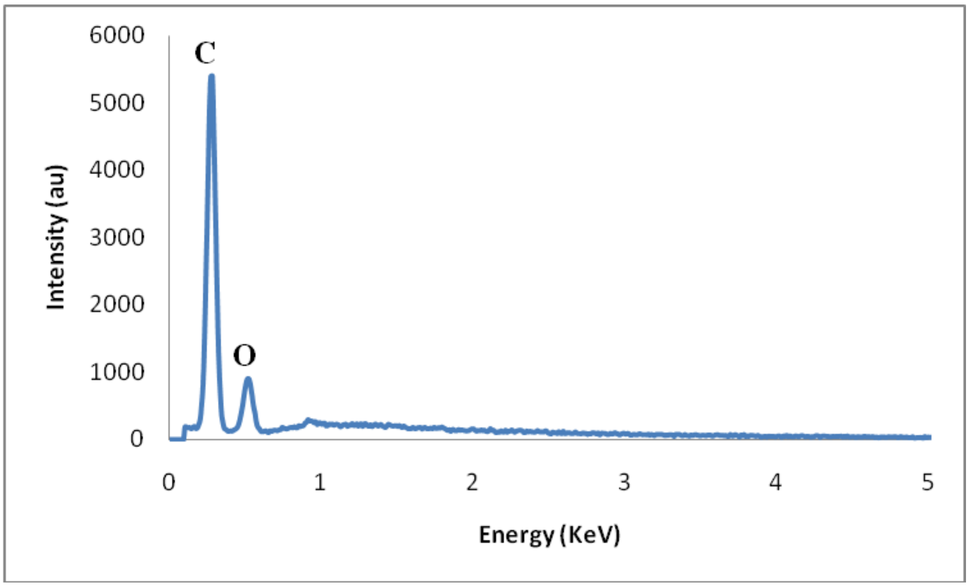


Figure 2. Energy dispersive Spectroscope (EDS) spectra of soot obtained from the combustion of candle wax.

Table 1. Table of energy dispersive spectroscopy of candle wax soot

Element	Element (% weight)	Atom (%)	Peak Height (au)
Carbon (C)	27.29	33.33	5396
Oxygen (O)	72.71	66.67	817
Total	100.0	100.1	

The Raman spectrum of the candle wax flame soot is presented in figure 3. The Raman spectra show two major bands at 1299.46 cm^{-1} and 1571.44 cm^{-1}. These bands are the *D* and *G* bands which indicate the presence of crystalline graphitic carbon in synthesized carbon nanotubes. The *D* band at 1299.46 cm^{-1} has been attributed to the presence of amorphous carbon [15-17] and surface defects in carbon nanotubes. This band which should resonate at about 1350 cm^{-1} in the Raman spectra of carbon nanotubes, is seen to resonate at 1299 cm^{-1}, a shift to lower wavelengths. The *G* band at 1571.44 cm^{-1} correspond to an E_{2g} mode of graphite which is related to the vibration of sp^2-bonded carbon atoms and the presence of ordered carbon nanotubes in a sample. The presence of this band in this Raman spectrum is an indication of the soot samples being composed of carbon nanomaterial as either nanotubes or nanospheres. The band at 1299.46 cm^{-1} of moderate intensity is associated with vibrations of carbon atoms in disordered graphite. The *D* and *G* bands should produce an overtone which should resonate at about 2600 cm^{-1}.

