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Research Article

CARBON NANOSPHERE SYNTHESIZED FROM MAIZE COB-HAIR FOR ARSENIC ADSORPTION FROM WATER

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ABSTRACT

Carbon Nanospheres (CNS) are synthesized by pyrolyzing Maize cob-hair using the CVD method for two hours, at 10000C in presence of Ni, Fe, or Co nanometals as the catalyst. The morphology of carbon nanomaterial was characterized by the HRSEM images. The XRD pattern and the Raman spectrum confirmed the graphitic nature of the carbon. Surface area measurement of CNM was done by the Methylene Blue test method. The impact of catalyst was noted on the surface area of the carbon nanosphere; the maximum being 95.018 m2/g when Fe nanoparticle was used as catalyst, 83.141 m2/g when Ni was used, and a minimum of 21.379 m2/g in presence of Co. These carbon nanospheres were used for Arsenic adsorption from water. Adsorption of Arsenic was assessed by Chand Pasha et al's method. It was found that after 12 hrs of exposure, CNS with 95 m2/g surface area could adsorb 32% arsenic; CNS (of 23.8m2/g surface area adsorbed 31% whereas CNS (surface area of 21.379m2/g. could adsorb only 16% arsenic. The arsenic adsorption capacity was found to be affected by the available surface area.

KEYWORDS

Maize cob-hair, carbon nanosphere, chemical vapor deposition, CVD, Arsenic-adsorption

INTRODUCTION

Several different fabrication methods have been developed to synthesize carbon nanomaterials such as arc discharge, pulsed laser evaporation, chemical vapor deposition (CVD), etc. CVD is the most popular and accepted technique for the preparation of nano forms of carbon. It is a process in which chemical species react in the vapor phase to form a solid product on some surface. Kong et al. (1988) first reported the catalytic CVD (CCVD) synthesis of single-wall carbon nanotubes (SWCNTs) at 900 °C to 1000 °C. Soon after the discovery of CNTs by Iijima (1991), using the same technique, Ando and Ijima (1993) reported CNT grown from benzene using the arc-discharge method. Later, MWCNTs were also grown from ethylene (Hernadi 1996), methane (Satishkumar 1999), and many other hydrocarbons. Dai et al (1996) first produced SWCNTs from disproportionation of carbon monoxide at 12000C catalyzed by molybdenum particles. Later SWCNTs were produced from benzene, acetylene (Satishkumar 1998) ethylene (Hafner 1998), and methane (Kong 1998) also using various catalysts. Against the conventional use of these petro hydrocarbons; a green plant product: camphor was found to be an efficient material for producing high yields of MWCNTs by Sharon's group (Mukhopadhyay et al 1994). Sharon and his research group are the first to have explored the use of plant-derived metabolites for the successful synthesis of various forms of carbon by the CVD process e.g., C60 (Mukhopadhyay 1994), glassy carbon (Mukhopadhyay and Sharon, 1997), DLC film (Mukhopadhyay 1997), semiconducting carbon (Sharon 1999), carbon nanofibers (Pradhan et al 2002), spongy carbon, etc. In 1988 carbon nanobeads or spherical and spongy CNM were synthesized by Sharon et al (1998a and 1998b). The idea behind exploring the precursors which are derived from plants was that these precursors could be harvested as and when needed and even in the event of scarcity of petroleum-derived precursors, production of carbon nanomaterials would not suffer.

The different plant parts possess different carbon-rich biochemical and anatomical compositions. Pyrolyzed products from these portions of the plant are expected to give some unique carbonaceous structure that might have some specific utilities. Considering these views, it was thought to select some agricultural wastes and pyrolyze them in an inert atmosphere to get carbon nanomaterials. For the present work, one such waste agro product Maize Cob-hair that grows on the tip of Maize cob (Figure – 1) was selected as a precursor for the synthesis of carbon nanomaterial (CNM).

