Asian Journal of Chemistry; Vol. 26, No. 15 (2014), 4553-4556

ASIAN JOURNAL OF CHEMISTRY

http://dx.doi.org/10.14233/ajchem.2014.15150

Characterization of Nano-Crystalline Carbon from Camphor and Diesel by X-ray Diffraction Technique

B. MANOJ

Department of Physics, Christ University, Bangalore-560 029, India

Corresponding author: E-mail: manoj.b@christuniversity.in

Received: 26 July 2013; *Accepted*: 6 December 2013; *Published online*: 16 July 2014; AJC-15523

Hydrocarbons are by far the most widespread precursors among carbon sources employed in the production of carbon nanotubes and carbon nanosphers. In the present study, diesel and camphor have been used as precursors for nanomaterials. Carbonaceous soot produced from combustion of diesel in engine shows the presence of significant amount of carbon nanomaterials. The γ band at about 19.28° has been attributed to the presence of amorphous carbon and surface defects in carbon nanotubes. The γ band at about 25.81° corresponds to e2g mode of graphite which is related to vibration of sp^2 bonded carbon atoms and the presence of ordered carbon nanotubes in diesel soot. The SEM micrographs provide a clear indication that nanoparticle formed in diesel soot are clusters of carbon nanospheres. Energy dispersive spectrum analysis of diesel soot confirms that the soot particles to be composed of primarily carbon and oxygen along with hydrogen. The camphor soot shows γ and π bands which reveals the presence of crystalline graphitic carbon. The SEM micrographs of camphor show the presence of carbon nanostructures. It is found nanomaterials formed in the diesel soot consists more of disordered carbon, whereas in camphor it is more of ordered graphite like carbon.

Keywords: Hydrocarbon, carbon nanospheres, X-ray diffraction, Camphor.

INTRODUCTION

Allotropes of carbon have been a subject of great interest among researchers for several years. Amorphous carbon is the name used for coal, soot and other impure forms of the element. These have neither graphite nor diamond like structures. Amorphous carbon has a wide range of properties that are primarily controlled by the different bond hybridizations possible in such materials. Hydrocarbons as one of the major sources for nanomaterials was reported by many authors[1-5].

In the present study, structural characterisation of diesel soot and camphor soot has been done using different analytical methods like X-ray diffraction (XRD), Scanning electron microscopy (SEM) and Energy dispersive spectroscopy (EDS). X-ray scattering is used to obtain structural information on the atomic scale, especially the average carbon-carbon distance and the average coordination number of the carbon atoms. Scanning electron microscopy study reveals information such as external morphology, chemical composition and orientation of materials making up the sample. With these analytical methods one could determine the structural parameters like aromaticity f_a, lateral size L_a, stacking height L_c and number of carbon per aromatic lamellae[1-6].

EXPERIMENTAL

The morphological features of nanomaterials were analyzed by SEM-EDS and XRD. The surface morphology and energy dispersive spectrum measurements were obtained by scanning electron microscope model JSM 6390 from JEOL Company in Japan. The XRD measurements were carried out using a Bruker AXS D8 Advance X-ray diffractometer. Diesel soot was collected from the engine of diesel vehicle was leached with acetone (DSA). The camphor soot was prepared by the thermal decomposition of camphor in open air. The collected soot was leached with acetone and washed with enormous amount of water. The air dried samples were analyzed for the nanostructure.

RESULTS AND DISCUSSION

Characterization of amorphous carbon from diesel soot: Burning of diesel to obtain soot is a thermal decomposition process in which diesel breaks up to form other substance. The formation and oxidation processes of soot particles are strongly influenced by the engine operating conditions and by the combustion system design. The engine borne thermolytic particles are extremely small and occur individually. Particles obtained from the combustion of diesel are a complex mixture of elemental carbon, nitrogen, sulphur and hydrogen.

The SEM micrograph of diesel thermolytic carbon nanomaterials is presented in Fig. 1. The surface morphology of the carbon deposit obtained is seen to be non-uniform. There are several grains which look like carbon nanospheres. These

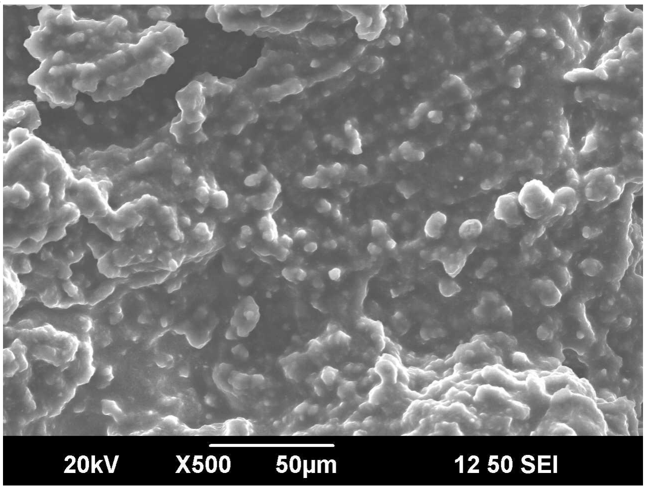

Fig. 1. SEM data of diesel soot treated with acetone

sphere join together to form chains of spheres. This chain like structure is seen throughout the surface. The particle size is measured to be ~109.72 and ~148.28 nm.