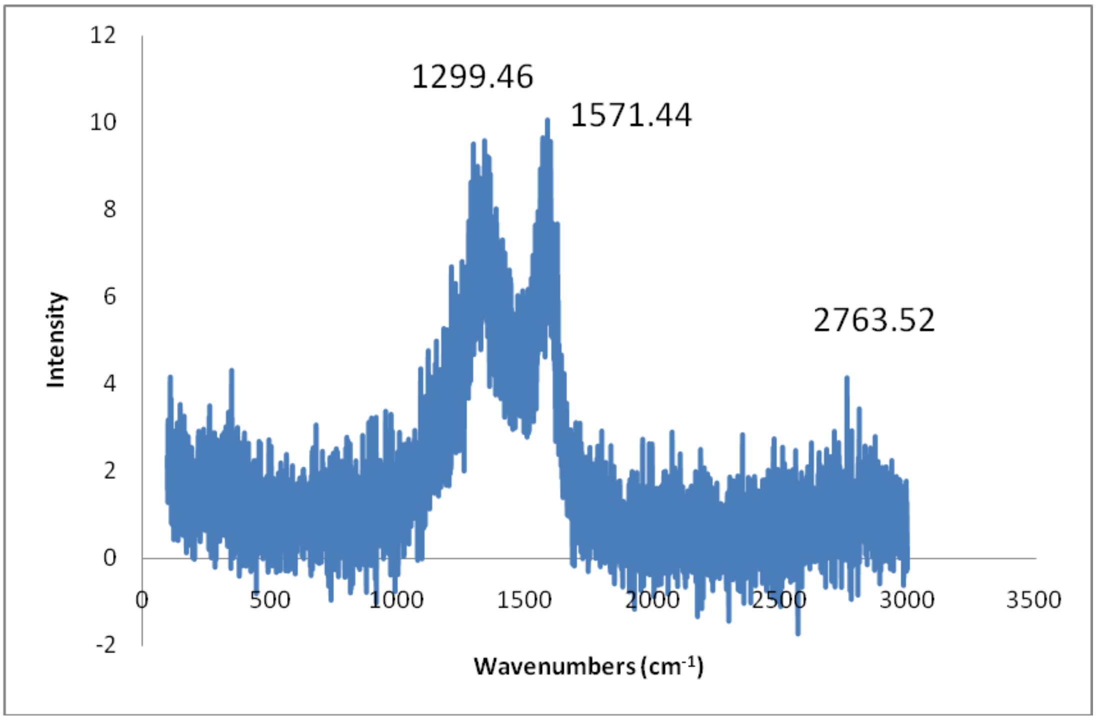


Figure 3. Raman spectra of soot obtained from the atmospheric combustion of candle wax.

A resonance peak at 2763.52 cm^{-1} is observed, a shift to higher wavelengths, indicating the absence of carbon nanotubes as could be observed in the FE-SEM micrograph. The presence of this resonance peak in the Raman spectra of carbon nanotubes (carbon nanotubes) synthesis could be related to the amount of carbon nanotubes produced in the synthesis as well as their purity. The absence of this resonance peak in the soot obtained from candle wax flame combustion is an indication that the candle wax flame soot had formed impure as well as few carbon nanotubes in the process. The intensity ratio of these two bands ($I_D/I_G = 0.9442$) is also considered as a parameter to characterize the quality of CNTs in samples under investigation. A high intensity ratio would indicate a higher degree of disorder in the CNTs. The intensity ratio for the two bands obtained, 0.9442, shows the nanomaterial formed in the candle wax soot to be more of amorphous carbon. The intensity of the two peaks are quite high indicating that there exits two dimensional disorder in the carbon nanomaterial produced from this thermolytic process.

The HR-TEM micrograph of the candle wax soot is presented in figure 4. HR-TEM micrograph of the carbonaceous soot at 100 and 200 nm show nanomaterials obtained from candle wax soot as spherical individual particles. Since no catalyst was used in the production of nanomaterials from the candle wax, catalyst particle are absent from this HR-TEM micrograph. Morphological details indicating that the nanomaterials obtained from the combustion of candle wax as nanospheres, observed in the Raman spectral analysis, is further corroborated in the HR-TEM micrograph [18].

Several nanospheres have agglomerated together in the HR-TEM micrograph, figure 4 (a-b). Single nanoparticles of soot indicating the nanospheres to be uniform could also be observed in the micrograph. The HR-TEM micrograph, figure 4 (c-d) reveal lattice fringe images of graphene layers of individual nanospheres. Two spheres drawn over two soot particles, figure 3d are shown. The crystalline presentation of the layers reflects the graphitization of the nanospheres.

