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Surface Area measurement of Carbon Nanomaterials obtained from Castor oil

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ABSTRACT

This document Carbon nanomaterials (CNM's) synthesized from castor oil by direct pyrolysis at 750°C in an inert atmosphere. CNMs was then characterized by (XRD) X-ray diffraction, SEM (Scanning Electron Microscope) and FTIR (Fourier Transform Infra-Red Spectroscopy)The specific surface area measurement of CNMs was done by using adsorption of methylene blue dye(MB). In this method a fixed concentration of aqueous methylene blue solution was allowed to adsorb on fixed quantity of carbon nanomaterial. The amount of adsorbed methylene blue dye was determined by UV spectrophotometer. It was found that the concentration of adsorbed methylene blue corresponds to the surface area of test sample. The surface area of CNM was found to be 32m2/g. The obtained data was correlated with that of BET surface area measurement and found to be comparable.

Keywords : Castor Oil, Surface Area, Carbon Nanomaterial.

I. INTRODUCTION

A carbon nanotubes discovery in 1991 [1] was a microscopic wonder, due to the porosity with nanometer size and large surface area. The synthesis of carbon nanomaterials from natural precursors receiving the interest because of its unique characteristics of utilization of waste material in different applications via synthesis of different CNM's. from it. Most of the researchers are using petroleum products for the preparation of carbon nano materials; sandesh Jaybhaye et al [2] at NTRC is able to produce these materials from plant derived precursors. There are various physical and chemical properties of CNM has been studied for different applications like hydrogen storage [3] super capacitor [4] solar cell applications [5] micro wave absorption for lithium ion battery [6] etc. has been carried out. Specific surface area is one of the important property of carbon nanomaterials that can be related to their physical or chemical behaviour. The most commonly used methods to evaluate the specific surface area of carbon nanomaterials are based on adsorption of nitrogen [7], water vapour [8], ethylene glycol mono ethyl ether [9] or colour dye [10]. A very simple adsorption of methylene blue hereafter will be called as MB is used in lab to determine the specific surface area of the carbon nanomaterials which is synthesized in laboratory using a castor seed oil. In this method MB is used to adsorb on CNM and is then evaluated by using a UV-Vis spectrophotometer.

II. METHODS AND MATERIAL

PREPARATION OF CATALYST

The catalyst is necessary for the synthesis of Carbon nanomaterials. The diameter of the catalyst particals should be in nano range ~ 40 to 60 nm. Particle of such size can be produced by the urea decomposition method [11].

A mixture of Nickel nitrate and urea; 5g and 10g each respectively was taken in a beaker and kept on heater with continuous stirring. A homogeneous liquid mixture was formed then this mixture was kept in muffle furnace in presence of air at 300°C till completely dry. As a result, urea gets decomposed and burned off, nickel was oxidized to form nickel oxides which was left in the form of residue.

Then this residue of Nickel oxide was collected and reduced in Hydrogen atmosphere at 650°C for 2 hours. This gave Nickel catalyst of nano size.

SYNTHESIS OF CNM

The CNM was synthesized from castor oil using a Nickel catalyst by chemical vapour deposition method(CVD).In this CVD synthesis method two electric furnaces A and B (vaporizing Furnace A and Pyrolising Furnace B) and one meter long quartz tube kept inside both the furnaces was used. The schematic representation of arrangement of all the apparatus was as represented in fig-1. Weighed 4 g of castor oil in quartz boat (C) and 200 mg of Nickel catalyst in other quartz boat (D) and kept both the boats in furnace A and B respectively.

The Hydrogen gas was allowed to pass through a quartz tube (Q) for five minutes with constant flow rate so as to remove the oxygen from tube. The temperature of pyrolysis furnace (B) was set to 750°C. Once the furnace B reaches a desired temperature. The furnace A was turned on and the temperature was set to the boiling point of castor oil (i.e.~350°C). The heating of furnace A was continued till all oil gets vaporized. The heating of furnace B was continued for one Hour. The furnace B was allowed to cool at room temperature and the material was collected from boat D.

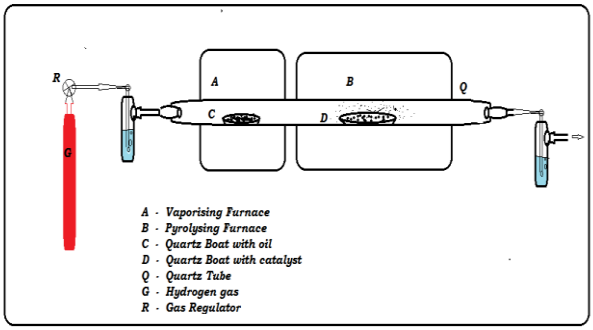

Fig 1.Schematic representation of CVD furnace

PURIFICATION OF CNM

The obtained CNM was treated with HNO3 and HCl so as to dissolve the traces of metal impurities and amorphous carbon. The purified CNM was then dried in furnace at 200°C for 1Hr.

III. RESULT AND DISCUSSION

The purified CNM was characterized by using a SEM, FTIR and powder XRD.

