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# A STUDY ON DISPERSIONS OF MULTI-WALLED CARBON NANOTUBES (MWCNTS)

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## ABSTRACT

Carbon nanotubes (CNTs) have attracted great attention for their use as effective electrocatalysts due to their distinctive electrical characteristics. High electrocatalytic activity of nitrogen doped CNTs for oxygen reduction has been demonstrated. They came to the conclusion that the presence of electron-accepting nitrogen atoms made it easier for the nearby carbon atoms to have a relatively high positive charge density, which opened up additional electron pathways for oxygen reduction. Additionally, theoretical calculations carried out have demonstrated the thermal dissociation of water at lower temperatures (1000 K) in the presence of CNT, whereas the temperature required was 2000 K when graphite was used as a catalyst. The dissociation of water on charged defective single walled carbon nanotubes. A MWNT is made up of closed graphite tubules with a typical diameter of the order of 10 to 25 nm and an interlayer spacing of 3.4, which is similar to the interlayer spacing in graphite. Due to their multiple cylinders made up of various helicieties and general metallic conductivity, MWNTs complicate any straightforward description of their electrical properties.

**KEY WORDS: Carbon nanotubes, Single-wall, Multi-wall, Solvent.**

## INTRODUCTION

Due to their distinctive structure and properties, many nanocarbons such fullerenes, nanotubes, and related carbon crystals have attracted a lot of attention in terms of discovery, synthesis, and characterisation. Carbon nanotubes (CNTs) are gaining significant attention in science and technology among the carbon-based materials. Single-wall carbon nanotubes (SWCNTs) and multiwall carbon nanotubes are the two different forms of CNTs (MWCNTs). SWCNTs are made from a single graphene sheet, whereas MWCNTs are made from several

graphene sheets [3]. Due to their large micrometre lengths and nanoscale level diameter, SWCNTs and MWCNTs exhibit high aspect ratios. Additionally, MWCNTs' fine dispersion can compensate for their low aspect ratio.

Entangled and bundled MWCNTs are produced as a result of the high aspect ratio and robust van der Waals interactions. Therefore, MWCNT dispersion is a difficult task for their use in nanoscale device applications. Applications for MWCNTs have been severely constrained by their poor dispersion in water and organic solvents. The MWCNTs were distributed in organic solvents and polymers and have further potential uses in nanoscience. Clean synthesis methods with lower energy input and less demanding conditions are necessary for the CNT dispersion process. Technology-friendly new processes are required; hydrothermal approaches could result in a repeatable way for fabricating carbon compounds. Hydrothermal processing, which is used to dissolve and recrystallize materials that are often intractable under normal circumstances, is a heterogeneous reaction that takes place in the presence of aqueous solvents or mineralizers at high pressures and temperatures. With faster reaction rates, smaller particle sizes, and stable reaction products, supercritical fluids (SCFs) constitute a preferable reaction media for hydrothermal processing of nanoparticles.

In the past, the current author used amorphous carbon and a hydrothermal reaction to create carbon nanocells and nanotubes. In the current study, an effort was made to disperse MWCNTs under various organic solvents using hydrothermal processes in the appropriate supercritical solvent conditions. SCFs transform mass from higher to lower concentrations and have increased diffusivity in solid materials. To determine the level of dispersion, the resultant materials were sonicated and examined by TEM, Raman spectroscopy, UV-vis spectroscopy, and Dynamic Light Scattering (DLS). Functionalization This is carried out with the use of:

(a) inorganic compounds, such as carbon allotropes (nanodiamonds, grapheme oxide), iodine doping, mineral acids and their mixtures, $CO_2$ and $SiO_2$, peroxides, inorganic salts;

(b) organic compounds: diazonium salts, organic acids and salts, sulfonates, amines, porphyrins, pyrene and other polyaromatic compounds, DNA, biomolecules, polymers, natural products, etc.

