

Synthesis of Nanocarbon Powder from Sesame Oil and Its SEM Characterization

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Abstract- In this work a simple pyrolysis route to synthesis nanocarbon powder from Sesame oil is followed. The precursor sesame oil used as the source of hydrocarbon by decomposition at high temperature, is a group I vegetable oil with long chain fatty acid containing 16 or 18 carbon atoms. Experiment was performed in an open air laboratory atmosphere and sophisticated laboratory conditions such as inert gas atmosphere, isolated chamber etc are avoided. The synthesized agglomerates are subjected to Atomic Absorption Spectroscopy to investigate its chemical contents. X-ray diffraction (XRD) study is carried out to analyze the grain structure of the fabricated powder. The functional groups present in the sample is investigated by employing the Fourier Transform infrared absorption spectra (FT-IR). Extensive Scanning electron microscopic (SEM) investigations showed that the size of the nano particles were within the range of 50 nm to 70 nm. The electrical conductivity of the sample is studied by four probe method within a temperature variation ranging from 30°C to 175°C.

Index Terms- Carbon nano-material, sesame oil, Carbonisation, Pyrolysis.

I. BACKGROUND

The production and use of carbon nano-material in different scientific areas have increased unbelievably during the past few years. 'Nano-material' will be the building block of forthcoming technology with carbon being the leading member. Nano structures, with at least one dimension between 1 nm to 100 nm have attracted steadily growing interest due to their fascinating properties as well as unique applications. Within the nano regime the fundamental characteristics of the material strongly differ from that of the bulk [1]. Nowadays the researchers concentrates their investigations on the special properties of metals, semiconductors, amorphous materials, layered structures, superconductors etc., when their grain size approaches to nano-scale. The exotic properties of nano material are largely due to the quantum confinement of electrons within a nano metre sized potential well [2,13]. Also, these materials are made in a very unique way, stacked atom by atom or atomic layer by layer [3]. Nano-material can be fabricated on different ways such as sputtering, gas condensation, spray conversion processing, electron deposition, physical vapour deposition, chemical method and mechanical milling [4,5]. All these methods being promising and effective but simpler and economical preparation methods are still the research emphasis and this work points in this direction.

The precursor sesame oil is used as a healing oil for thousands of years. In Vedas it is mentioned as excellent for humans and later the antibacterial property against common skin pathogens such as staphylococcus aureus, streptococcus aureus and many fungi are proved in different scientific studies [6,20]. Ayurvedic physicians all over the world uses this oil for the treatment of several chronic diseases including hepatitis, diabetes, migraine etc. Sesame seed oil is a potent antioxidant and will neutralize oxygen radicals [7,12].

Carbon, due to its versatility and diversity in different fields, is named as the 'king of elements'. The biochemical mechanism responsible for life are very much dependent on the role of Carbon either directly or indirectly [8,9]. Natural Carbon exists in two isotopic forms such as C₁₂ and C₁₃. The different allotropic forms of carbon are diamond, graphite, fullerenes, Carbon nanotubes and amorphous Carbon. There are some modified forms of Carbon such as diamond like Carbon, glassy Carbon and Carbon fibers [10,17].

Carbon nano-materials with its remarkable electronic and mechanical properties attracted the attention of the scientific world. Carbon served the world as electrodes, electronic components, absorbents for environmental protection or high strength carbon fibers, composites for structural applications, and now opening a new world of nanoscience and technology [11,14]. Now carbon nano-materials as layered materials, fullerenes, nanoclusters, nanofibres and nanotubes, are important tools for most of researchers all over the world. Carbon nanotubes with its immense industrial applications serve us as energy storage devices, hydrogen storage devices, sensors, electrodes for Lithium ion batteries and in electronic and communication industry[12,15]. Another industrial importance of carbon nanotube is due to its unbelievably high young's modulus[10]. Pyrolytic carbon with excellent mechanical properties such as wear resistance, fatigue resistance and bactericidal property and also being highly bio-compatible are now used for making artificial heart valves [13,19]. This paper is the result of a humble attempt by the authors to fabricate the carbon nanopowder by a simple thermal pyrolytic deposition method in normal atmospheric conditions.

