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SYNTHESIS OF CARBON NANO BEADS AND ITS CHARACTERIZATION

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Abstract

Carbon Nano materials have diverse tuneable physical properties as a function of their size and shape due to strong quantum confinement effect and large surface to volume ratio. One of the Carbon Nano material is Carbon Nano Beads (CNB) which are spherical, solid or hollow structures.

In the present work carbon nano beads were synthesised by chemical vapour deposition method using cobalt nano catalyst. The surface morphology of nano catalyst and nano beads prepared were studied by Scanning electron microscopy.

Key words: Nano materials, Chemical vapour deposition, Carbon nano beads, Nano catalyst

INTRODUCTION

One of the important question that scientist's working with CNM is, can it be used as drug delivery vehicle (S. Parihar *etal,* 2005). The nano scientist are fantasizing that ones CNM (Carbon Nanomaterials) along with drug is introduced into living system it can act as a self-driven syringe and deliver the medicine to the site of requirement. Not only that they are planning to make CNM a disposable syringe which can be removed from the system either by degradation or excreting it from the system. There has been reports about Carbon nano material being a cytotoxic material (S. Manchanda *et al*, 2021).

Morphologically Carbon Nano material exists in different shapes like tubular, spherical or fiber like structures. Carbon Nano Beads (CNB) are spherical solid or hollow structures where 5 - 7 beads are covered by a broken graphene sheet.

Properties of CNM

Some important properties of carbon nano materials are discussed below

1. Chemical Reactivity:

The chemical reactivity of CNM is usually more than that of the graphene sheet. CNM reactivity is directly related to s-orbital mismatch caused by an increased curvature. Therefore, a distinction must be made between the sidewall & the end caps of nano tubes for the same reason. A smaller nano tube diameter results in increased reactivity. Covalent chemical modification of either sidewall or end caps is possible.

Though CNMs are usually insoluble material, however the solubility of CNMs in different solvent can be induced or enhanced by creating chemical bonds.

2. Surface states/Dangling Bonds /Lewis acid/base:

In the case of carbon, the specific cluster C 60 do not have dangling bonds as the carbon cluster is arranged in geometrical form to eliminate all dangling bonds. The only reasonable structure was a spherical one—soccer ball—in which each carbon atom had the full complement of electrons.

But in case of carbon nanomaterials, due to its structure the surface Carbon atoms are not having the similar electronic environment as the atoms of the inner carbon atoms. Because of which some surface Carbon behaves as Lewis acids and some as Lewis base. This helps in covalent or non covalent bonding on the surface.

3. Mechanical strength:

CNTs have very high tensile strength; hence they exhibit very large young modulus in their axial direction. CNMs are highly flexible; therefore, they are potentially suitable for applications in composite material that need an isotropic property.

METHODOLOGY

CNB was prepared by CVD method (M. Sharon et al 1998) using the precursor mustard oil and the catalyst used was Cobalt.

Catalyst preparation by urea decomposition method

Cobalt Catalyst was synthesized by the urea decomposition method. 10gms of Cobalt nitrate [Co (NO3)2 .6H2O] was mixed with 30gms of urea in proportion of 1:3 by weight, grinded and then dissolved in distilled water. The solution was then stirred at room temperature for ten minutes in order to ensure that all the transition metal salts had completely dissolved; then the mixture was heated in air to get dry powder. This powder was slowly heated at 500°C in a muffle furnace for about 3hrs. The resulting product was metal oxide. Care was taken to prevent loss of material while heating, because during heating material has a tendency to froth like volcano. The metal oxides were reduced in hydrogen atmosphere using a horizontal furnace at 700°C for 3hrs (Fig. 1) by connecting the quartz tube (C) with hydrogen gas. The following chemical reactions take place during oxidation and reduction processes.

$$Co(NO_3)_2 \cdot 6H_2O + (NH_2)_2CO \xrightarrow{\text{Co-oxide}} 4\,CO_2 \uparrow + NH_3 \uparrow$$

$$\text{Co-oxide} + H_2 \longrightarrow \text{Co-Nano Particle}$$

The size of the particles was determined from SEM images.