## RESEARCH METHODOLOGY

In this investigation, MWCNTs from Sisco Research Laboratory in India were used. They had an outer diameter of 10–20 nm and a length of 10–30 m, and a 95% assay. Fisher Scientific in India provided the solvents, such as dichloromethane, ethanol, isopropyl alcohol, and hexane. MWCNTs were used in the studies at a concentration of 80 mg/L, combined with various organic solvents. For dispersion, various organic solvents including dichloromethane, ethanol, isopropyl alcohol, and hexane were utilised.

The 20 mL of the organic solvent were combined with 0.2 g of MWCNTs. Each suspension was put into an ultrasonicator (Sidilu, 20 kHz, 300 kV) and sonicated for 15 minutes. 20mL of the sonicated solutions were then put in Teflon liners and autoclaved using Morey style devices. For five hours, the autoclaves were continuously heated in a hot air oven to the critical temperature for each solvent. The autoclaves were then taken out of the oven and allowed to cool to room temperature after five hours. The final products were carefully collected in a beaker together with the solvent in a liner. They were next centrifuged (KEMI, 159 Watts/HP, 230V, 50Hz) at 10,000 rpm for 15 minutes to test the stability after being sonicated for further dispersion since the bundled MWCNTs settle down during spinning and the supernatant includes finely dispersed MWCNTs (at lower concentration). These final supernatant mixtures with finely distributed MWCNTS were put to use for additional characterization.

**Table 1: List of critical temperature for the different solvents**

| Solvents | Critical temperature ($^{o}$C) |
|---|---|
| Dichloromethane | 237 |
| Ethanol | 243 |
| Isopropyl alcohol | 235 |
| Hexane | 235 |

## CHARACTERIZATION TECHNIQUES

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To better understand the dispersion of MWCNTs, the dispersions of MWCNTs in various organic solvents were studied using a UV-vis spectrophotometer (Shimadzu UV-1800, optical cell length 10 mm, cell volume 3 mL) that was operated in the 200–1200 nm range. Pure solvents were used for the baseline correction, and the absorbance values were subtracted from the absorbance values of the dispersed MWCNTs. Then, fresh samples of the associated solvents were used to subtract these baseline corrections each time. Later, TEM was used to investigate the MWCNTs dispersions (HitachiH-7500). On carbon-coated TEM grids (300 mesh, 3 mm), a drop of 15 L of the dispersed solution was dropped. The grids were then examined under a microscope. This study provided information on the size of each MWCNT.

Using DLS (Microtrac-nanotrac wave- w 3231 Instruments Ltd., with a fixed scattering angle of 90 at room temperature), the average particle size of the scattered MWCNTs was determined. Pure solvents were used to remove any background noise before measuring the zeta potential and particle sizes of the distributed MWCNTs. Raman spectroscopy is a practical, potent, and nondestructive method for characterising carbon and carbon-based materials including carbon nanotubes, carbon black, and other similar materials, and it has emerged as a key tool for comprehending many fundamental features of all sp2 carbon systems. Raman spectroscopy has been used to evaluate the degree of disorder in sp2-hybridized carbon systems, diameter, and the impact of tube-tube interactions on the vibrational modes.

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## RESULTS AND DISCUSSION

### UV-VISIBLE ABSORBANCE SPECTROSCOPY

The bundled CNTs are not active in the same wavelength area as the scattered and individual CNTs, which exhibit distinctive bands between 200 and 1200 nm. Thus, UV-vis spectroscopy can be used to describe CNTs that have been disseminated. The UV-vis spectra of dispersed MWCNTs produced by hydrothermal processes in supercritical circumstances using the appropriate organic solvents and regular (untreated) MWCNTs are shown in Figure 5.1. Different organic solvents were used to evaluate the absorbance of MWCNTs that had been disseminated in raw solutions (before centrifugation) and supernatant solutions (after centrifugation). First, the absorbance of MWCNTs in the raw solutions was measured, and then in the supernatant solutions. Because of the variations in solution concentration, raw solutions had higher absorbance values than supernatant solutions. The absorbance of MWCNTs solutions peaks between 200 and 300 nm and gradually declines from UV to near IR, which is partially caused by scattering, particularly in the lower wavelength range. By using UV-vis spectroscopy, the supernatant solution of dichloromethane exhibits maximal absorbance, indicating the existence of a significant amount of dispersed MWCNTs with stable product after centrifugation.

