SINGLE, DOUBLE, MULTIWALLED CARBON NANOMATERIALS AND THEIR APPLICATIONS

Abstract

Carbon nanotubes (CNTs) are a primary nanomaterial that have outstanding physical and mechanical characteristics, and CNTs can be combined with cement-based materials to alter their heating characteristics. In this proposed we have planned to synthesis helical multiwalled carbon nanotubes (hMWCNTs) using solution combustion process. The synthesize materials were characterised by XRD, FESEM, TEM, Raman Spectroscopy. Experiments will be performed to determine the altered heating characteristics of the CNT cement mortars, medicine, energy and biological .application was more effective for modifying the heating characteristics compared to mixing SWCNTs and HMWCNTs with water with well-dispersed hMWCNTs obtains excellent mechanical and electrical properties, which lay a foundation for the utilization of hMWCNTs composite as the electrothermal materials in practical engineering.This review is discussing about the synthesis, advantages, disadvantages, applications, and effectiveness of the Helical multiwalled carbon nanotubes (CNT'S) synthesized by chemical vapor deposition method.

Keywords: CNT, HMWCNT, CVD synthesis, CNT applications, TEM.

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I. INTRODUCTION

Carbon nanotubes (CNTs) is emerging in the nanomaterial engineering and nanotechnology development. In past two decades, multiwalled carbon nanotubes have received a good scope and PubMed database showed an increase in CNTs research works. There is a gradual increase in publication in PubMed Database, on searching the keyword 'Carbon nanotubes" it displays 838 articles in 2005 whereas 2421 articles in 2022. This replicates that the CNTs synthesis and its application in various field were the recent trend of research. CNTs were first synthesized by Iijima.H in the year 1991, who tried to synthesis single walled CNTs, which exhibited excellent reinforcing property.[1] CNTs displays extraordinary properties like electrical, mechanical and thermal properties, chemical stability which contributed its research and development in various field of physics, chemistry, medicine and material science [2-6]

The CNTs are composed of carbon atoms arranged in benzene rings forming the graphene sheets, rolled to form cylinder or bundle. There are two types of CNTs, single walled carbon nanotubes (SWCNTs) and multiwalled carbon nanotubes (MWCNTs). Carbon nanotubes diameter ranges around 0.4 nano meter, theoretically and diameter reaches up to 1.2 nm based on the method of synthesis. [7] CNTs are available in various forms and arrangements such as straight, branched, bamboo, fibre, coiled and helical. MWCNTs are composed of concentric cylindrical graphite tubes of SWCNTs and it is easier to synthesis MWCNTs than SWCNTs in large scale. [8, 9]

1. Characterization of catalyst and carbon nanotubes

X-Ray diffraction (XRD) pattern of catalyst was obtained using a Cu-K radiation 9 0.154 1 nm). Scanning electron microscopy (SEM) image of the resulting product was measured. Transmission electron microscope (TEM) was performed with a microscope operating at 200 kV. Raman spectroscopy analysis of CNTs was achieved by Raman spectroscopy equipped with liquid nitrogen cooled Ge diode detector A with an output power of 350 mW Nd: YAG laser (wavelength = 1.06 µm) focused onto the samples.

A. Synthesis and Characterization of catalyst and CNT's

Various methods were available for the synthesis of single, double and multi-walled CNTs. They are

- Physical,chemical,biological method
- Precipitation method
- Solgel method
- Solvothermal&HydroThermal method
- Carbon Arc discharge[1,10,11]
- Laser ablation [12, 13]
- Catalytic Chemical Vapour Deposition (CVD) [14, 15]
- Plasma enhanced CVD
- Radio frequency enhanced CVD (RFECVD)
- Electrochemical method

Each methods have specific steps to synthesis the particular functionalized CNTs with own merits and demerits. Above synthesize methods was explained briefly in this article.

2. Physical & Chemical Method

The first step is the evaporation of the material and the second step involve a rapid controlled condensation to produce the required particle size.