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Figure – 1: Maizecob-hair

MATERIALS AND METHODS

Precursor

Maizecob-hair bunch that grows on the tip of Maize fruit (Figure –1) was used as the precursor. These long shiny fibers at the top of an ear of corn is actually an elongated style of stigma. An ear of corn may have 300 to 600 hairs. An initial elemental analysis of hair showed that it is composed of 40.753% carbon, 12.672% nitrogen, and 4.977% hydrogen; making it a fairly good source of carbon.

Synthesis Procedure

As mentioned above CVD method was followed for the pyrolytic reduction of hydrocarbons present in the precursor into the nanocarbon.

Parameters

Parameters considered for CVD of Maize-hair were subjected to four different variables of the following parameters during the CVD process viz. reactor temperature, type of carrier gas, catalyst, and duration of pyrolysis.

Preparation of Catalysts

Three different catalysts (Nickel, Iron, and Cobalt) were used for the synthesis of CNM. Crystal metal powder of $Ni(NO_3)_2$, $Fe(NO_3)_3$, and $Co(NO_3)_2$ was separately mixed with urea in the proportion of 1:4 by weight, ground, and then dissolved in distilled water. The solution was stirred at room temperature for 10-15 minutes, to ensure that all the particles of metal salts were completely dissolved. The mixture was then dried and heated in a muffle furnace for about 3 hrs, followed by calcination in air at 6000C for 3 hrs. The resulting product was pure metal oxide i.e. nickel oxide, iron oxide, and cobalt oxide respectively. The metal oxides were then reduced in the atmosphere of hydrogen gas using a horizontal furnace at 6000C for 3 hrs.

The following reaction took place during the oxidation & reduction processes:

1. $Ni(NO_3)_2\ 6\ H_2O + (NH_2)_2.CO \rightarrow$ Ni-Oxide + $4\ CO_2 + NH_3$

Ni-O + $H_2 \rightarrow$ Ni- nanoparticles + H_2O

2. $Fe(NO_3)_3.6\ H_2O + (NH_2)_2.CO \rightarrow$ Fe-Oxide + $4\ CO_2 + NH_3$

Fe-oxide + $H_2 \rightarrow$ Fe- nanoparticles + H_2O

3. $Co(NO_3)_2.6\ H_2O + (NH_2)_2.CO \rightarrow$ Co-Oxide + $4\ CO_2 + NH_3$

Co-oxide + $H_2 \rightarrow$ Co-nanoparticles + H_2O

Preparation of Carbon Nano Materials

Details of the CVD setup are given in figure – 2. In the furnace, a cylindrical quartz tube was inserted and it was connected to the temperature controller. One end of the quartz tube was connected to the carrier gas i.e. Argon cylinder through a flow meter and the other end was connected through a tube with pressure gauze and a safety valve. Finally, the tube was inserted into a water trap through which the excess gas could be allowed to escape. One gm precursor was placed in a quartz boat along with a catalyst and the boat was placed inside the furnace. (Figure 2). The furnace was heated to different temperatures. It was kept at this temperature for three hrs. Prior to removing the sample, the furnace was allowed to cool down to room temperature. Thus synthesized carbon was scraped from the quartz boat and purified.