II. EXPERIMENTAL PROCEDURES

The basic knowledge that the pyrolysis of sesame oil produces Carbon vapour is adopted in the experiment. Sesame oil being a vegetable oil is a complex mixture whose primary components are triglycerols (TAGS) of saturated and unsaturated fatty acids [12]. The precursor sesame oil has a percentage

composition of oleic acid 45, linoleic acid 41 and polyunsaturated fatty acids in trace[13,21]. Pyrolysis is carried out with different source substrate separation. In each case rate of evaporation, amount of carbon produced, approximate grain size, etc. are analyzed. Finally substrate is localised in position giving minimum rate of evaporation and producing carbon with lowest grain size. Since the experiment is performed in open air atmosphere, highly hygienic laboratory condition is ensured. The oil is burnt in a small pot with the help of highly cleaned cotton thread medium. To restrict the carbon vapour in upward direction a cylindrical chimney made of aluminium sheet is used. A glass substrate is used to deposit the Carbon vapor. The distance from the flame to the substrate is adjusted as 1.5 m to deposit particle of lowest size. The presence of oxygen as a component in the reaction system, making possible the formation of carbon monoxide reduces the tendency to deposit non fibrous carbonaceous material [11,15]. During pyrolysis the sesame oil decomposes and reacts forming clusters on which carbon nano material including carbon nano tubes may nucleate and grow [12]. To get appreciable deposition substrate is exposed 7 to 8 hours to the flame.

The prepared sample is soaked in 100 ml of 1M HCl and heated at 80°C for 20 minutes. After cooling at room temperature for 24 hours it is filtered and then washed with distilled water five times. Finally it is rinsed with acetone to remove the traces of water and then dried in oven at 120 °C for 24 hrs. This purified sample is subjected for characterization studies.

III. RESULTS AND DISCUSSION

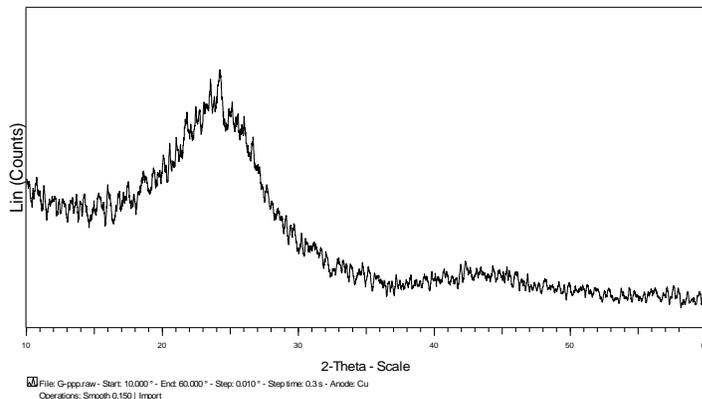
3.1. Atomic Absorption study

Atomic absorption analysis of the purified sample is carried out by an Australian Make AA-20 Atomic Absorption Spectrometer. The analysis of data gives knowledge about its various contents as Calcium 76 mg/kg, Magnesium 50 mg/kg, Iron 51 mg/kg, Manganese 2.3 mg/kg and Potassium 162 mg/kg. This quantifies that the sample is 99.9% pure in Carbon.

3.2. XRD analysis

The X-ray diffraction (XRD) study was conducted by a Bruker AXS D 5005 X-ray diffractometer, using Cu- α radiation filtered by a graphic monochromator at a setting of 40 KV and 130 mA. The X-ray diffraction pattern obtained is shown in Fig. 1.

The absence of peaks in the spectrum clearly indicates the absence of long range order. The broad hump observed around 20-22° is a characteristic of disordered Carbon [16].



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Figure 1: XRD Pattern of the Sample

3.3. UV-vis-NIR Spectral analysis

UV-visible-NIR Spectroscopy of the sample is done in solid phase using Nujol Mull in the region 190 nm to 2550 nm. The analysis is carried out by a Varian-Carry 5000 spectrophotometer. The figure below depicts the UV-vis-NIR spectrum of sample. No major absorption peaks are observed in this range and the only peak available at 203.2 nm is due to the solvent (nujol). This also indicates the purity of the sample.