### PARTICLE SIZE DISTRIBUTION

Because of the Brownian motion of the particles, DLS is the most useful technique for characterising nanomaterials in solutions. Using DLS, the particle sizes of scattered MWCNTs were examined. The MWCNTs that have been disseminated in dichloromethane have disentangled from aggregation and have an average particle size (cumulative average) of 38 nm as opposed to the untreated MWCNTs, which have an average particle size of 112 nm (Figure1 (a). The ethanol solution contained particles with an average size of 143 nm. The particle sizes of the isopropyl alcohol and hexane solutions are 207.7 and 356 nm, respectively, showing slightly scattered nanotube particles. These results also agree with TEM examination, which shows both aggregation and dispersion.

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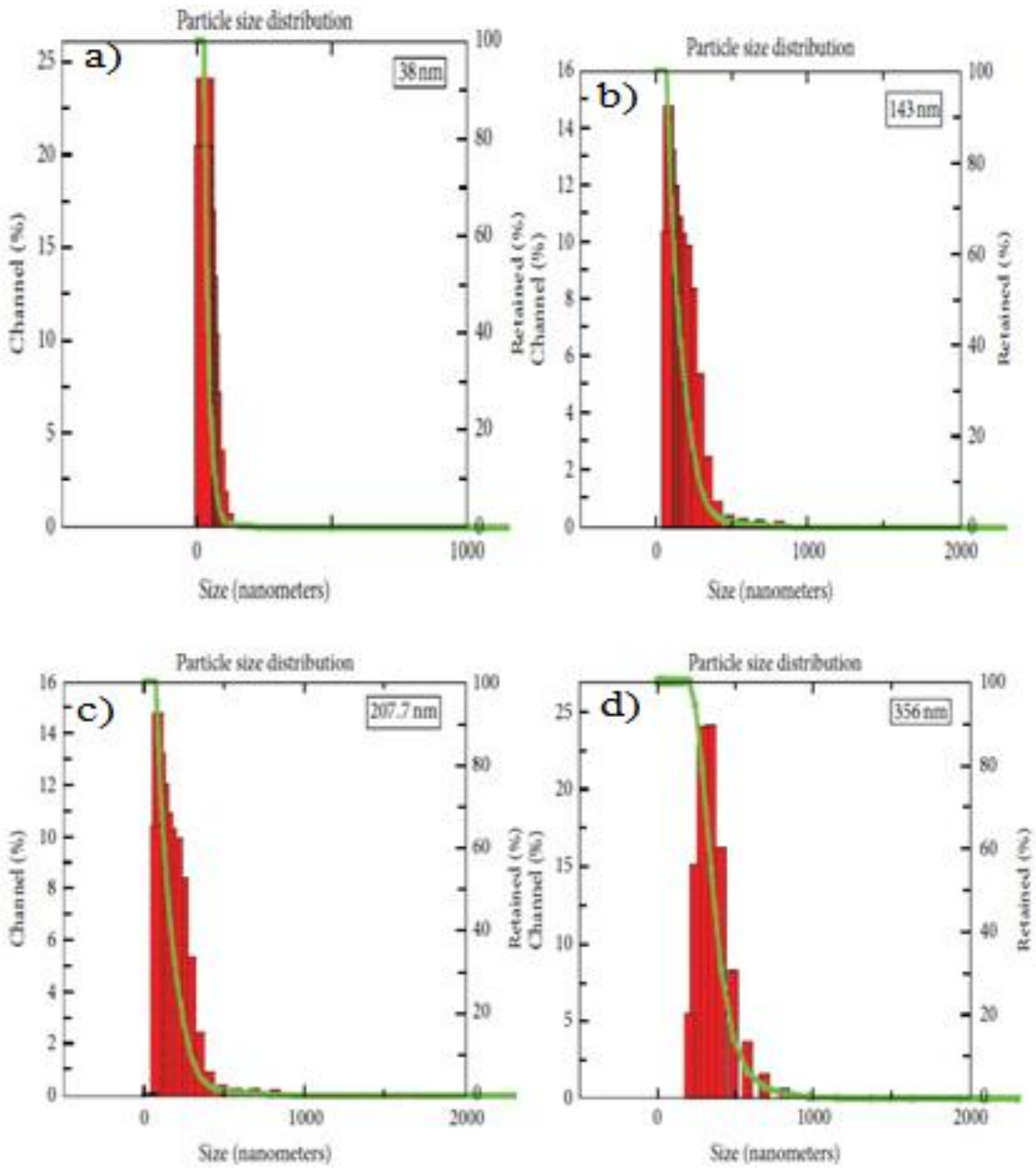