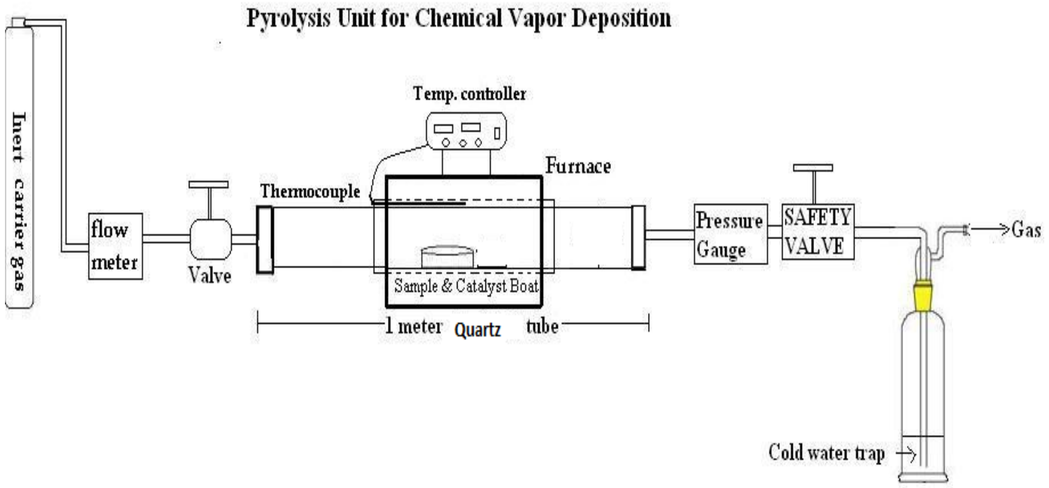


Figure -2: Schematic diagram of chemical vapor deposition setup used.

Purification of Carbon Nano Materials

To get rid of impurities like graphite sheets, amorphous carbon, the remnant of metal catalyst and another plant residue, etc; acid treatment was done. The synthesized carbon materials were first soaked in 100ml of 10M HCl and heated to 800C for 20 min. After keeping it at room temperature for 24 hrs it was filtered, washed with distilled water five times, and finally rinsed with acetone to remove the traces of water and then dried in an oven at 1200C for 24 hrs.

Characterization of Catalysts and Carbon

The morphological observations of catalysts (Ni, Fe, and Co) as well as synthesized and purified carbon materials were carried out by using a Hitachi (S-4700) High-Resolution Scanning Electron Microscope (HRSEM); by placing the samples on conductive carbon tape.

XRD of carbon nanomaterials was performed with a powder X-ray diffractometer with a CuKα source to calculate the crystallographic parameters.

Raman spectra were measured in a back-scattering geometry at room temperature using an Ar ion laser (488nm).

Measurement of Surface area of Carbon by Methylene blue method

To study the uptake of arsenic by CNMs it is necessary to know the surface area of the CNMs. The surface area of carbon material synthesized from Maizecob-hair using different catalysts was measured using the Methylene Blue adsorption method. The assessment involved:

Determination of λ max of Methylene blue dye solution

It was done by measuring the optical density of 0.1x 10-5 M to 1 × 10-5 M solution of Methylene Blue dye prepared in distilled water; using a Spectrophotometer. The λ max of the Methylene blue solution was found to be at 660 nm wavelength. At the λ max OD of Methylene blue solutions of different concentrations was determined and a graph of OD Vs. the corresponding concentration of the Methylene blue solution was plotted (Figure- 3 Left) and used as a standard graph to determine the concentration of Methylene blue in a solution containing an unknown concentration of Methylene blue.

Studying the equilibrium adsorption of Methylene blue solution –

To study the equilibrium adsorption of Methylene blue solution, CNMs synthesized from Maizecob-hair were heated in an oven for half an hour at 80oC to remove any previously adsorbed gases. After cooling, 5mg of CNM was added to 100 ml Methylene blue solution (1 x 10-5 M). At an interval of every 2 hrs, a small amount of the liquid was taken and centrifuged in a tube to settle down carbon material. The supernatant liquid was taken for measuring the OD at 660 nm wavelength of remaining Methylene Blue. The variations in the O D were plotted Vs. Time (Fig. 3 Right).