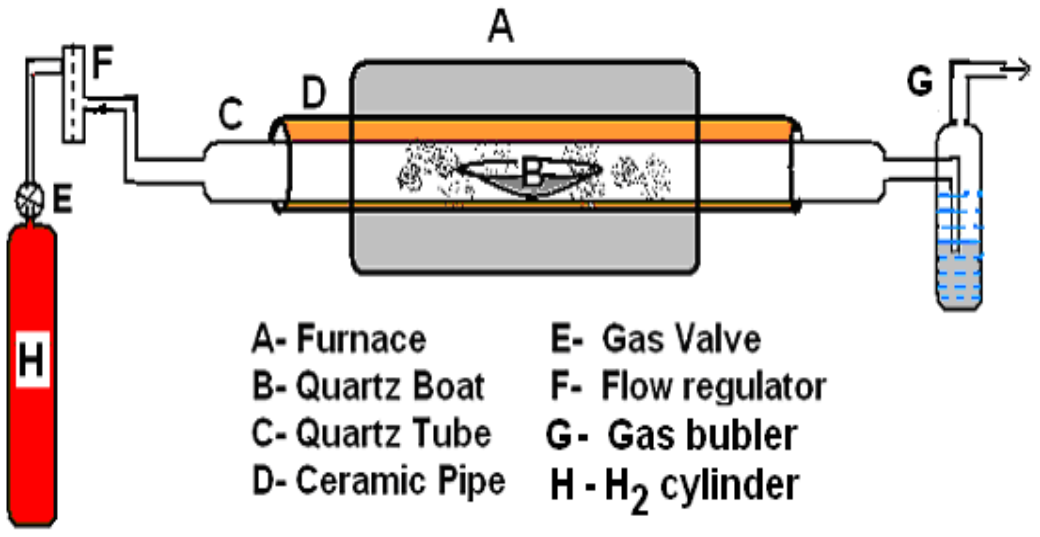

Figure 1 Schematic diagram of a furnace used for the reduction of metal oxide powder and for synthesis of CNB

Protocol for Synthesis of Carbon Nano Beads (CNB)

Preparation of CNB from terpene oil was done by pyrolyzing it in an inert atmosphere of nitrogen in horizontal furnace (Figure 1) at high temperature.

A cylindrical furnace controlled by PID controller to the accuracy of ± 1°C was used for the pyrolysis. A cylindrical quartz tube was inserted into the furnace. The one end of the quartz tube was connected to the nitrogen gas cylinders through a flow meter and the other end of the quartz tube was connected with water bubbler through which the excess gas could be allowed to escape to atmosphere.

Mustard oil, the precursor for carbon nano beads was kept in a quartz boat and inserted in the quartz tube. The positioning of the precursor containing boat was placed in the tube at such a position that it could get uniform temperature. In another quartz boat, the catalyst Cobalt was kept.

After keeping the material in the furnace, carrier gas was passed to flush out the air. After few minutes of flushing, furnace was switched on and when the required temperature of 7500C had reached, it was kept constant for the

desired duration i.e. 2 hours for continuing the pyrolysis and then the furnace was switched off and cooled to the room temperature. Carbon material deposited on the catalyst containing quartz boat was taken out from the quartz tube.

Purification of Carbon Nano beads

CNB synthesized contained some amorphous carbon and also metal catalyst as an impurity. The following procedures were adopted for the removal of impurities from CNB.

Soaking in acid

CNB obtained by CVD method was soaked in 300 cm^3 of 12M nitric acid in a 500 cm^3 beaker and kept at room temperature for 12 h and then filtered.