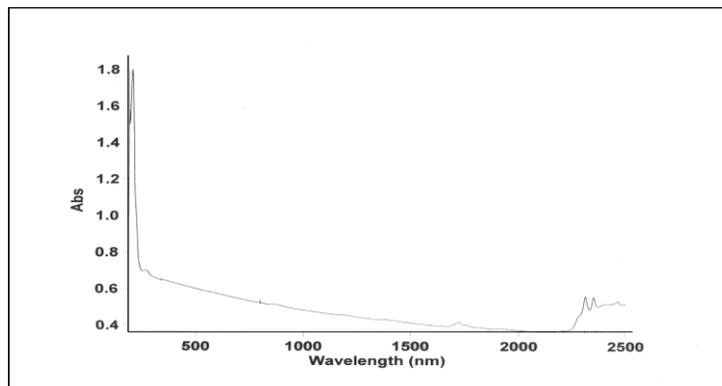


Figure 2: UV-visible-NIR Absorption Spectra of Sample

The minor peaks of absorption around 1725 nm indicates the presence of C=O (ketone) and at 2300 nm and 2350 nm is due to the presence of C≡N.

3.4. FT-IR Analysis

The FT-IR spectrum of sample in the form of KBr pellets is analyzed by a FT-IR spectrophotometer, Thermo Nicolet-AVATAR370-GTGS. The graph is plotted with wave numbers on X axis and percentage of transmittance on Y axis.

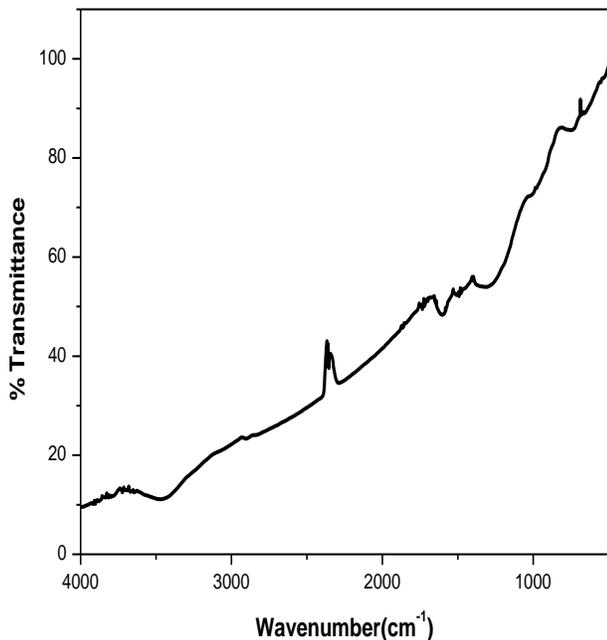


Figure 3: FT-IR Spectra of Sample

Analysis of the spectrum shown in Fig. 3 indicates no major absorption bands. However minor bands at 3473.43 cm^{-1} are connected with ν (O–H) vibrations in hydroxyl groups present in the sample [16]. This is due to the moisture content in the sample. The weak band defined at 1598.41 cm^{-1} is due to ν (C=C) vibrations. Also the absorption indicated at 2282 cm^{-1} leads to the presence of C≡N stretching .

3.5. Scanning electron microscopic study

The surface morphology of the synthesized sample was characterized by scanning electron microscopy. Low and high magnification images are presented in figure 4. Synthesized particles are found as almost identical spherical particles with uniform morphology.

Fig. 4 shows typical SEM image of Carbon film grown in this study recorded by the JEOL-JSM 5600 LV instrument with an accelerating voltage of 15 KV.