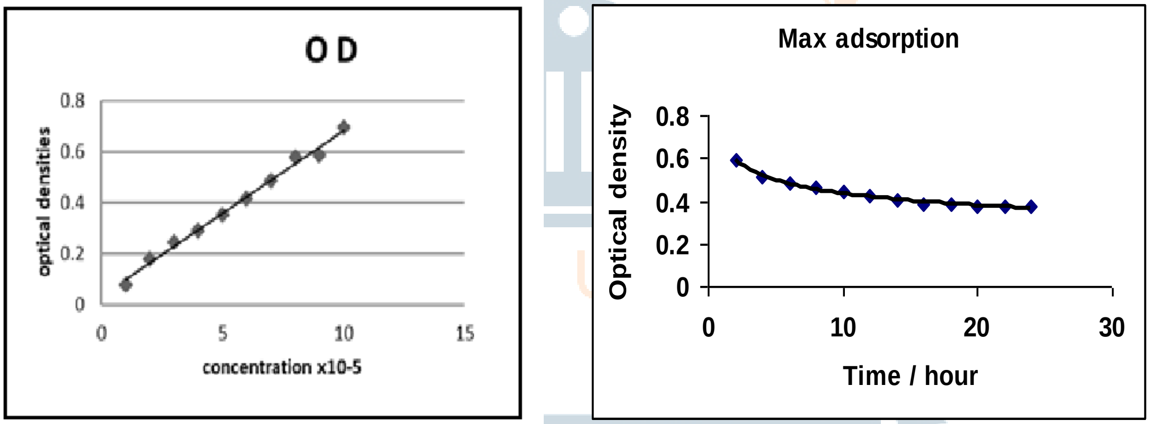


Figure - 3: (Left) Calibration Curve of Absorbance vs. Concentrations of Methylene blue (Right) Kinetic curve for adsorption of Methylene blue onto carbon material

Determination of Methylene blue adsorbed on CNMs to calculate Surface Area:

5mg of heated (at 80oC for half an hr) and cooled CNM was added to 100 ml of (1 x 10-5 M) Methylene blue solution. The mixture was stirred till the CNMs were submerged in the dye solution and kept at room temperature for 24 hours. During this storage period, the color of Methylene blue faded, which was due to the adsorption of dye on the surface of CNT. After 24 hrs the change in the Methylene blue color concentration remained constant as the equilibrium of adsorption reached after 24 hours. The optical density of the solutions was measured, to calculate the amount of Methylene blue left unused. The corresponding concentration of Methylene blue left was determined with the help of a standard graph (figure 3).

Calculation of the Surface area of CNMs:

It was done by estimating the concentration of Methylene blue adsorbed by the carbon material. Methylene blue uptake onto carbons was calculated from the difference between the Methylene blue concentration in solution, before and after adsorption by the CNM.

Therefore, the numbers of molecules adsorbed are;

$$n = \frac{6.023\times10^{23}}{M}\times\left(\frac{M\times C\times V}{1000}\right)grams$$

$$\therefore n = \frac{6.023\times10^{23}}{1000}\times\left(C\times V\right)grams$$

Here concentration difference is C moles/dm3

This was adsorbed by W grams of carbon material in V ml of solution

If the surface area of a single-molecule of Methylene blue is a then the total surface area of the adsorbed molecules will be,

$$= 6.023 \text{ x } 10^{20} CVa$$

This is the surface area of W grams of Carbon, so the surface area will be

$$SurfaceArea = \frac{6.023\times10^{20}\ CVa}{W}\quad A^{o2}/gm.$$

$= 6.023 \text{ x } 10^{20} \text{ x } 197.2 \text{ x } C \text{ x } V/ W \quad A^{o2}/gm.$

$= 6.023 \text{ x } 10^{20} \text{ x } 197.2 \text{ x } 10^{-20} \text{ x } C \text{ x } V/ W \quad m^2/gm.$

$= 1187.736 \text{ x } C \text{ x } V/W \quad m^2/gm.$

(Where the surface area of a single Methylene blue molecule is 197.2 Ao2)

Assessment of Arsenic adsorption by CNM synthesized using spectrophotometric method

For the present work, Pasha and Narayana's (2008) Spectrophotometric method using Safranin-O was used for the determination of Arsenic present in the water. The method is based on the reaction of arsenic

(III) with potassium iodate in an acidic medium to liberate iodine. The liberated iodine bleaches the pinkish-red color of Safranin-O. The decrease in absorbance at 532nm is directly proportional to the As (III) concentration and obeys Beer's Law.