SEM STUDY

Scanning Electron Microscopy study of CNM represents the morphology of CNM as shown in fig below

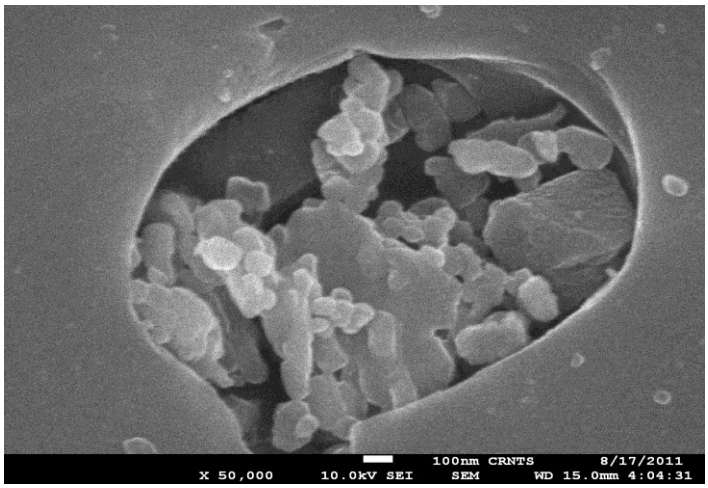

Fig.2 SEM image of CNM obtained from castor oil

FTIR STUDY

FTIR spectrum of CNM is as shown below. It shows a characteristics band of carbon at 1218 cm-1 which represents the presence of carbon only and the absence of any other impurities.

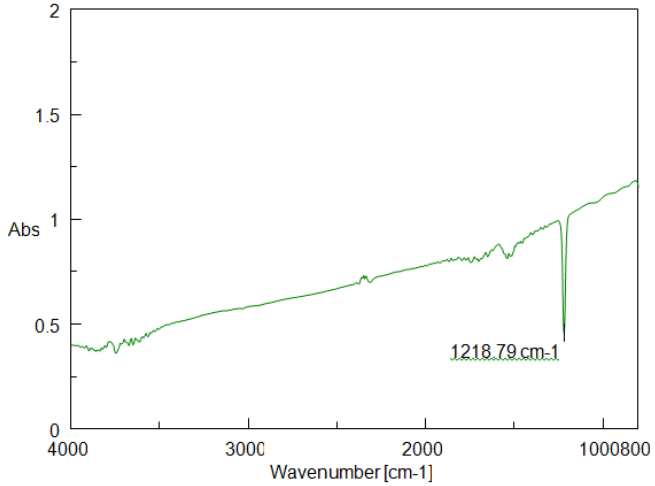

Fig.3: FTIR spectra of CNM obtained from castor oil

XRD STUDY

The diffractogram of CNM shows two peaks one at 26.46° and another at 44.38° which are the characteristics peaks of CNM.

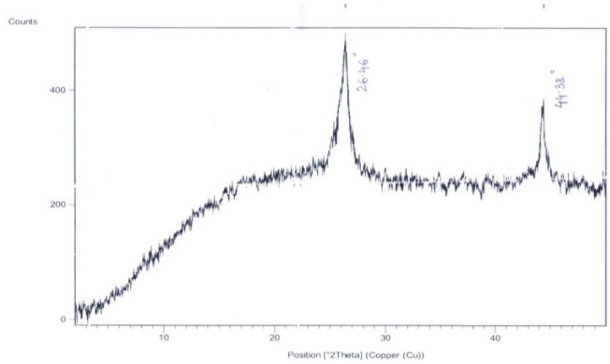

Fig.4.XRD diffractogram of CNM obtained from castor oil

SURFACE AREA MEASUREMENT OF CNM BY METHYLENE BLUE

CNM prepared by pyrolysis of castor oil is directly used for this experiment. Surface area of this CNM is measured using methylene blue adsorption method. Methylene blue solution was prepared in distilled water and the concentration of methylene blue solutions was analyzed by measuring its absorbance at 662 nm on UV/Vis spectrophotometer. This wavelength corresponds to the maximum absorption peak of the Methylene blue. First of all a calibration curve of O.D. of methylene blue against its standard concentration (1 to 5 ppm) is plotted and is shown in fig.5. The regression factor is found to be 0.99

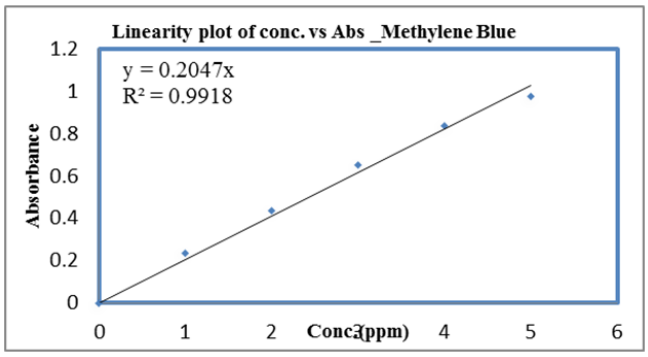

Fig.5. Calibration Curve of Absorbance Vs conc.

0.050 g of CNM is taken in 50ml conical flask containing 5 ppm solution of methylene blue. The mixture is stirred until all the CNM gets submerged into the solution. This mixture is kept at room temperature and shaken periodically for 10 mins and allowed to settle the small particals. After 10mins, the methylene blue uptake onto carbon was calculated from the difference between the methylene blue concentration before and after adsorption onto the carbon and difference corresponds directly to the active surface area of carbon.

The Specific surface area measured by using this technique is found to be 30.03M^2/g

IV. CONCLUSIONS

Adsorption of methylene blue allows the determination of the specific surface area of Carbon Nanomaterial. This work has shown that the method was simple and requires less elaborate apparatus and time than other methods. Using this technique, we have shown that the CNM obtained from castor oil have different specific surface areas. Consequently, this finding makes it possible to use specific surface area measurements directly as a characteristic in quality control, much like other mechanical properties of CNMs. This method was found to be comparable to that of existing BET surface area measurement technique.

Table 1 : Surface Area Results

		Surface Area m^2/g	
Sr.No.	Sample Details	BY BET	Methylene blue
1	CNM from Castor Oil	30.69	32.0

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