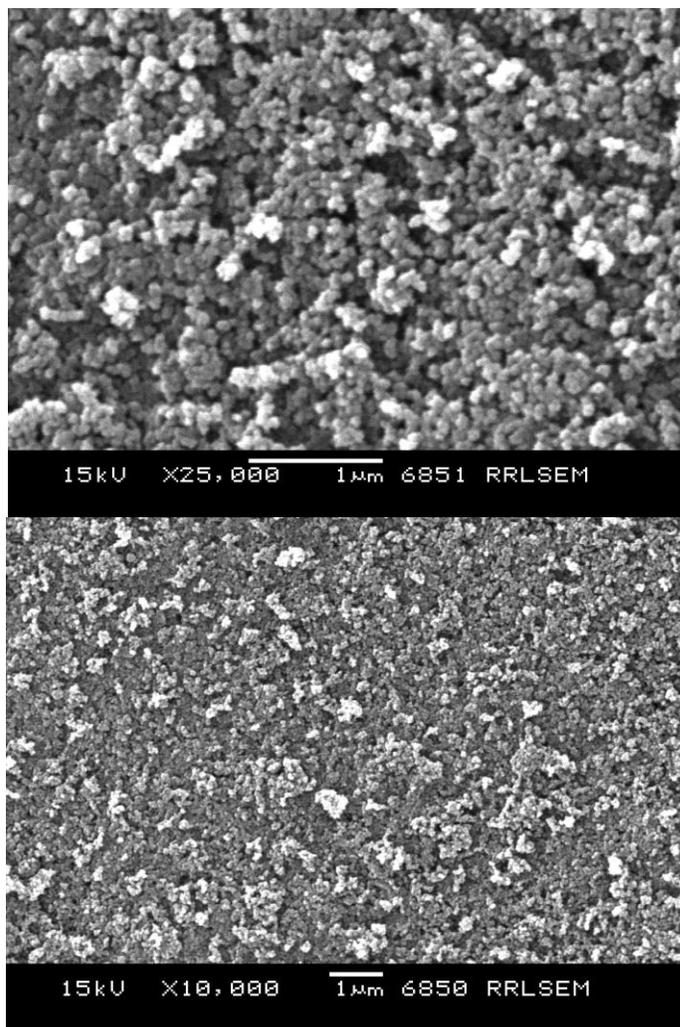


Figure. 4: SEM micrograph of the sample

The particle size calculated from the SEM micrograph ranges from 60 nm to 70 nm and is well within the nano regime .

3.6. Electrical conductivity studies

The conductivity study is carried out by measuring the resistivity by conventional four probe method. The resistivity is measured within a temperature variation ranging from 30°C to 175°C . . A graph is plotted between the logarithm of resistivity with inverse of temperature . The plot is shown in Fig. 5.

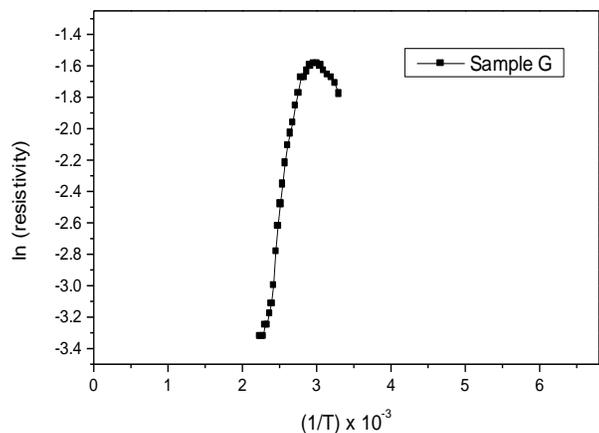


Figure 5: Resistivity of Sample as a Function of Inverse of Temperature

The resistivity of the sample is found to vary from 0.03604 Ω -m to 0.2048 Ω -m within the temperature variation from 30°C to 175°C. The initial decrease in conductivity with increase in temperature as observed in the Figure is due to the moisture content in the sample [18]. After this region the sample behaves as a perfect semiconductor with an increase in conductivity with temperature.

IV. CONCLUDING REMARKS

To conclude, our humble attempt is to demonstrate, nanoparticle is not only the result of complicated reactions and sophisticated laboratories but it can also be prepared by simple means in normal atmospheric conditions. The atomic absorption analysis study proves the purity of the prepared sample. Also the XRD line profile analysis clearly indicates the absence of long range order in the deposited carbon powder. The FT-IR spectra points towards the presence of hydroxyl groups, C=C stretching and C≡N bending. SEM micrograph analysis proves Carbon powder consists of quasi spherical particles and the particle size is well within the nano regime and it ranges from 50 nm to 70 nm. Finally in the electrical property studies, the sample confirms its semiconducting behaviour. Further studies on the antibacterial and wound healing properties of carbon material prepared from sesame oil is to be continued.