**Figure 1:** Particle size measurement of dispersed MWCNTs using DLS in (a) dichloromethane, (b) ethanol, (c) isopropyl alcohol, and (d) hexane.

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The stability of CNTs in solutions is explained by their zeta potential, which was measured using DLS and has an absolute value greater than 30mV. Zeta potential measurements for MWCNT solutions in dichloromethane, ethanol, isopropyl alcohol, and hexane are 31, 27.9, 24.8, and 13.5 mV, respectively. When compared to other solutions, the dichloromethane solution's absolute zeta potential value of 31 mV resembles stronger stability and shows that nanotubes were disseminated independently without aggregation.

**Table 2: Zeta potential value of dispersed MWCNTs**

| MWCNTs dispersed in solvents | Zeta potential |
|---|---|
| Dichloromethane | 31.0 |
| Ethanol | 27.9 |
| Isopropyl alcohol | 24.8 |
| Hexane | 13.5 |

## SURFACE ANALYSIS

Using TEM analysis, the dispersion conditions of MWCNTs were made visible. Figure 5.3 displays TEM images of untreated MWCNTs and MWCNTs that have been disseminated in ethanol, isopropyl alcohol, hexane, and other organic solvents. Before TEM studies in order to obtain high-quality pictures, the organic solutions containing dispersed MWCNTs (after hydrothermal reactions) were ultrasonically treated for 15 min. The MWCNTs dispersed in dichloromethane had a diameter of about 5–10 nm and smaller bundle sizes, according to the TEM image of Figure-2 (a). The highest dispersion of MWCNTs in dichloromethane was achieved during the hydrothermal reaction under supercritical conditions, as shown in Figure-2 (b). Numerous photos are needed in order to assess the dispersion and bundle sizes of MWCNTs because only a relatively tiny volume of the sample was examined during TEM examination. The diameter and dispersion of MWCNTs are revealed by the TEM research. Figure-2 (c) demonstrates that MWCNTs with outer diameters of around 6-10 and a reduced bundled size were detected after being hydrothermally dispersed in ethanol (Figures -2 (c) and 2 (d)). The MWCNTs were dispersed in isopropyl alcohol, and the high resolution TEM

images (Figures 2 (e) and 2 (f)) clearly showed that the tubes were slightly distributed and ranged in diameter from 20 to 50 nm. With outer diameters of around 50 nm, the maximum MWCNT aggregation was seen by TEM analysis in hexane solvent (Figures 2 (g) and 2 (h)).

