IOSR Journal of Applied Physics (IOSR-JAP)
e-ISSN: 2278-4861.Volume 3, Issue 6 (May. - Jun. 2013), PP 51-58
www.iosrjournals.org

Nanocarbon Material from Edible Oils: Synthesis and Characterization

M. V. Jayaprakasan[1], K. Viswanathan[1] and P. P. Pradyumnan[2]
1. Department of Physics, Karpagam University, Coimbatore, Tamilnadu, India-641021
2. Department of Physics, University of Calicut, Kerala, India-673635

Abstract: *Nano-sized Carbon materials are synthesized from edible oils by a simple pyrolysis route. Three different vegetable oils, coconut oil with medium chain fatty acid, Mustard oil with short chain fatty acid, and Gingelly oil with long chain fatty acid were selected as precursors. The process conditions were an open air laboratory atmosphere and no chamber or inert gas ambient was used. The prepared agglomerates were subjected for Atomic absorption analysis to investigate their chemical contents. X-ray diffraction analysis was performed to analyze the crystallinity of the samples. A Fourier transform infrared spectroscope (FTIR) was used to identify the inorganic components and surface organic functional groups of the grown material. The electrical conductivity of the sample is investigated by conventional four probe method within a temperature range of 30 to 180 Degree Celsius. SEM micrographs reveal the Carbon powder constituted very fine and quasi spherical particles which are aggregated and the size of the particle ranges from 60 nm to 90nm.*

Key Words: *Edible oils, Tryglycerol, Carbon nano material, Pyrolysis, Carbonisation .*

I. Introduction

Material whose dimensions are in the order of 10^{-9} m has wide applications in all current fields of technology such as chemical, manufacturing, medical, agricultural etc. Carbon nano materials have gained a lot of importance due to their unique physical, electrical and mechanical properties. They have been extensively used in manufacturing fuel cells, molecular sieves, electric nano-conductors, Li-ion secondary batteries, hydrogen adsorbents etc. Carbonaceous materials such as activated Carbon being porous adsorption material with abundant micro pores (0-2) nm, meso pores (2-50) nm and macro pores (> 50) nm with high specific areas are largely used in water and waste water treatment, chemical processing and in gas purification and separation [1,2,7]. The unique tubular structure of Carbon nano-tubes (CNT) makes it best suited for Hydrogen up take. The known synthesis methods of Carbon nano material includes arc discharge, laser vaporization and chemical vapor deposition [3,4,5]. But scientific world is always in search of far simpler and economical methods of synthesis. The different methods proved for the synthesis of Carbon nano material are broadly classified in to two, called Physical and Chemical activation methods [6,16]. Physical activation comprises Pyrolysis (or Carbonization) step followed by controlled oxidation to activate the Carbon in the presence of an activating agent like steam, Carbon dioxide etc. Pyrolysis method of organic compounds and oils is one of the growing methods of nano-material synthesis.

Vegetable oils are important natural raw material in the food sector [7,14]. They are complex mixtures whose primary components are triglycerols (TAGs) of saturated and unsaturated fatty acid units esterifies to glycerol units [8,9,10]. The special interest in their use is mainly due to their large availability, low cost and easy conversion to useful chemicals and new materials. These fatty acids can be saturated or unsaturated with Carbon chain length ranging from 6 to 20 Carbon atoms [11,13,19]. This paper is the result of a humble attempt by the authors to synthesize Carbon nano powder by a combined pyrolysis and particle deposition method using three different edible oils as precursors. The simplicity is the attraction and continuous 6 to 8 hours of pyrolysis is the investment of this experiment.