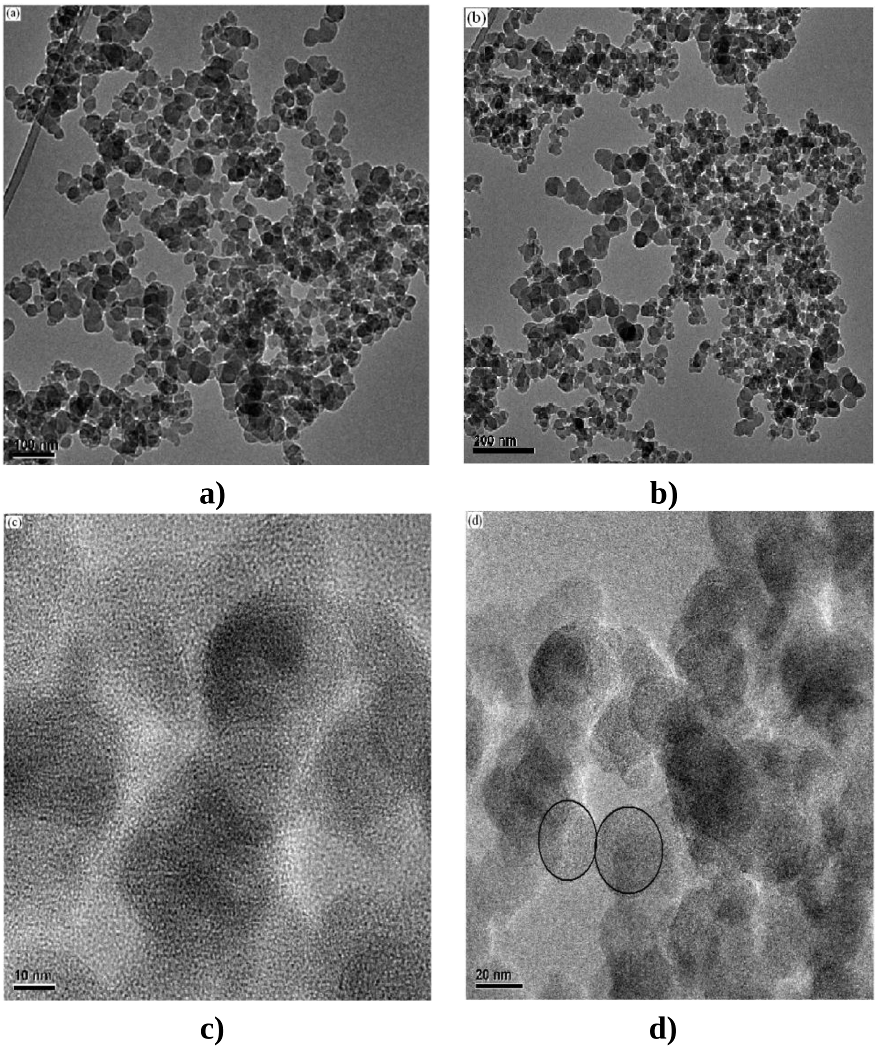


Figure 4. Transmission electron micrograph (HR-TEM) of soot obtained from the combustion of candle wax.

The X-ray diffraction (XRD) pattern of candle wax flame soot is shown in figure 5. The carbonaceous soot obtained was used directly. The Bragg diffraction peaks at $2\theta = 23.24^o$, 41.30^o, 81.30^o and 115.10^o, are the only peaks obtained in the X-ray diffraction analysis. The peaks $2\theta = 23.24^o$ and 41.30^o correspond to hexagonal graphite lattice of multi-walled carbon nanotubes [19-21]. The peaks at $2\theta = 23.24^o$ is a moderately high intensity broad peak which indicates the presence of large amounts of amorphous carbon material in association with nanotubes. The low intensity of the peak at $2\theta = 41.30^o$, is an indication of the low quality of carbon nanomaterial present in the soot.

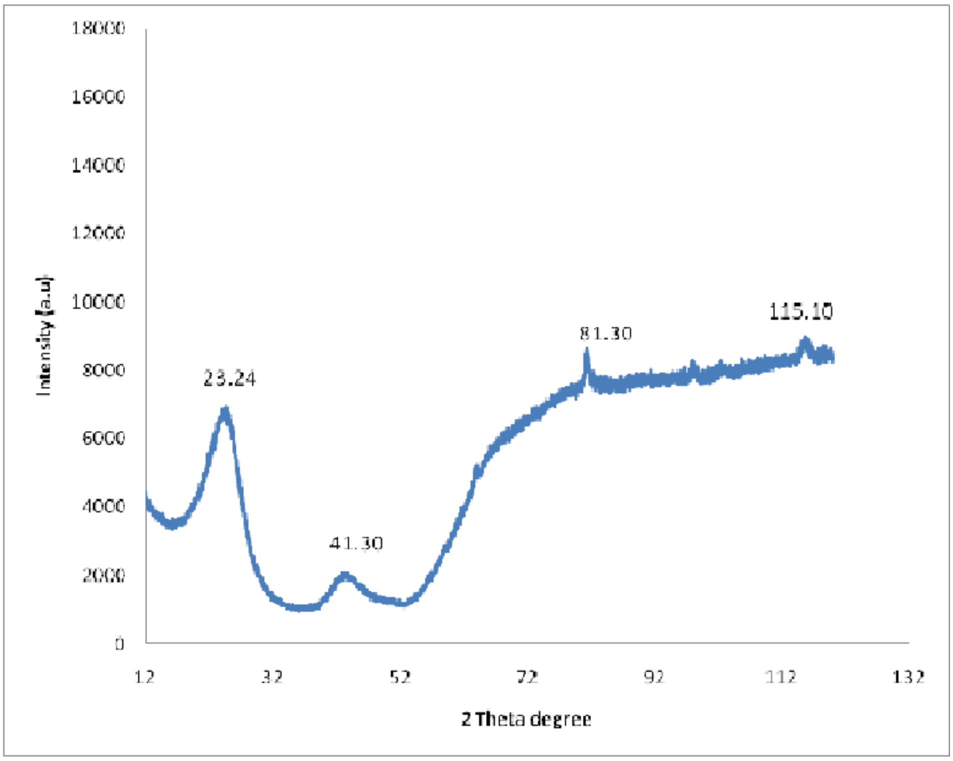


Figure 5. X-ray diffraction spectra of soot obtained from the atmospheric combustion of candle wax.

4. CONCLUSION

Carbonaceous soot produced from candle wax flame without a catalyst precursor show the presence of significant amount of carbon nanomaterial. The HR-TEM micrographs provide a clear indication that these nanoparticles are carbon nanospheres. Raman spectroscopy and X-ray diffraction investigation shows the presence of carbon nanotubes in association with amorphous nanomaterial due to the presence of the *D* and *G* bands found in carbon nanotubes. EDS analysis of candle wax flame soot provide strong evidence of soot particles to be composed primarily of carbon and oxygen. No other metals were observed in the EDS spectra.

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