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# Structural Characterization of Paraffin Wax Soot and Carbon Black by XRD†

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From past few decades, an exponential increase in the research related to carbon nanomaterials and their excellent applications has been witnessed. Realizing the need for new potential precursors and cost effective production methods, we have investigated two precursors-paraffin wax soot (CS) and carbon black (CB). Structural and morphological features of the samples are analyzed by various techniques such as X-ray diffraction, high resolution scanning electron microscopy and electron dispersive spectroscopy. The lateral size of the aromatic lamellae, stacking height, the average spacing of the (002) crystallographic planes ($d_{002}$) and aromaticity are found to be 15.12 Å, 44.30Å, 3.57 Å, 0.912 and 15.26 Å, 43.23 Å, 3.68 Å, 0.986 respectively for paraffin wax soot and carbon black. Very low γ and π band intensity ratio shows a low amount of disorder in the samples. SEM micrographs of the samples reveal non-uniform carbon nanospheres of particle sizes 26-94 nm.

**Key Words: Nanocrystalline structure, Paraffin Wax Soot, Carbon Black, Stacking height, XRD.**

## INTRODUCTION

Carbon and its various allotropic forms is a blooming and extensively investigated field for the past few decades. The revolution which started, with the discovery of fullerenes in 1985 continues with the newly discovered wonder material graphene and have never failed to amass the interest of scientific community. After all these years it still stays as a hot topic of research is primarily because of their unique physical as well as chemical properties. Due to this reason, they are suitable for a whole host of applications ranging from thin film technology to nano-medicine. But, the production cost of these wonder materials is an issue which shadows the glory and hence it is essential to find out efficient and cost effective sources for these materials. In this study, we have presented two hydrocarbons-paraffin wax soot and carbon black-as the potential precursors of the carbon nanomaterials.

Soot refers to carbon particles formed from incomplete combustion of hydrocarbons. Paraffin wax is mainly composed of mixture of alkanes with the number of carbon atoms that falls within 20-40 range[1-6]. Carbon black is virtually pure carbon in the form of colloidal particles resulting from the incomplete thermal decomposition of hydrocarbons. Different analytical methods used for the study of various structural parameters of both the samples include X-ray diffraction, scanning electron microscopy and energy dispersive spectroscopy.

## EXPERIMENTAL

The hydrocarbons selected for this study are paraffin wax and carbon black. The paraffin wax is made to undergo thermal decomposition in the open air and the experiment is terminated when sufficient amount of the sample got accumulated on the horizontally placed clean ceramic tile above the experimental set up. The second sample is made from the finely grinded carbon black.

**Structural analysis:** Structural characterization has been done using different analytical method like XRD, SEM and EDS. SEM micrographs and EDS measurements are obtained using Model JSM 6390 from JOEL Company, Japan and a Bruker AXS D8 Advance X-Ray Diffractometer is employed for obtaining the XRD profile of the samples. The aromaticity ($f_a$), lateral size ($L_a$), stacking height ($L_c$) and number of carbon atoms per aromatic lamellae are determined using Scherrer equations from the XRD data[7-9].

## RESULTS AND DISCUSSION

**XRD analysis:** The XRD profile of the carbon black (CB) and paraffin wax soot (CS) are presented in the Fig.1. The Bragg diffraction peaks at 2θ *ca.* 24° and *ca.* 42° are the only notable peaks obtained in the XRD spectra. The former peak is referred to as π-band and is due to the presence of aromatic ring structure and the latter one reveals the presence of $sp^2$

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hybridized hexagonal graphite-like carbon lattice. The calculation of mean crystallite sizes using Scherrer equations reveals that $L_a$ is 44.30Å and 43.23 Å respectively for paraffin wax soot (CS) and carbon black (CB). $L_c$ values for paraffin wax soot and carbon black are calculated as 15.12 Å and 15.26 Å respectively (Table-1). The relatively low intensity of the π-band observed in XRD profiles of the samples suggests highly ordered crystalline structures. The intensity ratio of the γ and π bands ($I_\gamma$/ $I_\pi$ also known as the D/G intensity ratios) from XRD analysis are determined to be 0.220 (CS) and 0.431 (CB), which indicates the high quality and order of the nanostructure formed. The $d_{002}$ spacing is found to be 3.57 Å for paraffin wax soot and 3.68 Å for carbon black which is very near to the reported value of graphite. The aromaticity of the sample paraffin wax soot is found to be 0.91 and that of carbon black is calculated as 0.98.