II. Experimental

The knowledge that pyrolysis of vegetable oil leads to synthesis of Carbon materials is adopted here [12,20]. A large number of vegetable oils are pyrolysed in a clay pot using a highly cleaned cotton medium. In each case rate of evaporation, amount of material deposited, approximate grain size are analyzed. Finally three different vegetable oils such as gingelly oil, (with long chain fatty acids containing 14 to 18 Carbon atoms), coconut oil (having medium chain fatty acids with 10 to 12 Carbon atoms) and mustard oil (with short chain fatty acids having less than 10 Carbon atoms) are selected for the experiment [13,14]. Edible oils were used as the precursor due to its decomposition at high temperature. Pyrolytic methods involve thermal decomposition of hydrocarbons in the presence of Hydrogen and Oxygen [15,20]. The process conditions were an open air laboratory atmosphere and no chamber or inert gas ambient is used. During the nano Carbon growth process the continuous availability of precursor and Oxygen is maintained. The material deposited is collected on a highly cleaned glass substrate maintained at a height of 1.2 m to 1.5 m from the flame. The pyrolysis product in the form of vapor is maintained in the vertical direction using a cylindrical hollow aluminum pipe. The diameter and surface density of particle are controlled by flame temperature and flame substrate separation.

The deposited samples from coconut oil (sample C), mustard oil (sample M), and gingelly oil (sample G) are collected carefully and separately, and then washed thoroughly with water. Thereafter the product is washed with 0.1M HCl and finally with distilled water until the pH of the solution is between 6.0 and 7.0. The sample is then dried at a temperature of about 120 degree Celsius for more than 12 hours and subjected to characterization studies for physical and chemical properties. Acid wash separates the soluble salts and Potassium compounds [16]. Distilled water removes the Chlorine content from the Carbon [17].

III. Characterization.

Atomic absorption analysis of three samples (C, M, and G) is done by an Australian make AA-20 Atomic absorption spectrometer. X-ray diffraction analysis was performed to analyze the crystallinity of the deposits by a Bruker AXS D 5005 X-ray diffractometor using a Cu-Kα radiation with an accelerating voltage of 30 KV. UV-vis-NIR spectroscopy of the three samples was recorded using Nujoll Mull in the region from 190 nm to 2500 nm using a Varian-Carry 5000 spectrophotometer. The Vibrational behaviour of the Carbon samples was studied by a FT-IR spectrophotometer, Thermo Nicolet AVATHAR 370 GTGS. The electrical behaviour of the samples is analyzed by conventional four probe method. Finally the surface morphology and the particle size are investigated by scanning electron micrography using a JEOL-JSM 5600 LV instrument with an accelerating voltage of 15 KV.

IV. Results And Discussion

4.1 ATOMIC ABSORPTION ANALYSIS

The data obtained from the Atomic absorption analysis of the prepared Carbon samples are tabulated below.

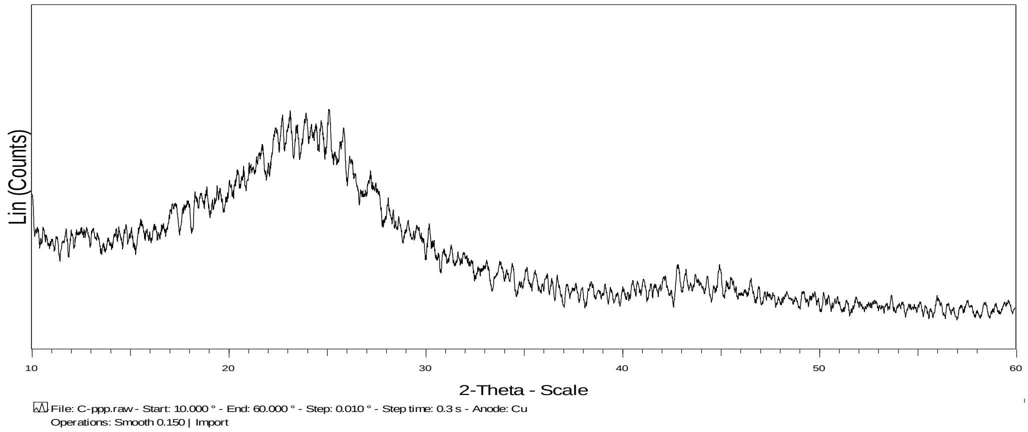
SAMPLE CODE	Ca (mg/Kg)	Mg (mg/Kg)	Fe (mg/Kg)	Mn (mg/Kg)	K (mg/Kg)
C	Trace	153	897	9.4	2015
M	63	75	84	4.5	205
G	77	52	51	2.2	165

Table.1. Atomic absorption analysis data for sample C,M and G.