3. Precipitation Method

This method involves dissolving a salt precursor of desired metal (in the form of chloride, nitrate or acetate) in water or any other desirable solvent. The resulting metal precursor is precipitated with strong or mild base to give its metal hydroxide. After precipitation, the powder is separated and washed in order to remove alkali ions, reagent anions, and excess deposit on the outside of the particles. Deposition occurs at two steps; firstly sols precipitate in bulk and pore fluid, and then interact with support surface. If hydroxyl groups of the support surface enter into reaction so that the pH of the surface region is higher than in the bulk solution, best results can be obtained. Then, precipitation occurs preferentially and uniformly on the surface.

Rapid nucleation and growth in the bulk solution may cause sols to be too large. In that case dilute alkali may be added, drop by drop, with rapid agitation to disperse the droplets before local concentrations become excessive. Alternatively, both the salt and alkali mixed in a container of water. Very often, control of size and chemical homogeneity in the case of mixed metal oxides are difficult to achieve. However, the use of surfactants, sono chemical methods, and high-gravity reactive precipitation appear as novel and viable alternatives to optimize the resulting solid morphological characteristics. Precipitation is the preferred deposition route for loadings higher than 10%-20%. Below this value, other techniques are usually practiced (Richardson 1989).

4. Solgel Method

Process is a more chemical method (wet chemical method) synthesis of various nanostructure especially metal oxide nanoparticles in the method the molecular precursor (usually metal oxide)is dissolved in water or alcohol and converted to gel by heating and stirring by hydrolysis /alcoholysis .[55]

5. Solvothermal & Hydro Thermal Method

Solvothermal Techniques changes to Hydrothermal techniques when the solvent used in water the process involves the mixing of the precursors in the solvent and then sealing then in an autoclave. The autoclave is then heated at temperature above the boiling point of the solvent.[55]

II. CARBON ARC DISCHARGE METHOD

Fullerene based double walled carbon nanotubes are synthesized by arc-discharge method. In this method anode and cathode was added in ratio of 2:1 to synthesize high quality CNTs at 85-110 A and 30-40 V with 2µm gap between anode and cathode. Slurry is prepared by soaking fullerene derived soot in 200ml of ferric chloride solution under stirring. Another 200ml of sodium hydroxide solution is slowly added into the slurry to make paste in a low-pressure container. About 100-150gm of DWCNTs was synthesised on a single run. The HR-SEM images shows exibit the particle size and morphology of the catalyst (Fig 2). The particles were observed to be relatively uniform in size, indicating good dispersion, which is essential for maximizing the active surface area. favourable for improving reactant accessibility and enhancing catalytic activity [1,16]

1. Laser ablation method

Graphite rods are melted using laser to prepare CNTs. In this method, catalyst like cobalt and nickel were mixed in the ratio of 50:50at 1200uC and heated under vacuum to remove the fullerenes. A second laser pulse is sent to break the bigger particles and ablate them into growing nanotubes. This method produces 10-20mm wide and 100mm length carbon nano ropes which are mostly SWCNTs. Both the arc discharge and laser ablation methods requires tedious purification and growing process, which makes questionable large-scale production of CNTs [17]. Increase in the temperature changes the CNTs growth rate, alignment, location and length of the CNTs. Laser ablation produces nanotubes up to 90% purer than arc discharge method. TheHR-SEM Images particles size observed to be relatively uniform in size, indicating good dispersion, which is essential for maximizing the active surface area.(figure -6).

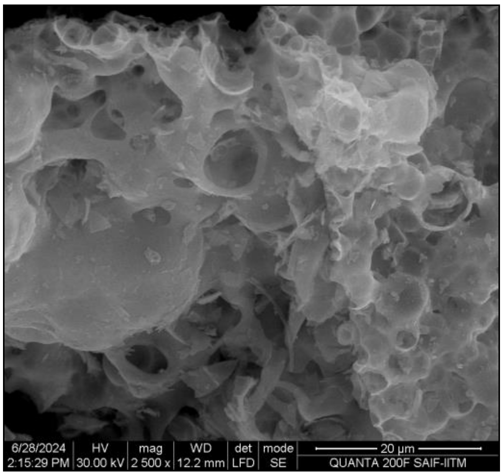

Figure 1: Schematic representation of Laser ablation method & HR-SEM LAM Method images OF 20µm