Energy dispersive spectrum (EDS) of diesel soot is presented in Fig. 2. The spectra show the presence of carbon and oxygen as the combustion product diesel along with sulphur, traces of calcium and iron in the studied surface. The soot consists of about 94.81 wt % carbon and 0.78 wt % oxygen. Hydrogen is absent in the studied sample.

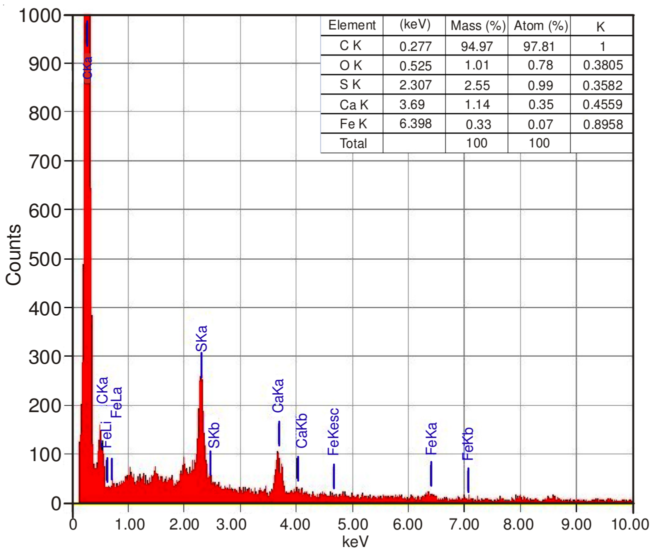

Fig. 2. Energy dispersive spectrum data of diesel soot treated with acetone

Characterization of amorphous carbon from camphor soot: Camphor exhibits highest CNT-production efficiency with minimal waste production. Moreover every atom of camphor plays a positive role in carbon nanotube synthesis, which accounts for maximum carbon-to-CNT conversion efficiency which is reported by many authors earlier[4]. Camphor, a botanical hydrocarbon, on burning undergoes hermal decomposition to give soot. Atmospherically burned camphor produces thermolytic particles which are extremely small. The soot formed is extremely fine powder, which consists of carbon, oxygen, aluminium, silicon and potassium. The EDS analyses (Fig. 3) revealed 93.29 % carbon, 3.19 % oxygen, 0.63 %

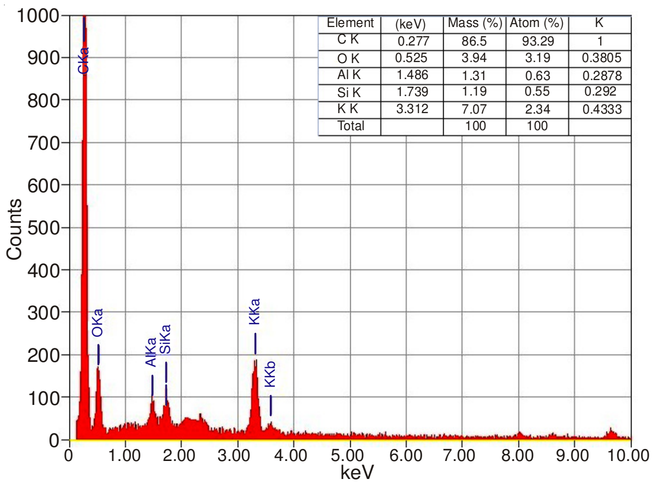

Fig. 3. Energy dispersive spectrum data of camphor

aluminium, 0.55 % silicon and 2.34 % potassium in the soot formed. Unlike the diesel soot, the presence of aluminium and silicon are reported in the sample.

The SEM micrograph of nanomaterials formed from camphor is presented in Fig. 4. The surface morphology of the carbon deposit is non uniform. The SEM images revealed that particles of carbon are in the form of spheres. The size of the particle formed is about 70-90 nm diameter.