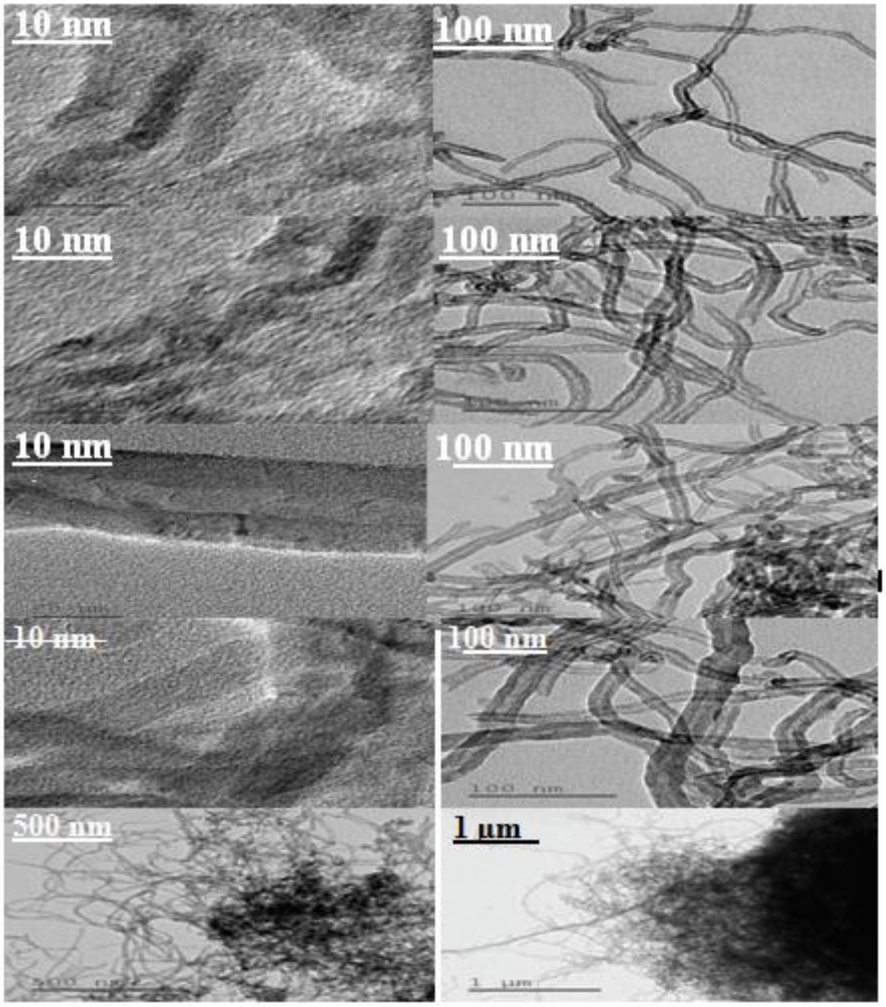


**Figure-2:** TEM images of in MWCNTs dispersed in the solvent (a, b) dichloromethane, (c, d) ethanol, (e, f) isopropyl alcohol, (g, h) hexane and (i, h) MWCNTs before dispersion.