2. Chemical Vapour Deposition (CVD)

CVD is cost-effective, reproducible, easy method for synthesizing CNTs. CNTs were prepared in three steps by CVD process, they are

Step 1: Synthesis of catalysts
Step 2: Growth of CNTs
Step 3: Purification of CNTs

3. Synthesis of Catalyst

CNTs catalyst can be prepared by using single/biocatalyst can be used and they are copper, nickel, iron, aluminum. An aqueous solution of iron nitrate, magnesium nitrate, and ammonium molybdate were mixed together and citric acid was added to the prepared solution followed by 10 minutes Sonication. The prepared solution may be directly fed into furnace and fired or sintered at 450/550°C for 2-3 hours. After the combustion process, the product undergoes pulverization using mortar. The weight proportion of catalyst and temperature plays main role in % carbon yield. Temperature above 650°C minimizes the yield of CNTs [18,19].

4. Growth of Helical-Shaped MWCNTs

The CVD process was carried out at atmospheric pressure in a horizontal quartz reactor. About 100 mg of fresh catalyst was placed on a quartz boat located at the middle of the reactor. These FeMo/MgO powder were loaded immediately into the CVD chamber. The reduction process of the catalyst powder was done by purging H2 gas at 800◦C for 30 min. Then at the same temperature, the mixture of N2 and C2H2 gas was purged into CVD chamber at a flow rate of 200 mL/min and 60 mL/min, respectively. After the reaction completed, acetylene flow was stopped, whereas nitrogen flow was maintained to cool down the furnace. The xrd results shows well crystalline observed and this is the proof of absence of amorphous carbon clearly mentioned .The surface morphology of roughness and the presence of mesopores suggest that the catalyst has a well improved size nm and porous structure.

5. Purification of Helical-Shaped MWCNTs

The as-produced CNTs were dipped and stirred in warm HCl aqueous solution (36%) at around 60◦C for 30 min in order to extract the CNTs by dissolution of MgO and part of the Fe and Mo. The product was then rinsed with distilled water until the filtrate become neutral and dried at 120◦C to obtain purified CNTs. The surface morphology of the formed CNTs was analyzed by HRTEM, and Raman spectroscopy The synthesis of helical CNTs using a FeMoMgO catalyst can influence these properties and the Raman spectrum serves as an effective method for characterizing these effects. The D-band, typically observed around 1309 cm^{-1}, is associated with the disorder in the graphite-like structure of carbon Fig(6e,f). Fiber like Carbon nanotubes (f-CNTs) are fabricated on Cu–Mo/Al2O3 nano-powders catalyst.

The CVD procedure was performed in a horizontal alumina tube furnace. About 100 mg of Cu–Mo/Al2O3 catalyst in quartz boat was positioned at the centre of the alumina tube. Furnace temperature was increased slowly up to the reaction temperature, under continuous N2 flow. Then N2 flow was blocked when it attains the reaction temperature and feedstock gas of acetylene was then passed with different flow rates to fabricate CNTs. Once reaction was concluded, feedstock flow was terminated and N2 flow was continuously purged to CVD chamber till to the room temperature. The obtained carbon product yield subsequent to the reaction was considered using the below where m_{cat} and m_{tot} are the mass of the catalyst before and after the reaction, respectively. The preliminary percentage yield of carbonaceous deposit obtained after CVD was 500% on the basis of the weight of catalyst used [20].