From the table the purity of the sample is maximum for sample G and minimum for C. But the heavy element concentration in all the three samples is comparatively small.

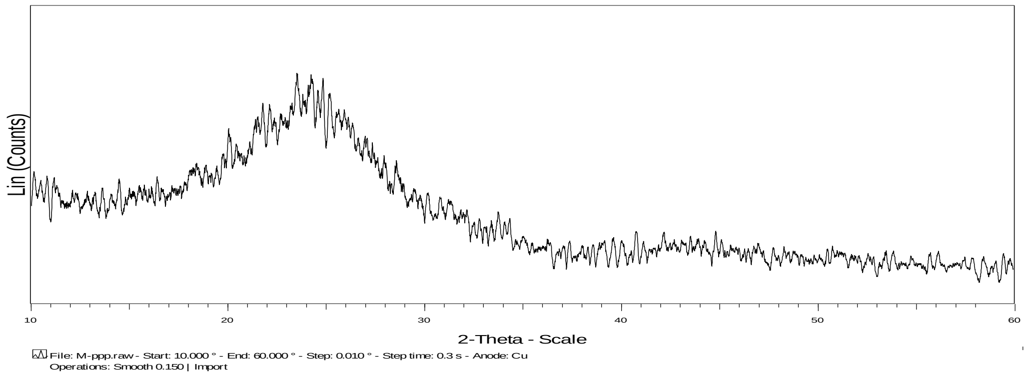
4.2 X-Ray DIFFRACTION STUDIES

The X-ray diffraction studies of acid treated samples are shown in fig 2(a), 2(b) and 2(c).



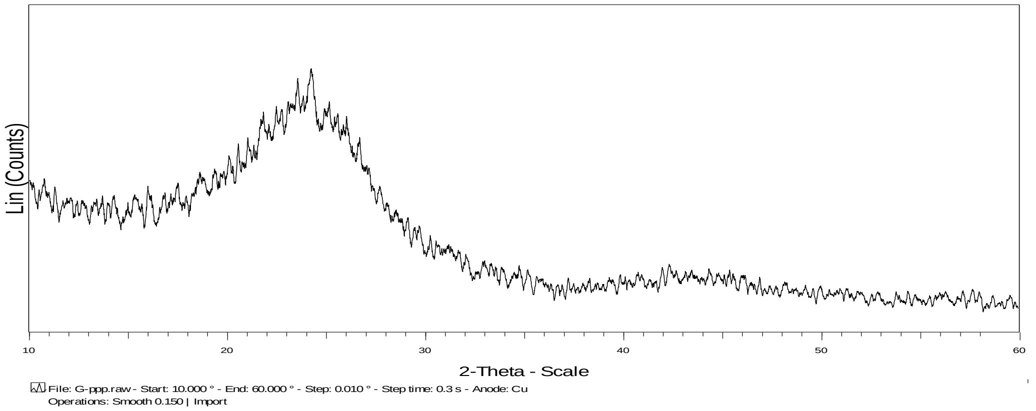
C

Fig.2 (a) X-ray diffraction pattern of sample C



M

Fig.2(b) X-ray Diffraction pattern of sample M



G

Fig.2 (c) X-ray diffraction pattern of sample G

The samples prepared from coconut oil, mustard oil, and Gingelly oil after purification gives almost same X-ray diffraction spectra. No sharp peaks are observed and the broad hump around 22 degree is a characteristics of short range order[18,19].

4.3 UV-VISIBLE - NIR SPECTROSCOPY

Fig 3(a), 3(b) and 3(c) are the UV-vis-NIR spectroscopy results recorded in solid phase using Nujoll Mull in the region from 190 nm to 2500 nm.