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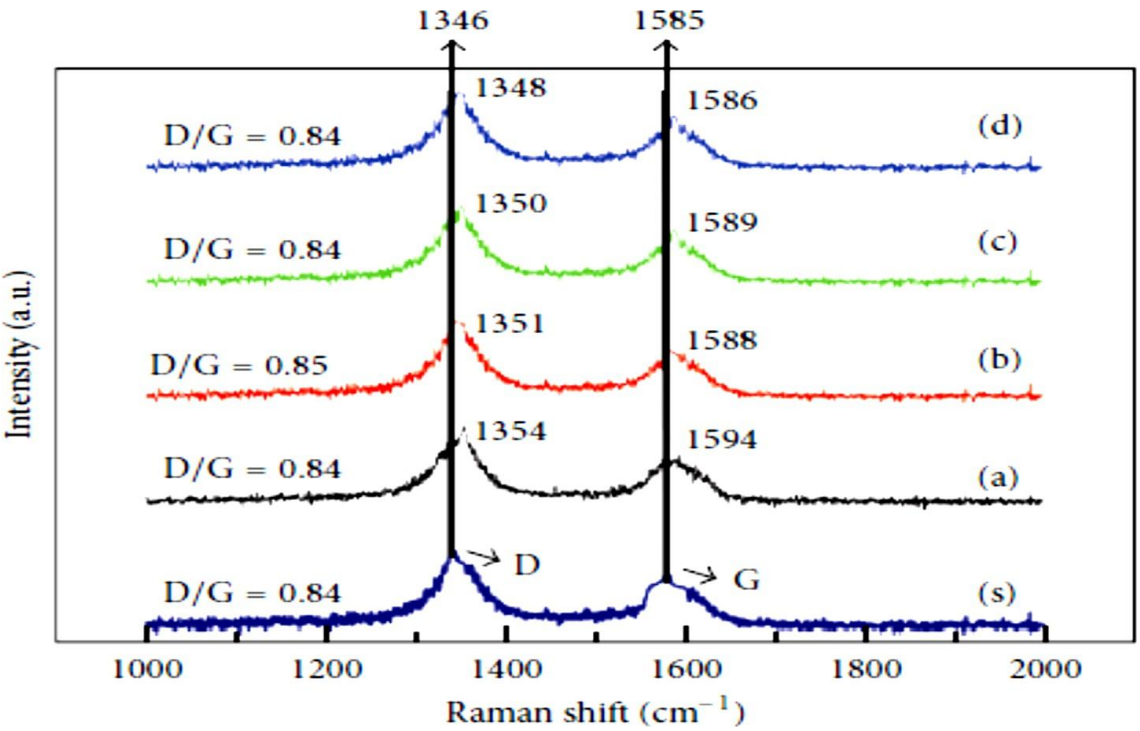
**RAMAN SPECTROSCOPY** The Raman spectroscopy of MWCNTs dispersed in various organic solvents is shown in Figure-3. To assess the separation of nanotube bundles, the Raman spectra of untreated MWCNTs are contrasted with those of MWCNTs dispersed in various organic solvents. Untreated MWCNTs' Raman spectroscopy (Figure-3(s)) has revealed two typical bands at 1585 and 1346 cm-1, which unmistakably denote the plane vibration of the C-C bond (G band) and disorder band, respectively. Figure 3 ((a), (b), (c), and (d)) shows that the D band and G band of treated MWCNTs have both shifted toward higher wave numbers, indicating debundling of the tubes. The ratio of the D to G band has not altered, which also suggests that the hydrothermal reaction's tube diameter has not changed. The debundling and dispersion of MWCNTs in dichloromethane, among all the solutions, are more pronounced with larger wave number shifting of the D and G bands from 1346 to 1354 cm1 and 1585 to 1594 cm1, respectively. Studies using Raman spectroscopy can also be




combined with TEM, UV-vis, and DLS characterisation.

**Figure-3: Raman spectra of MWCNTs dispersed in the solvent (a) dichloromethane, (b) ethanol, (c) isopropyl alcohol, and (d) hexane.**

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## VISUAL OBSERVATION AND STABILITY OF DISPERSED MWCNTS IN THE SOLVENT:

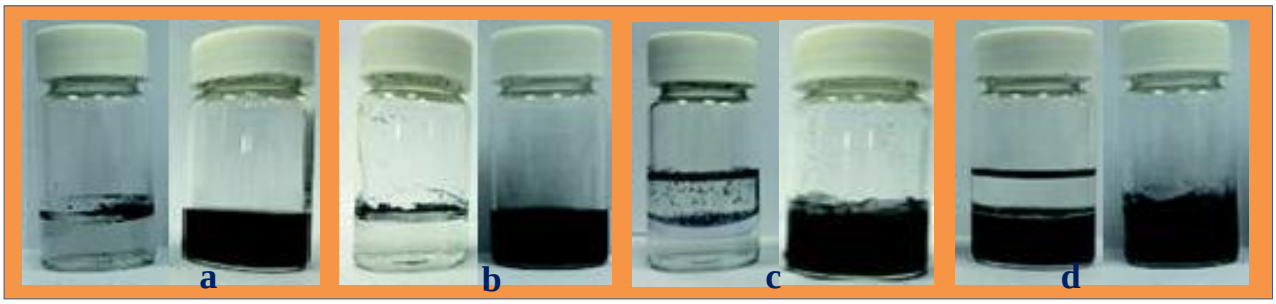


**Figure-4 :** Visual observation of MWCNTs before and after dispersion in solvent (a) dichloromethane, (b) ethanol, (c) isopropyl alcohol, and (d) hexane.