5ml of a sample solution containing 0.1-1.0µgml-1 of As (III) was transferred into a series of 50 ml standard flasks; to which 5 ml of 2 %Potassium iodate and 5ml of 1M hydrochloric acid were added. The mixture was gently shaken until the appearance of yellow color indicated the liberation of iodine. This was followed by the addition of 2,5 ml of 0.01% Safranin-O and 10.0 ml of sodium acetate solution. The solution was kept for 2 minutes and made up to the mark with distilled water. The absorbance was measured at 532 nm against the corresponding reagent blank. Reagent blank was prepared by replacing the analyte (arsenic) solution with distilled water. The absorbance corresponding to the bleached color, which in turn corresponds to the analyte (arsenic) concentration, was obtained by subtracting the absorbance of the blank solution from that of the test solution.

The calibration curve passing through the origin was plotted by considering the different concentrations of arsenic on X-axis and the absorbance corresponds to the arsenic concentration on the Y-axis (Figure-4).

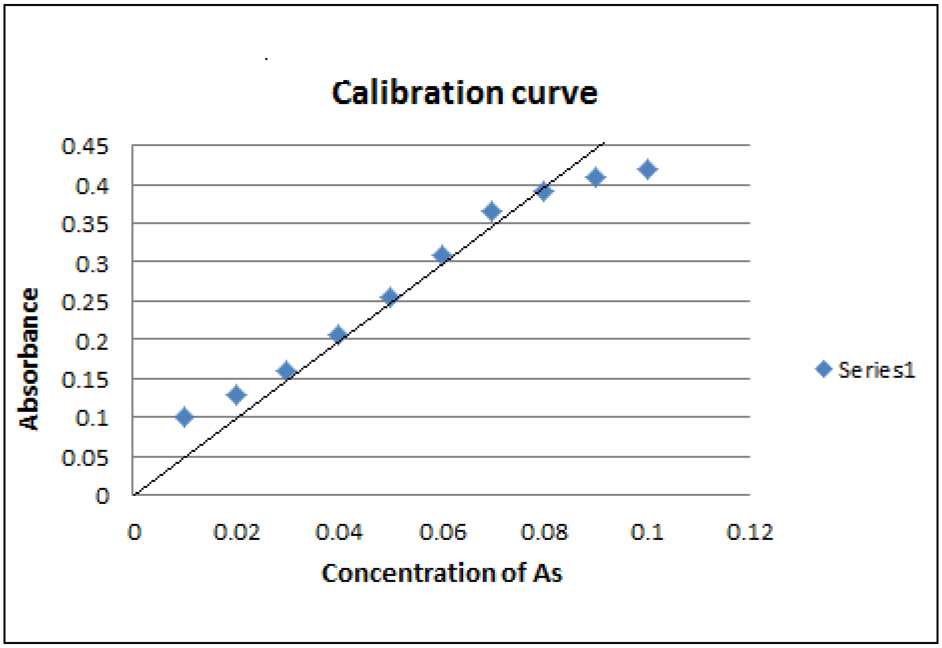


Figure -4: Standard Calibration Curve for As

To study the concentration of arsenic adsorbed by each sample of synthesized CNMs in 5.0 ml of 0.1 x 10-5 M As(III) solution, 5 mg of synthesized CNMs (synthesized in presence of different nano-metal catalysts) was added into a series of 50 ml calibrated flask and the mixture was kept for overnight. To this solution 5.0 ml of 1M HCl and 5.0 ml of 2% potassium iodate were added and mixed thoroughly; followed by the addition of 2.5 ml 0.01% Safranin-O and 10.0 ml of sodium acetate solution. The solution was kept for 2 minutes and made up to the mark with distilled water. The resulted solution was then filtered through Whatman filter paper no.41. The absorbance of the resulting solution was measured at 532 nm. From the observed absorbance of each solution, the concentration of arsenic adsorbed by the CNMs was calculated as % sorption.