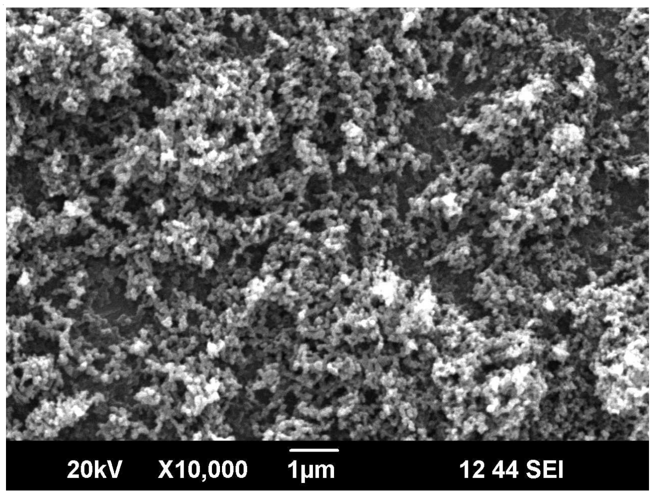

Fig. 4. SEM data of camphor soot

Comparative study of diesel and camphor soot: A comparative study of the XRD profiles of the two kinds of soot along with pure graphite is shown in Fig. 5. From the X-ray diffraction profile, it is found that diesel soot is having more disordered carbon in it compared to camphor soot. The interlayer spacing d_{002} is found to be 3.45 Å for diesel soot, which is very near to the d- spacing of pure graphite[8]. From the SEM analysis, it is observed that camphor soot contains more of ordered nanospheres while diesel soot contains more of nanoclusters with more disordered carbon in it. The ratio of intensity of disorder carbon to ordered carbon (I_{20}/I_{26}) is given in Table-1. A higher intensity ratio would indicate a higher degree of disorder in the carbon nanotubes. The intensity ratio for the two bands treated with acetone is observed to be 1.83. This value indicate that comparatively low percentage

TABLE-1
STRUCTURAL PARAMETERS OF DIESEL SOOT TREATED WITH ACETONE (DSA) AND CAMPHOR SOOT (CA)

Sample	I_{26}/I_{20}	I_{20}/I_{26}	f_a	L_a (Å)	L_c (A^0)	d_{002} (Å)	N	n
DSA	0.55	1.83	0.16	117.94	81.45	3.45	24.63	194.12
CA	6.30	0.16	0.91	89.13	23.39	3.57	7.55	18.24

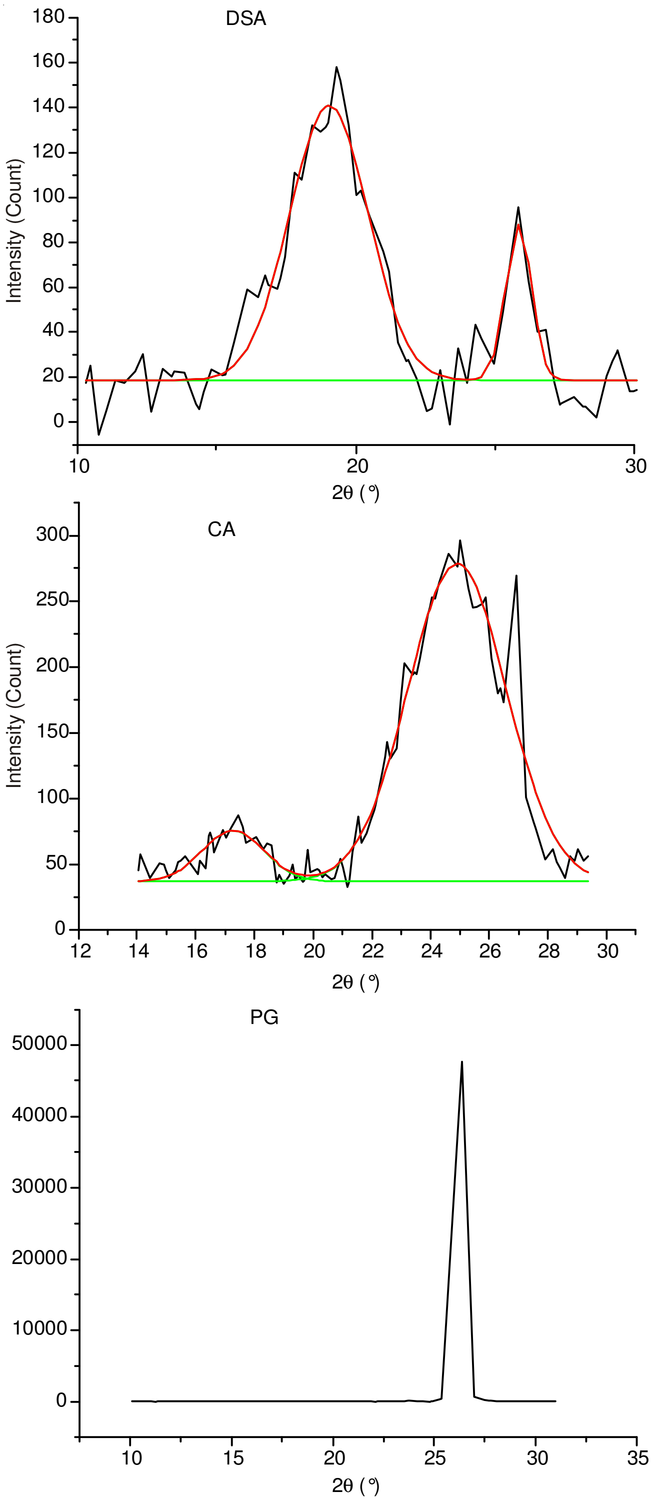

Fig. 5. XRD data analysis of diesel soot treated with acetone (DSA), camphor soot (CA) and pure graphite (PG)

of carbon nanotubes are formed in the diesel soot and higher disorder in the sample[4].