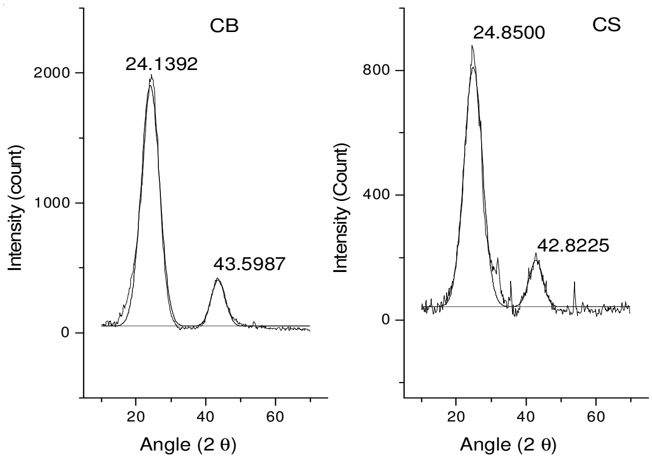


Fig. 1. XRD profile of carbon black (CB) and paraffin wax soot (CS)

TABLE-1
STRUCTURAL PARAMETERS OF PARAFFIN WAX SOOT (CS) AND CARBON BLACK (CB) CALCULATED FROM XRD DATA

| Sample | G band 2θ (°) | Ratio $I_D/I_G$ | $d_{002}$ (Å) | $L_c$ (Å) | $L_a$ (Å) | N | n |
|---|---|---|---|---|---|---|---|
| CS | 24.8 | 0.22 | 3.57 | 15.12 | 44.30 | 5 | 9 |
| CB | 24.1 | 0.431 | 3.68 | 15.26 | 43.23 | 5 | 8 |

**SEM and EDS analysis:** The SEM micrograph of carbon black and paraffin wax thermolytic carbon nanomaterials is presented in Fig. 2. The surface morphology of the carbon deposit obtained is seen to be non-uniform. There are several grains which look like carbon nanospheres. The SEM micrographs shows the presence of non-uniform carbon nanospheres having particle sizes within the range of 48-94 nm (for paraffin wax soot) and 26-61 nm (for carbon black).

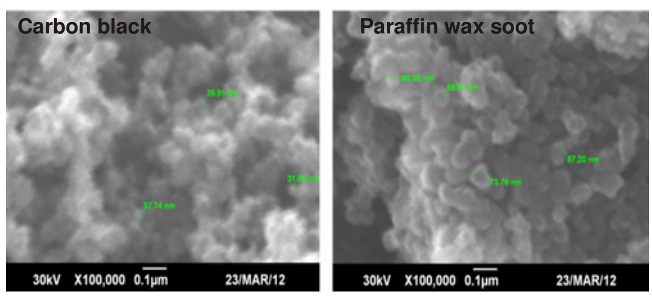


Fig. 2. SEM micrographs of paraffin wax soot and carbon black

Energy dispersive spectrum of paraffin wax soot and carbon black are presented in Fig. 3. EDS analysis of both paraffin wax soot and carbon black shows a carbon content above 96 % along with the traces of oxygen and sulphur.