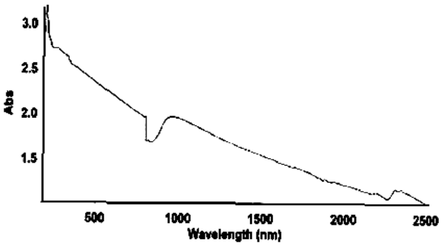


Fig. 3(a) UV-VISIBLE NIR SPECTRA OF SAMPLE C

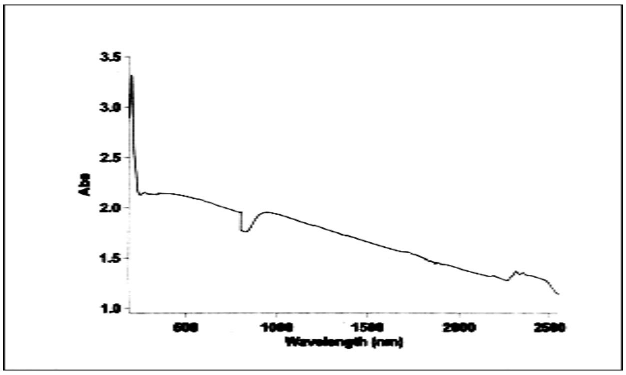


Fig.3(b)UV-VISIBLE-NIR SPECTRA OF SAMPLE M.

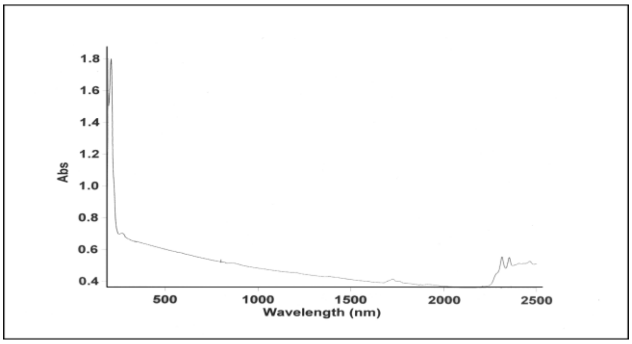


Fig.3(c) UV-VISIBLE-NIR SPECTRA OF SAMPLE G.

The absorption peak observed at 203.2 Nm in all the samples is due to the presence of solvent (Nujoll). On comparison with standard absorption spectra no major absorption peaks were observed in all the three samples. Each sample shows some minor absorption around 1752 nm indicating the presence of C=O; Ketone. Also minor peaks at wave lengths 2300 nm and 2350 nm indicates the presence of C≡N. This indicates the purity of the samples.

4.4 FT-IR SPECTROSCOPIC STUDIES

The functional and composition quality of the synthesized samples are analyzed by FT-IR Spectroscopy within a wave length range of 500 cm^{-1} to 4000 cm^{-1}. The graphs are depicted in fig 4(a), 4(b) and 4(c).

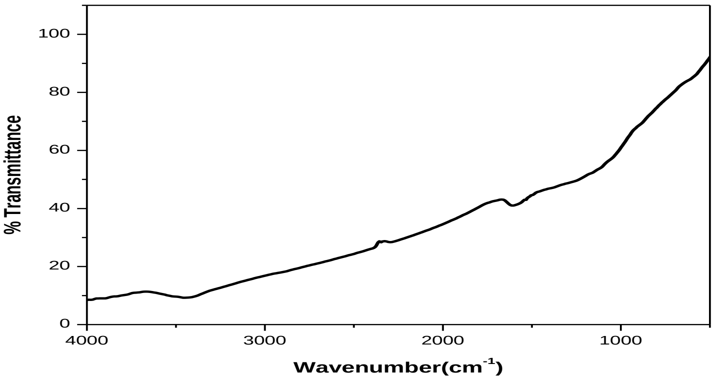


Fig. 4(a) FT-IR spectra of sample C.

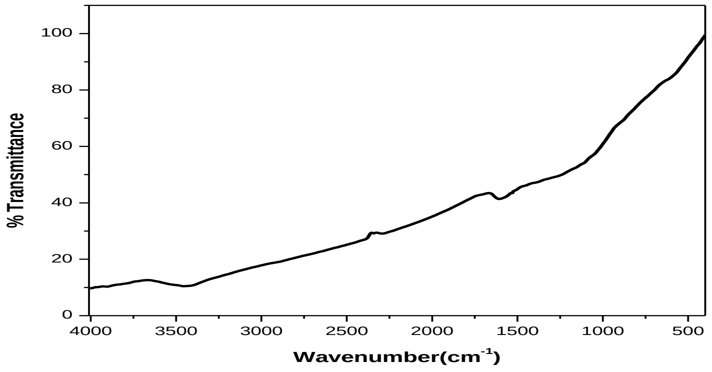


Fig.4 (b) FT-IR spectra of sample M.