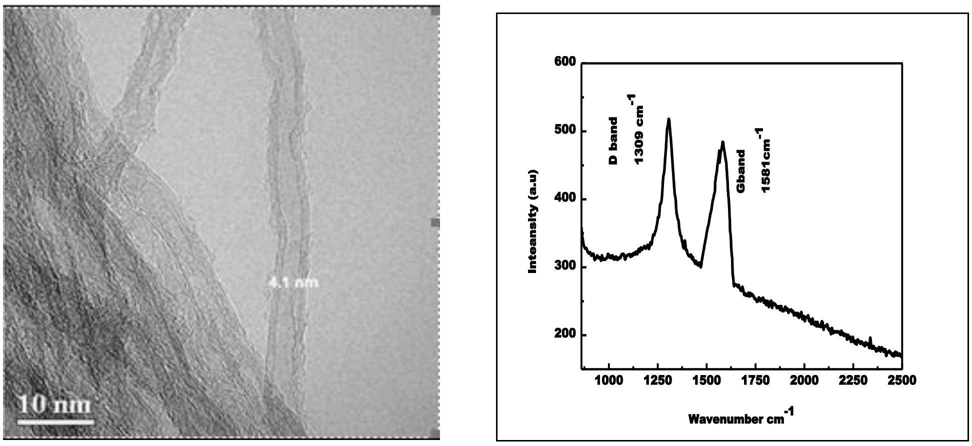

Figure 2 : TEM image of 10 nm CNTs with lower diameter& Raman spectroscopy CVD Method [23]

III. PLASMA ENHANCED CVD METHOD

Plasma-synthesized p-CNTs were compared to widely available CVD-produced CNTs (n-CNTs). MWCNTs. shows this study's two CNTs' basic features. n-CNTs typically have N90% purity and 140 m2/g surface area. However, p-CNTs have the same outer diameter but 180 m2 /g surface area, which may increase cement matrix bonding. p-CNTs have a high surface zeta potential of 33.2 mV, which implies a greater repelling force between them, improving dispersion. This study's two CNTs' basic features. n-CNTs typically have N90% purity and 140 m2/g surface area. However, p-CNTs have the same outer diameter but 180 m2 /g surface area, which may increase cement matrix bonding. p-CNTs have a high surface zeta potential of 33.2 mV, which implies a greater repelling force between them, improving dispersion.

This work used a 70-W ultrasonic bath for diffusion. One n-CNTs solution used a polycarboxylate-based superplasticizer as solvent, The polycarboxylate-based superplasticizer disperses CNTs in water for cementitious materials. Dispersed liquids were placed in 100 cm3 glass jars and left unagitated for 3 days at room temperature. Sedimentation was observed for 3 days after ultrasonication. To assess CNT deposition over time, 20 cm3 of liquid was removed from the top of the CNT solution. Ultracentrifuge centrifugation is another way to test CNT solution stability. [21] SEM Images surface morphology of size and structure clearly displayed at fig(7).

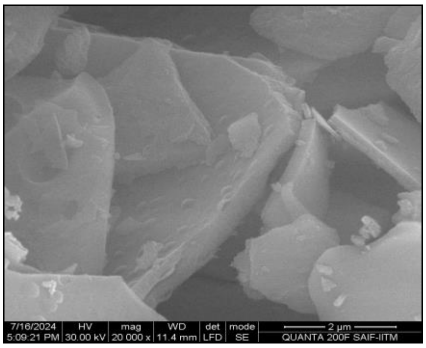

Figure 3 : (Plasma Enhanced CVD Reactor) & HR-SEM image at 02µm

1. Radio Frequency Enhanced -CVD (RFE- CVD)

Ultra-pure semiconducting carbon nanotube blend Quartz lowered capacitance. To make a uniform, high-density network, ultra-high-purity semiconducting carbon nanotube fluid was mixed 10 times with xylene. Quartz surfaces were washed with pure xylene after 1.5 hours in the reduced solution. Quartz surfaces were heated for 1 hour at 200 °C to clear liquids.RF diodes Source and sink electrodes were electron-beam-deposited titanium/palladium (1 nm Ti/50 nm Pd).Oxygen plasma cut outside channel carbon nanotubes. Electron-beam lithography created the T-shape gate, then 140 nm aluminium was heated vaporised and oxidised in air at 120 ° C. Electron-beam depositing a 10-nm fig(4) palladium film as self-aligned source and drain contacts cut channel length to 120–140 nm. [21]