The concentration of arsenic adsorbed by each sample of CNMs was correlated with their surface area determined by Methylene blue.

RESULTS AND DISCUSSIONS

Yield of Carbon

From the table-1, it can be seen that 29 to 36% CNS (carbon nanospheres) were obtained from the Maizecob hair. An initial elemental analysis of Maize hair has shown that it contains 40.8%Carbon, 12.7% Nitrogen, and 5% Hydrogen (Shukla 2012). This shows that out of 40.8% Carbon got converted to 24 – 36% CNM, thus making it a good precursor for the synthesis of CNM.

Catalyst is one of the important parameters, which controls the size and morphology of CNM produced by the CVD technique. Hence it was imperative to study the morphology of CNM obtained

Morphology of Carbon as observed under HRSEM

Cob hair was studied under HRSEM, before (Figure-5), and after pyrolysis using different catalysts (Figure-6). As it can be seen from figure -6, the HRSEM images show that in the presence of all the three nanometal catalysts i.e. Ni, Fe, and Co Carbon Nanospheres (CNS) were formed. However, their surface area varied. The Maximum being 95.018 m2/g when Fe was used as a catalyst and the minimum was 21.379 m2/g when Co was used as a catalyst. (Table-1)

Synthesis Parameters		Yield of Carbon/g cob-hair	Morphology of Carbon	Surface Area Of CNS	% Adsorption of Arsenic in 12 hrs.
Catalyst	Temperature				
Co	1000^{0}C	301 mg	Nanosphere *	21.379 m^2/g	16
Ni	800^{0}C	365 mg	Nanosphere *	83.141 m^2/g	32
Fe	600^{0}C	299 mg	Nanosphere *	95.018 m^2/g	31

Table-1: Yield. Surface area and Percentage of Arsenic adsorption by Carbon nanospheres (CNS) obtained by pyrolysis of Maize cob-hair using different catalysts.

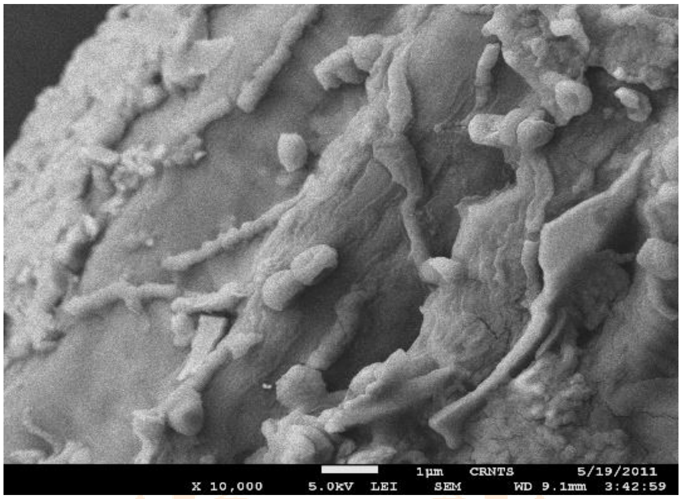
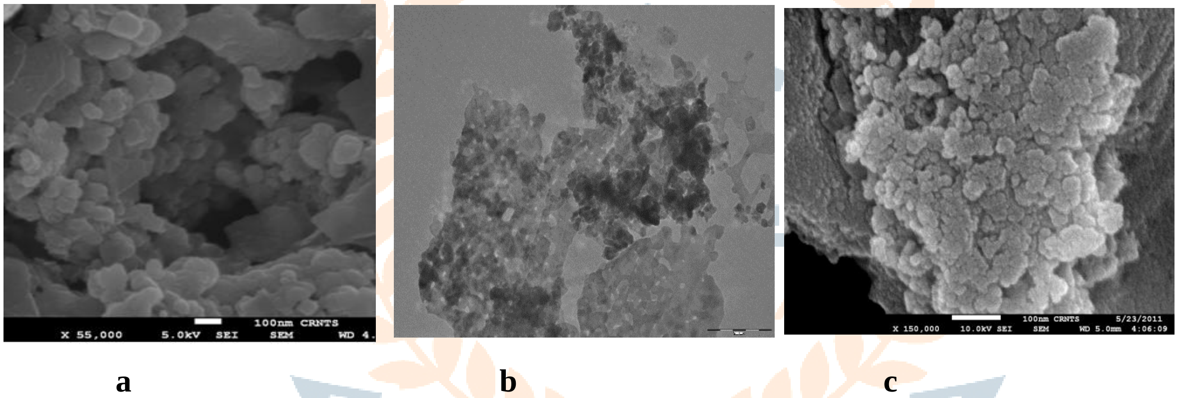