Removing acids

The acid treated carbon nano beads were washed with distilled water for five times (till neutral pH) and filtered. Finally, it was rinsed with acetone (99.5% pure) to remove the traces of water and then dried in an oven at 150°C for 24h.

Morphological observations of as-prepared and purified carbon nanofibers were carried out by SEM images.

Morphological Characterization of synthesized CNB was done by Scanning Electron Microscopy.

Scanning Electron Microscope (SEM) observation was conducted on FEI Quanta 2.

Sample for SEM was prepared by the following procedure:

The filter paper containing the sample was placed in a Petri dish containing acetone. The separated material was teased into small pieces. The sample material was placed on copper grid and air dried. The sample was labelled and stored until subjected to SEM.

First the grid with the sample on it was observed under a light microscope. The grid was then placed on a square block and then affixed. The block was fixed at the end of a stainless steel rod. The rod with grid was then inserted in SEM. The vacuum was switched on for 5 minutes to reach 5 Pascal. Sample was inserted by pushing the rod further and was scanned under the scanning electron microscope.

RESULTS AND DISCUSSIONS

Scanning electron micrographs of Cobalt (Figure 2) shows nano structured Cobalt catalyst and CNBs (Figure 3) reveals that they are spherical bead like structure attached to some graphitic structures. It was difficult to separate each bead even by sonication.

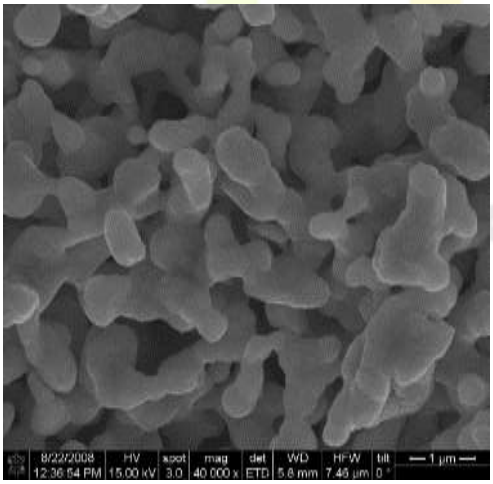

Figure 2 SEM of Cobalt Catalyst

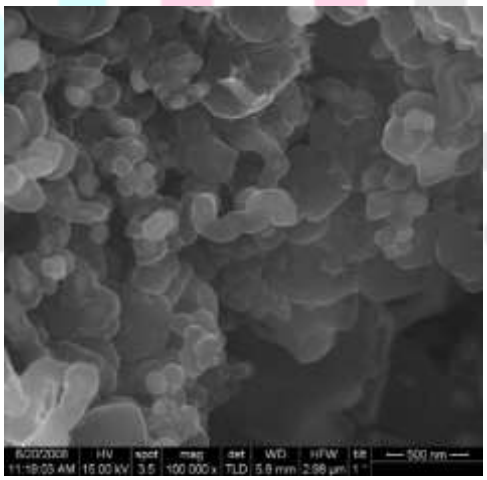

Figure 3 SEM of CNBs prepared by CVD method

CNB that was synthesized by CVD method using Mustard oil as precursor and Co as catalyst was found to be in the range of 80 – 100nm in diameter. However, they were in the form of clusters and many of them were joined together at the base.

CONCLUSION

With the advent of nanomaterials; interdisciplinary research has taken a big leap. The present work was a small effort to enter in this magnificent field of science having immense possibilities. Application of Nanotechnology in medicine found its way in late 1970s (Birrenbch and Speiser 1976 and. Kopf et al 1976, 1977). Initially polymers having capacity to form micelle, and later polymers having dendritic nature were tried for this purpose.

After going through the books and published papers it was realized that apart from nano-sized organic polymers; nano-metals and carbon nano materials are also being considered as possible drug delivery vehicle e.g. Fullerenes & CNT (Bianco et al, 2005)

In the present work the effort was to synthesize Carbon Nanobeads from natural oil using nano sized cobalt catalyst.

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