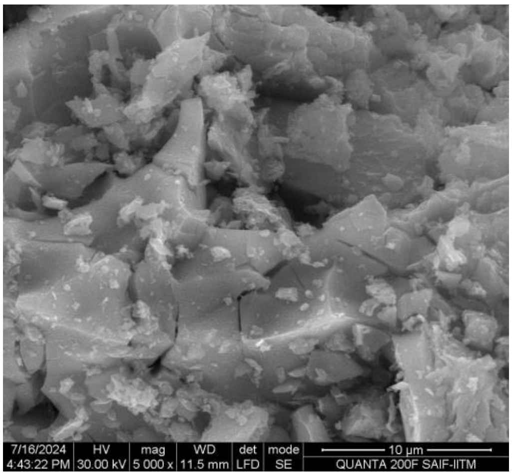

Figure 4: HR-SEM IMAGES OF 10µm.

2. Electrochemical Method

Catalytic CVD of acetylene over cobalt and iron catalysts on silica or zeolite produces lots of CNTs. The catalyst's cobalt amount affects carbon deposition activity, while catalyst preparation pH affects CNT specificity. The carbon/zeolite catalyst created multi-walled nanotubes with fullerenes and single-walled nanotube bundles. Researchers are trying to make CNTs from methane. In the ethylene atmosphere, supported catalysts like iron, cobalt, and nickel, which contain one or more metals, seem to cause the growth of solitary or bundled single-walled nanotubes. Molybdenum and molybdenum-iron metal catalysts can produce single- and double-walled nanotubes. Carbon was CVD in thin alumina template holes with or without a nickel catalyst.

Nickel-catalysed CVD used 545uC ethylene and 900uC uncatalyzed. Carbon nanoparticles without tips are produced. Carbon sources include methane. It has produced "nanotube chips" with isolated single-walled nanotubes at regulated places. Catalytic decomposition of H2/CH4 over well-dispersed metal particles like cobalt, nickel, and iron on magnesium oxide at 1000uC gives high rates of single-walled nanotubes. Selective reduction in an H2/CH4 atmosphere of oxide solid solutions between a nonreducible oxide like Al2O3 or MgAl2O4 and one or more transition metal oxides yields composite solids with well-dispersed CNTs. At.800uC, the reduction creates tiny transition metal fragments.

CH4 decomposition over newly formed nanoparticles stops their growth and produces a high percentage of single-walled nanotubes and few multi-walled ones. Ball milling Ball rolling and heating produce CNTs easily. Thermal annealing of carbon and boron nitride powders created [21].

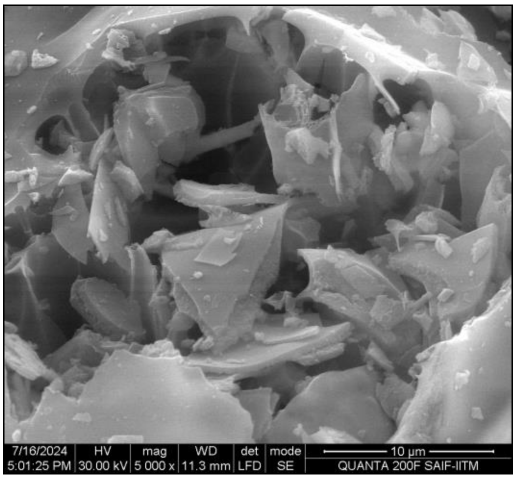

Figure 5: HR- SEM-IMAGES OF at 10µm

IV. CONCLUSION

MWCNTs have tremendous properties which made them utilize in wide range of fields. The use of MWCNTs has increased in the field of building construction. The present study discussed the comprehensive review of CNTs synthesis. MWCNTS is a potential fullerene and generates wide range of application in the field of Nano chemistry, biological, physics, medicine, in particular construction. Various researches were carried to justify those applications. Inclusion of MWCNTs with cement paste/construction material shown a significant effect has been discovered. It describes about the advantage of PECVD technique. Then we described about the simulation of CNT structures. We have calculated the structural parameters of types of CNT's. Then we mentioned about the various tube distortion in CNT and calculated energy per atom with respect to radius. We reviewed two important applications of CNT and finally some of the properties of CNT being mentioned and simulated according to the structure. Debonding and crack bridging of MWCNTs are the main reason for the enhancement of matrix toughness.

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