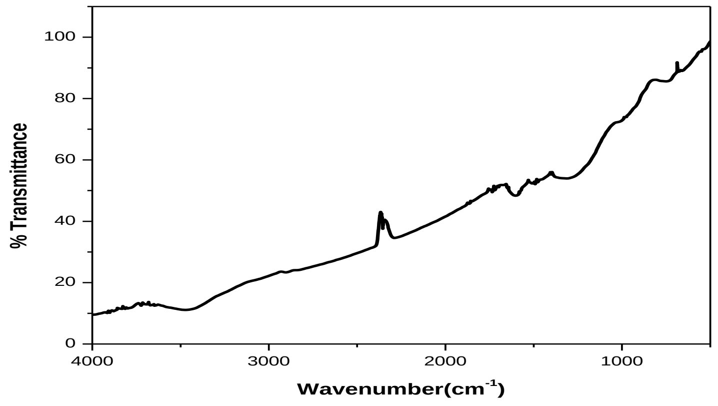


Fig. 4 (c) FT-IR spectra of sample G.

The sample C (fig 4(a)) shows some peaks at 3390 cm^{-1} indicating the presence of O-H bond due water of crystallization. The absorption available at 1450 cm^{-1} and 1600 cm^{-1} strongly supports the presence of C=C and C-O stretching vibration[20].

Compared with other samples, sample M (Fig4(b)) shows very few absorption peaks, other than the presence of O-H bond due to the water of crystallization and the peak at 1610.96 cm^{-1} which is due to the C=C stretching.

In sample G (fig 4(c)) the absorption at 3473.43 is due to the O-H group in the moister content. The minor absorption at 2282 cm^{-1} is due to C≡N and that at 1598.41 cm^{-1} is due to C=C stretching.

4.5 ELECRICAL CONDUCTIVITY STUDIES

To investigate the electrical characteristics, conventional four probe method is employed. Fig 5(a), 5(b) and 5(c) depict the graph plotted between the logarithms of resistivity with inverse of temperature within a temperature variation from 30 Degree Celsius to 175 Degree Celsius.

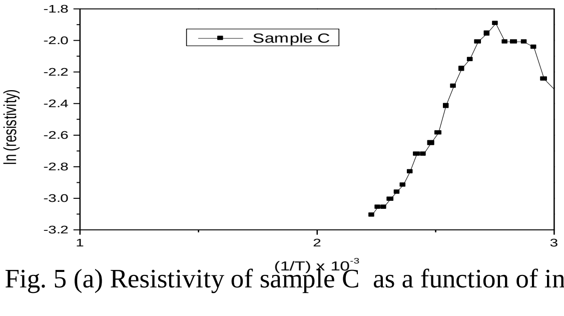


Fig. 5 (a) Resistivity of sample C as a function of inverse of temperature.

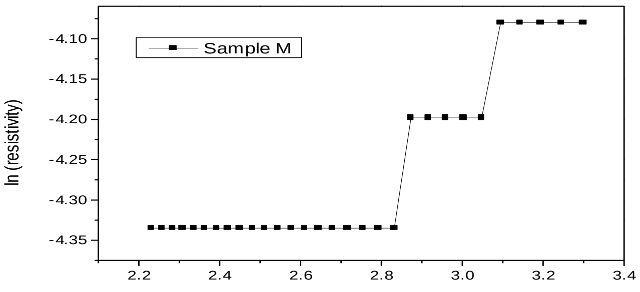


Fig. 5 (b) Resistivity of sample M as a function of inverse of temperature.

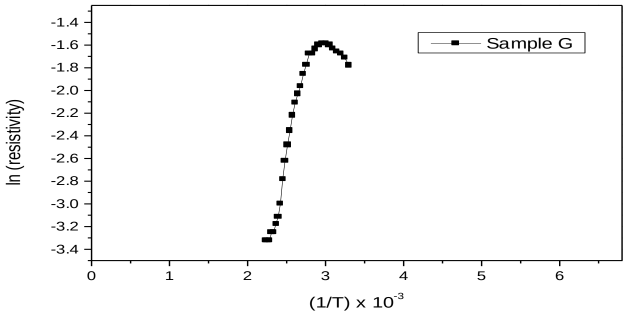


Fig. 5 (c) Resistivity of sample G as a function of inverse of temperature.