REFERENCES

[1] C. Delerue, M.Lannoo, *Nanoclusters theory and modelling*, Springer-Verlag-India (2004).
 [2] C. Charitidis .S, Logothetidis.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).
 [3] S. R. Elliot, *Physics of amorphous materials*, Longman Scientific-2nd Edition(1990)
 [4] K. K. Nanda, A. Maisels, F.E.Fissan and S. Strappert, Higher surface energy of nano particles, *Phy.rev.lett* 91, 106-112 (23).

[5] Changxin Chem, Wenzhe Chen, Yafai Zhang. Synthesis of Carbon nanotubes by pulsed laser ablation at normal pressure in metal nano-sol, *Physica, E* 28, 121-127,(2005).
 [6] S. Bhardwaj, A. Gupta,S.Pandey, G.Oza, S.Kawale, N.Misra,Maduri Sharon, A.Durve, M. Thandu,. Maheswar Sharon and C. Cepek; ‘Methylene blue absorption Isotherm for Carbon Nano Material Synthesized from Menthol’ in V. Rajendran, K. Hillbr and K. Swaminathan and K.E. Geskeler Ed.*Synthesis and characterization of Nano structured materials*;399-404,Macmillan Publisher India Ltd. 2010.
 [7] M. Raheena Beegam. *A Text book of Foods,Nutrition*, Jaypee brothers Medical publishers pvt. Ltd. New Delhi (2002).
 [8] Seema Yadav, *Food Chemistry*, Anmol Publishers. New delhi (1997)
 [9] Ebbsen T W, Ajayan P M, Storage of Hydrogen SWCNT, *Nature*, 358, 220-222, (1992).
 [10] Ruoff. R.S., Lorentz.D.C.,Mechanical and thermal properties of Carbon nanotubes. *Carbon*, 33, 925-930, 1995.
 [11] Hauert R.Areview of modified DLC coatings for biological applications. *Diam.Relat.Mater* ; 12; 583-589; 2003
 [12] Sumati.R, Mudambi,Shalini.M.Rao, *Food Science*, New age International (P) Limited publishers. New Delhi. (1997)
 [13] *The wealth of India. A Dictionary of Indian Raw materials and Industrial products*, Raw materials Vol. IV, CSIR-New Delhi (1956)
 [14] Suman Tripathi, Maheshwar Sharon, N.N.Malder,Jayashri Shukla,Madhuri Sharon, Carbon Nano Spheres and nano tubes synthesized from Castor oil as precursor; For removal of Arsenic dissolved in water..*Archives of applied science research*, 4(4), 1788-1795,2012.
 [15] Nepal D., Balasubramanyan. S., Simonian, A.L and Davis,V.A Strong antimicrobial coatings;single walled carbon nano tubes armored with biopolymers. *Nano let* 8,1896-1901. 2008.
 [16] Colin N Banwell, Elaine M Mc Cash,*Fundamentals of molecular spectroscopy*, Tata Mc Graw-Hill, New Delhi 1996
 [17] Suzi Demirbag, Cuneyt Atabek, AhmetGüven, Emin ostaz, İlhami sürer, Haluk oztürk, Nartar Yesildaglar, Effects of Pyrolytic Carbon on post-operative adhesion formation in rats. *Arch. Gynecol obstet*, 279, 11-15, (2009).
 [18] Suchet.I.P., *Electrical conduction in solid materials*, Pergamon Press, Oxford (1975).
 [19] J.P.Srivastava,*Elements of solid state physics*. Prentice Hall of India (2001).
 [20] Carlsson. A S.. Plant oils as feed back alternatives to petroleum .A short survey of potential oil crop platforms, *Biocheimic*, 91, (665-670), 2009.
 [21] 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).

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