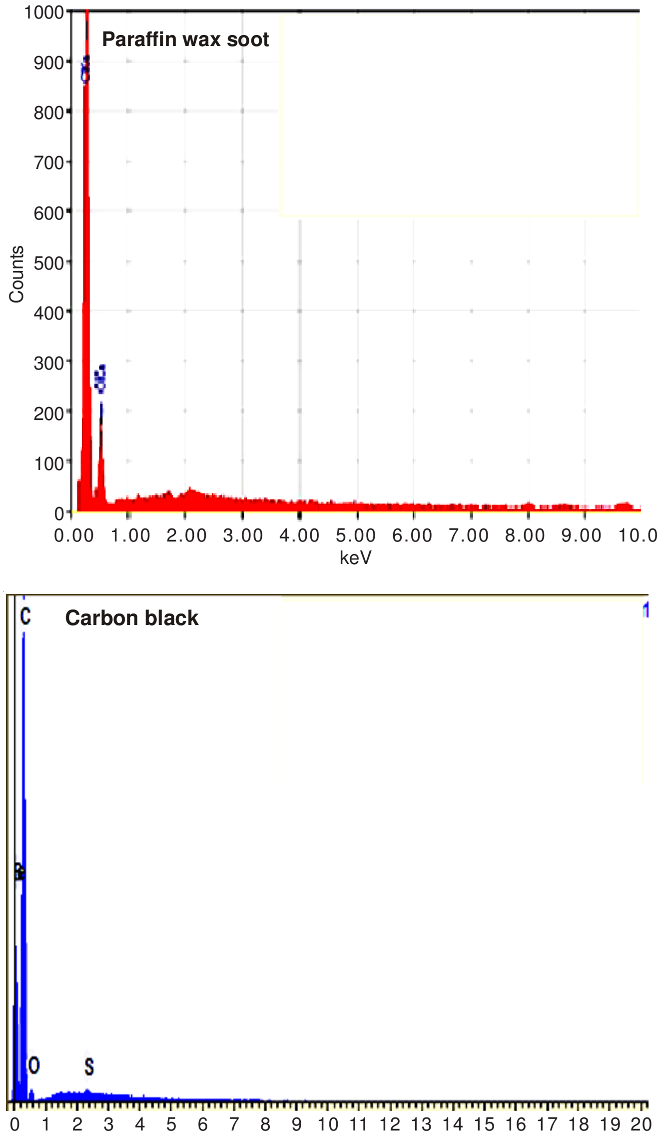


Fig. 3. EDS data of paraffin wax soot and carbon black

## Conclusion

The existence of (100) band in the XRD of both the samples at 2θ *ca.* 42° is an indication of the presence of the graphite like structures. The appearance of strong π-band in both the samples indicates that carbon nanostructures formed are composed of crystalline graphitic carbon. The presence of weak γ band in the diffractogram of the samples is a signature of the low amorphous carbon content and lattice disorders. The SEM micrographs clearly reveal the presence of non-uniform carbon nanospheres and EDS analysis of the samples shows a high carbon content with traces of oxygen and sulphur as the only impurities thereby avoiding the need of any further purifi-cation techniques. Thus, paraffin wax soot and carbon black are very effective and suitable potential candidates for the mass production of carbon nanomaterials.

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## REFERENCES

1. A.N. Mohan and B. Manoj, *Int. J. Electrochem. Sci.*, **7**, 9537 (2012).
2. M. Kumar and Y. Ando, *Diamond Rel. Mater.*, **12**, 1845 (2003).
3. T. Ungár, J. Gubicza, R. Gabor and C. Pantea, *Carbon*, **40**, 929 (2002).
4. D.N. Shooto and E.D. Dikio, *Int. J. Electrochem. Sci.*, **6**, 1269 (2011).
5. E.D. Dikio, *Int. J. Electrochem. Sci*., **6**, 2214 (2011)
6. B. Manoj and A.G. Kunjomana, *Int. J. Min. Met. Mater.*, **19**, 279 (2012).
7. H. Takagi, K. Maruyama, N. Yoshizova, Y. Yamada and Y. Sato, *Fuel*, **83**, 2427 (2004).
8. B. Manoj and A.G. Kunjomana, *Int. J. Electrochem. Sci.*, **7**, 3127 (2012).
9. B. Manoj, A.N. Mohan, S. Sreelakshmi and A.G. Kunjomana, *Int. J. Electrochem. Sci.*, **7**, 3215 (2012).