On analyzing the resistivity plots for sample C, M and G the resistivity variation for sample C is 0.0447 Ohm-m to 0.1506 Ohm-m within the given temperature variation. The band gap energy calculated is 0.528 eV. Within the same range sample M shows the resistivity variation from 0.0131 Ohm-m to 0.0169 Ohm-m and band gap measurement gives a result of 0.5883 eV. Resistivity variation is comparatively higher for sample G, from 0.03604 Ohm-m to 0.2048 Ohm-m and the band gap energy value is estimated 0.8167 eV. All the three samples show an initial decrease in conductivity due to the humidity content in the samples.

4.6 SEM-MICROGRAPHY.

The SEM micrographs at low and high magnifications for samples C, M, G are shown in fig 6(a), fig 6(b), and fig 6(c).

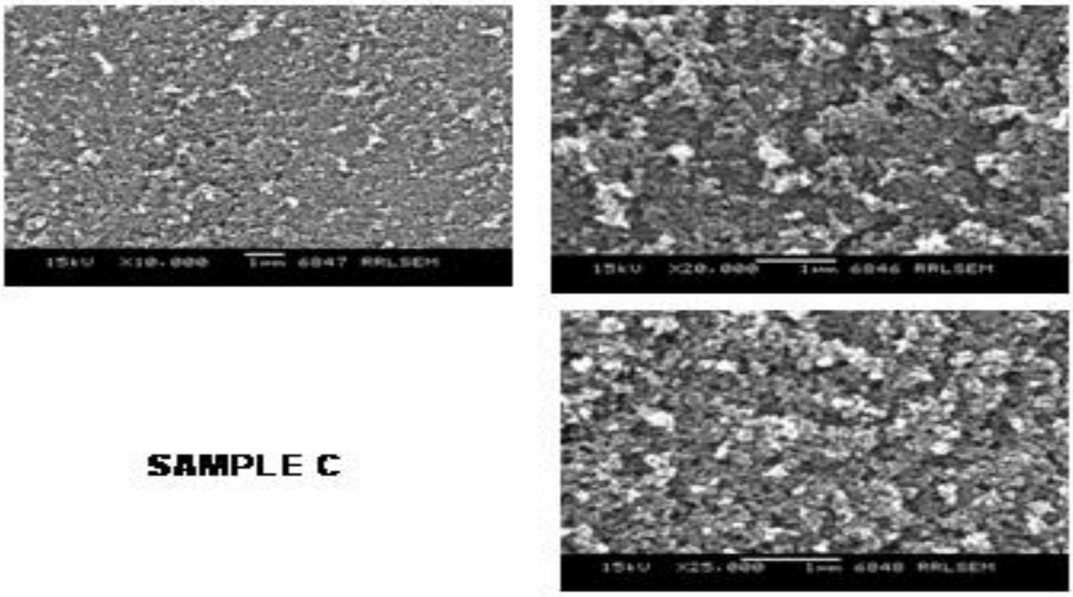


Fig. 6(a) SEM micrographs of the sample C

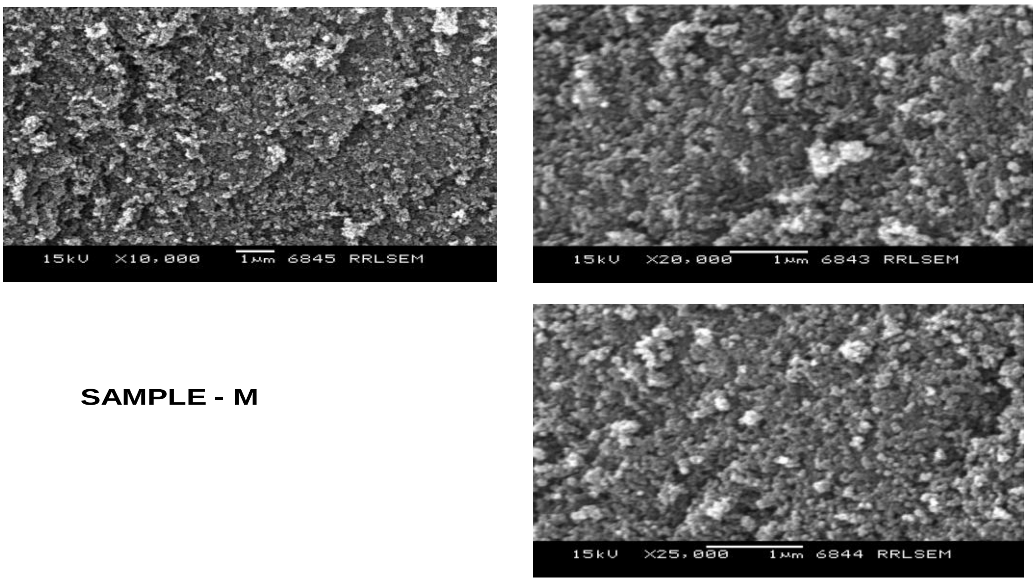


Fig. 6(b) SEM micrographs of the sample M

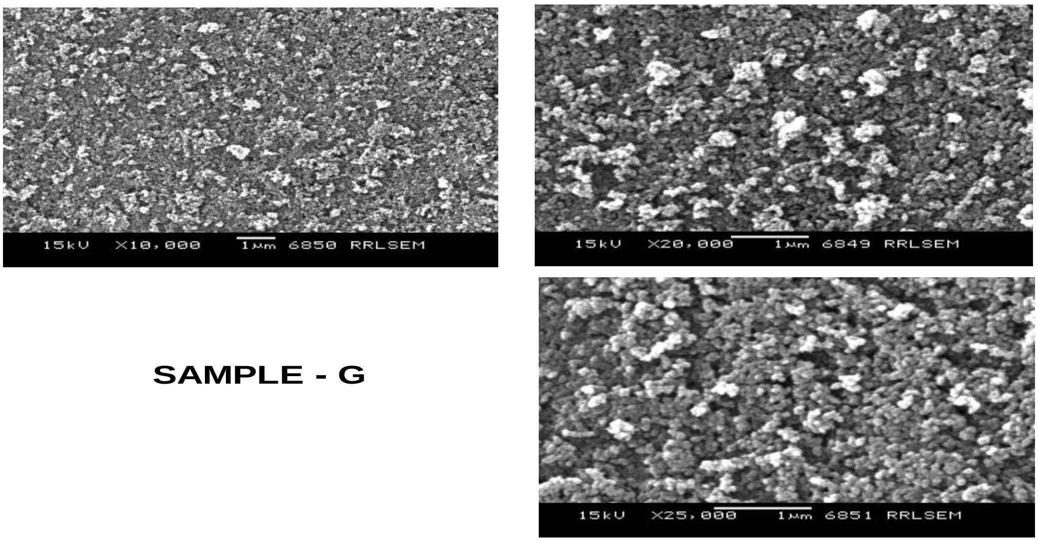


Fig. 6 (c) SEM micrographs of the sample G

SEM micrographs are recorded for each sample in three different magnifications. The low magnification images reveal that all the samples consist of large number of petite identical spherical particles. The corresponding high magnification image shows the morphology of the samples are almost same. From the high magnification images the particle diameter estimated for sample C is in the range of 60 to 70 nm, that of sample M is 70 to 80 nm and for sample G is 90 to 100 nm. This indicates the particle diameter is well within the nano regime.

V. Conclusion

We successfully synthesized nanocarbon materials in a humble inexpensive method at normal laboratory condition. The atomic absorption analysis proved the major content Carbon in all the three samples. XRD line profile denied the long range order. FT-IR spectra points the presence of hydroxyl groups, C=C stretching and C≡N bending. The semiconducting behaviour of samples is confirmed by conductivity studies. Finally coconut oil with medium chain fatty acids gives the smallest size nano particle even though other samples also lie within the nano regime. Due to the growing interest in nano particles especially in biological systems, further studies will be continued to investigate the antibacterial properties of nanocarbon, synthesized from coconut oil.