Figure – 5: Maize Hair Before pyrolysis



a b c

Figure – 6: HRSEM of CNM synthesized from Maize Hair using different nanometal catalysts showing the formation of Carbon nanospheres in presence of (a) Co synthesized at 10000C;(b) Ni synthesized at 8000C; (c)Fe synthesized at 6000C.

XRD Analysis of CNS

XRD pattern of carbon nanospheres, from Maize cob-hair, was recorded (Figure7). XRD analysis shows a diffraction peak at 260 {002}, However, the graphitic peaks are not very prominent in CNS samples

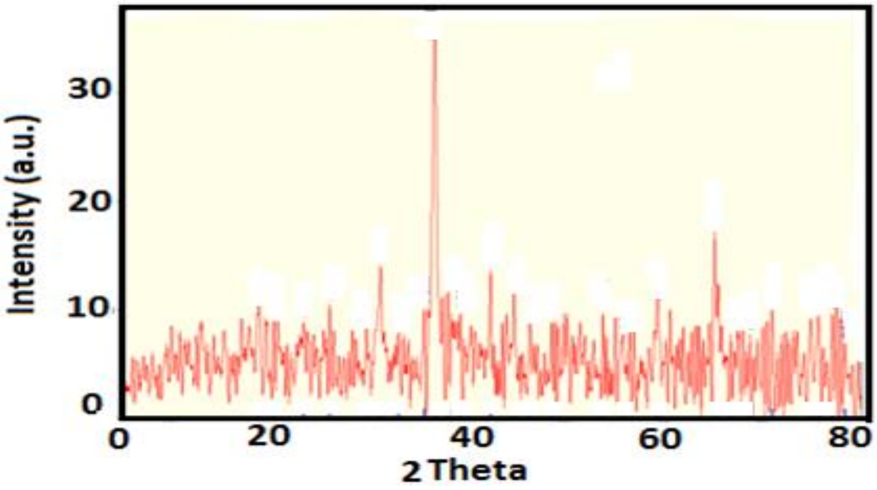


Figure-7: XRD pattern of CNS synthesized from Maize cob-hair

Raman spectroscopic analysis

Raman spectroscopic analysis of CNS shows the D band as well as the G band, thus confirming its graphitic nature (Figure-8). Raman spectroscopy is used to differentiate between graphite, multi-walled, and single-walled CNTs and layers. The Raman spectrum of the CNS sample (Figure – 8) shows two typical peaks of MWCNT one at around 1562 cm−1 and the other at around 1318 cm−1; which corresponds to the typical Raman peaks of graphitic carbon and defects in graphitic carbon respectively. The peak at 1562 cm−1 is attributed to the Raman active E2g in-plane vibrational mode and is related to the vibration of sp2 bonded carbon atoms in a two-dimensional hexagonal lattice. The peak at 1318 cm−1 is associated with vibrations of carbon atoms with dangling bonds in-plane terminations of disordered graphite.