The lateral size of the aromatic lamina (La) formed is found to be 11.7 nm where as stacking height (Lc) is found to be 8.1 nm for the diesel soot. The intensity ratio of the two peaks (I_γ/I_π) for camphor soot is found to be 0.158. This value indicates that comparatively higher quality carbon nanotubes are formed in camphor soot which also implies highly ordered structure[4]. The nanospheres formed during the thermal decomposition of camphor are of better quality than that formed from diesel. It is observed that as the aromaticity increases, the I_{20}/ I_{26} decreases, which indicates lower degree of disorder in the *sp*[2] hybridized carbon. In the case of camphor soot, the lateral size of the aromatic lamina (La) and stacking height (Lc) formed is found to be 8.91 nm and 2.34 nm, respectively. The d_{002}-spacing of the lamellae is found to be 3.57 Å which is close to the value of graphite layer reported in various studies[8].

In the present study all the samples show strong and broadened π bond, suggest that the carbon nanosphers (CNS) formed is composed of crystalline graphitic carbon. Higher intensity of this band indicates higher degree of crystallinity/ graphitization. The nanostructure growth takes place by adding up of hexagonal and pentagonal rings as building blocks, not individual carbon atom. These radicals are in highly active state and capable of forming a stable structure among them. The temperature generated during the combustion of diesel/ camphor plays a crucial role in the formation of graphene layers. The retentention time of the *sp*[2] carbon atoms in the reaction core is not long enough to form multi-layer graphene (graphite). Instead, only few-layer graphene is kinetically favored. Many author's reported the presence of peak at ~42.33° to hexagonal graphite lattice of multi-walled carbon nanotubes[1-8]. Very weak peak at 42.33° observed in the present study is an indication formation carbon nanomaterial in the soot. Also aromaticity is very high for the nanomaterials formed from camphor (0.91) compared to diesel (0.16). Camphor being a natural hydrocarbon will be a better precursor for the production of nanomaterials.

Conclusion

Carbonaceous soot produced from combustion of diesel in engine show the presence of significant amount of carbon nanomaterials. The γ band at about 19.28° has been attributed to the presence of amorphous carbon and surface defects in carbon nanotubes. This band is associated with aliphatic side chains in the disordered carbon atom. The π band at ~25.81° corresponds to e2g mode of graphite which is related to vibration of *sp*[2] bonded carbon atoms and the presence of ordered carbon nanotubes in diesel soot. The SEM micrographs provide a clear indication that nanoparticle present in diesel soot are clusters of carbon nanospheres. Energy dispersive spectrum analysis of diesel soot indicates that the soot particles to be composed of primarily carbon and oxygen along with hydrogen. The camphor soot shows presence of γ and π bands, which indicates the presence of crystalline graphitic carbon. The SEM micrographs of camphor show the presence of carbon nanostructures. The EDS analysis shows more of carbon and oxygen along with aluminium, silicon and potassium. The nanomaterials formed in the diesel soot consists more of disordered carbon, whereas in camphor it is more of ordered graphite like carbon.

REFERENCES

1. H. Darmstadt, C. Roy, S. Kaliaguine, G. Xu, M. Auger, A. Tuel and V. Ramaswamy, *Carbon*, **38**, 1279 (2000).
2. T. Ungar, J. Gubicza, G. Ribárik, C. Pantea and T.W. Zerda, *Carbon*, **40**, 929 (2002).
3. M. Kumar and Y. Ando, *Diamond Rel. Mater.*, **12**, 1845 (2003).
4. A. Ilyin, N. Guseinov, A. Nikitin and I. Tsyganov, *Physica E*, **42**, 2078 (2010).
5. N. Mohan Anu and B. Manoj, *Int. J. Electrochem. Sci*., **7**, 9537 (2012).
6. H. Takagi, K. Maruyama, N. Yoshizawa, Y. Yamada and Y. Sato, *Fuel*, 83, 2427 (2004).
7. B. Manoj and A.G. Kunjomana, *Int. J. Miner. Metull. Mater*., **19**, 279 (2012).
8. B. Manoj and A.G. Kunjomana, *Int. J. Electrochem. Sci*., **7**, 3127 (2012).