References

[1] C.Kropf, C. Hundaiker, M.Heller and C.Wild, nano sized Zinc oxide in hygiene products, *U S patent application*, U S 2004/0033270 Al, (2004).

[2] C.Schwandt, A.T.Dimitrov, D.J.Fray., The preparation of nano-structured carbon materials by electrolysis of molten lithium chloride at graphite electrodes. *J of Electroanalitical chemistry 647 , 150-158 ,2010.*

[3] W.H.Qi and M. P. Wang, Size effects on the cohesive energy of nano particles, *J.mat.sci. Lett 21, 1743-1745, (2002).*

[4] K.Nanda , A. Maisels , F.E.Fissan and S.Strappert, Higher surface energy of nano particles, *Phy.rev.lett 91, 106-112 (2003).*

[5] Huiping zhang, Ying Yang, Lichung Yang, Preparation of active carbon from saw dust by ZnCl activation, *Adsorption 161-166, (2010).*

[6] Ebbsen TW, Ajayan PM, Storage of Hydrogen SWCNT, *Nature, 358, 220-222, (1992).*

[7] Guo.T, Nicoleav P, Ihess A , Colbert D.T, Smalley R.E., Catylytic growth of single walled Carbon nano tube by laser vaporization, *Chem Phys let, 243, 49-54, (1995).*

[8] M.Raheena Beegum, *A Text book of Foods and Nutrition* Jay pee brothers medical publishers pvt Ltd New Delhi (2002).

[9] D.G.Deriabin , E.S.Aleshina , A.S.Tlyagulova. Acute toxicity of Carbon based nanomaterials to Escherichia Coli is partially dependent on the presence of process impurities. *Nanotechnologies in Russia, Vol-6 528-534 ,2011.*

[10] Carlsson.A S, Plant oils as feed back alternatives to petroleum , A short survey of potential oil crop platforms , *Biochimie, 91, (665-670),(2009).*

[11] Suman Tripati , Maheswar Sharon , N.N.Maldar , Jayasri Shukla , Madhuri Sharon. Carbon nano spheres and nanotubes from Castor oil as precursor : for removal of Arsenic dissolved in water. *Arch.Appl.Sci.Res., 4(4):1788-1795 ,2012.*

[12] Merten. B, Pfeur M and Schreze Meir j. Medium chain triglycerides,*Int Dairy J 16, 1374-1382, (2006).*

[13] Papamandjaris . A. A, MacDougal. D.E. and Johns P.J.H, Medium chain fatty acid metabolism and energy expenditure, Obesity treatment implications, *Life sci., 62 ,1203-1215, (1998).*

[14] Guillot.E., Vaugelade. P., Lemarachal.P.Reral A, Intestinal absorption and liver uptake of medium chain fatty acids in non –anaesthetised pigs, *Br.J Nutr, 69, 431-432, (1993).*

[15] Rodriguiz –Reynosa. F, *Hand book of porous solids,* wiley-vch Verlag Gmbh, weinhein, Germany p 481, (2002).

[16] Georgea Vigli , Angellos Philippides , Appostalos Spyros, Photis Dois, Classification of edible oils by employing 31P and 1H NMR spectroscopy with multivariate statistical Analysis, *J.Agricultural and Food Chemistry , 53, 5715-5722,(2003*).

[17] Seoktae Kang, Mathieu Pinault, Lisa D Pfefferle and Menachem Elimelech, Single walled Carbon nano-tubes exhibit strong antimicrobial activity. *Langmuir 23, 8670-8673, (2007).*

[18] C.Charitidis .S, Logotheditis .M.Gioti , A comparative study of the nano scratching behavior of amorphous Carbon films grown under various deposition conditions *,Surface coating tech. 123, 201-206, (2000).*

[19] Gurpreet kaur, Raman preet Singh, Antibacterial and membrane damaging activity of Livistona chinesis fruit extract, *Food and chemical Toxicology 46, 2429-2434, (2008).*

[20] Changxin Chem., Wenzhe Chen, Yafai Zhang. Synthesis of Carbon nano-tubes by pulsed laser ablation at normal pressure in metal nano-sol, *Physica, E 28, 121-127, (2005).*