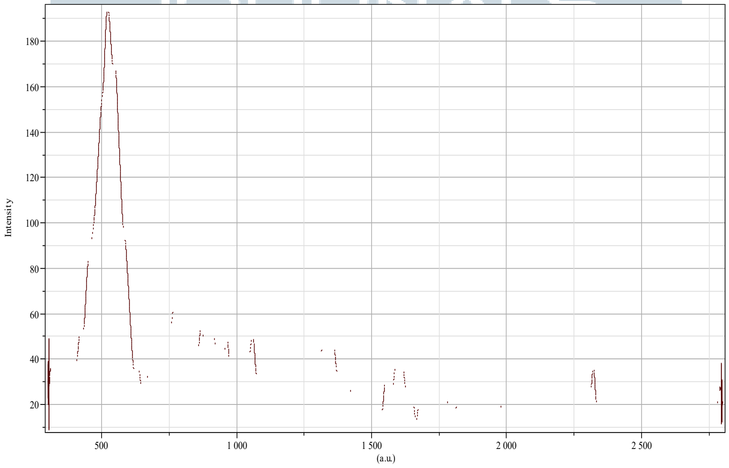


Figure 8: – Raman Spectrum of Carbon Nanospheres synthesized from Maize cob-hair.

Impact of Catalyst and Temperature on the Surface area of CNM

As far as the morphology of the CNM is concerned all three catalysts tried did produce spherical carbon nanomaterial, however, the temperature requirement for producing CNS varied for each of them, being 10000C for Co, 8000C for Ni, and 6000C for Fe. It was interesting to note that CNS produced at the lowest temperature had more surface area (Table-1). It can be concluded that surface area was dependent on the temperature at which the CNS was synthesized. Similar results were reported by Tripathi et al (2013) have also reported CNB/CNS synthesis using Neem oil as a precursor in presence of Co as a catalyst but at a much lower temperature of 5000C; whereas Castor oil produced CNS in presence of Ni at 7000C. It must be mentioned here that a catalyst not only seeds the formation of CNM but also increases the speed of a reaction during the synthesis of CNM by lowering the activation energy for the reaction, acting as a facilitator, and bringing the reactive species together more effectively, or creating a higher yield of one species when two or more products could be formed.

Arsenic Adsorption by CNS

Since Arsenic is a poisonous element that has detrimental effects on the health of living beings at high concentrations; hence the presence of arsenic in drinking water is very dangerous. Therefore, the use of CNS has been focused on the removal of arsenic from water. In the present study, the CNS synthesized from the waste material of corn cob hair using nanometal catalysts, is used as an adsorbent for the removal of arsenic from the aqueous solution spectroscopically. From the experimental data, it was observed that the surface area of CNS is an important factor in the adsorption of arsenic. The correlation between the surface area of CNS and arsenic adsorption by them is presented in table – 1. It was observed that the concentration of adsorbed arsenic on the surface of CNS is directly proportional to the surface area of each synthesized CNS; i.e. CNS that had a minimum surface area of 21.379 m2/g could adsorb 16% As, whereas having a higher surface area of 95.0 m2/g and 183.7 adsorbed as much as 31-32% As.

Many forms of carbon have been tried for the removal of As from water e.g. activated carbon by Ansari and Sadegh (2007); CNM by Ghanizadeh et al (2010); and CNT by Naghizadeh et al (2012)

CONCLUSIONS

Pyrolysis of waste part of Maize plant i.e. cob-hair using the CVD method, and three different nanometal catalysts, namely Co, Fe, and Ni, produced temperature-dependent Carbon nanospheres. The impact of these three catalysts was noted on the yield, and surface area of produced CNS. XRD and Raman spectroscopic analysis of CNS showed it had graphitic nature of carbon. The CNS with a higher surface area was found to adsorb more Arsenic from water. This is attributed to its highest surface area of 95m2/g.

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