Under hydrothermal conditions, the MWCNTs were dispersed in a variety of organic solvents, and the stability of the dispersion was evaluated. After being set aside for five hours following hydrothermal treatment, the scattered MWCTs were compared to the MWCNTs both before and after dispersion. The MWCNTs were seen to be dispersed in organic solvents, however the stability varied depending on the solvent. It demonstrates that different combinations have varying stability times. Dichloromethane has greater stability during a five-hour period, i.e., it did not settle out following a hydrothermal reaction.

## MECHANISM INCLUDING SUPER CRITICAL FLUID EXAMPLE

Any substance at a temperature and pressure above its critical point, when separate liquid and gas phases do not exist, is referred to as a supercritical fluid (SCF). It has the ability to dissolve substances like a liquid and effuse through solids like a gas. Additionally, around the critical point, slight variations in temperature or pressure cause significant variations in density, enabling the "fine-tuning" of numerous supercritical fluid properties. In a variety of industrial and laboratory operations, supercritical fluids can replace organic solvents. The most popular supercritical fluids are carbon dioxide and water, which are utilised for power generation and decaffeination, respectively.

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The solubility of the material in the fluid is one of the most crucial characteristics. With fluid density, solubility in supercritical fluids tends to rise (at constant temperature). Solubility tends to rise with pressure because density tends to rise with pressure. The connection to temperature is a little trickier. Solubility will rise with temperature for a given density. If the mixture's critical point is exceeded, a single phase may be ensured because all supercritical fluids are entirely miscible with one another. Altering the reaction solvent's properties can enable single-phase reactions or phase separation for product removal. Diffusion-controlled reactions are sped up by rapid diffusion. An essential process that occurs when a solute's saturation point is exceeded by dilution, depressurization, or a combination of these processes is the creation of microscopic particles of a material with a narrow size distribution. In comparison to liquids, these reactions happen more quickly in supercritical fluids. Recent research on supercritical fluids has demonstrated their ability to shrink particles as small as 5-2000 nm.

**Table -3: Comparison of gases, supercritical fluids and liquids**

| **Phase** | **Density ($Kg/m^3$)** | **Viscosity ($\mu Pas^{-1}$)** | **Diffusivity ($mm^2/s$)** |
|---|---|---|---|
| Gases | 1 | 10 | 1-10 |
| Supercrtical Fluids | 100-1000 | 50-100 | 0.01-0.1 |
| Liquids | 1000 | 500-1000 | 0.001 |

## APPLICATION OF DISPERSED MWCNTS IN COMMERCIAL PURPOSE

The MWCNTs with uniform distribution are used for a variety of industrial applications. MWCNTs were mixed with cement using water to create cement/MWCNT composites in the cement manufacturing sectors, which can improve performance and have a high compressive strength. The electrical conductivity of the polystyrene/MWCNT nanocomposite is increased from 1.91x10-7 to 1.15x10-5 S/cm by the selective dispersion of MWCNTs.

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## CONCLUSION

In the scientific community, MWCNT dispersion in organic solvents is more useful and important. It is enjoyable to carefully disperse MWCNTs using hydrothermal technology. Under their supercritical condition, different solvents have displayed varying characteristics. In MWCNTs with mass transitions in supercritical conditions, the solvents liquefy and exhibit higher diffusivity. Diffusivity and mass change, however, cannot occur at room temperature or under normal circumstances. As a result, the use of MWCNT dispersion in supercritical hydrothermal solvent reaction yields positive results. The MWCNTS are present as single, unbundled MWCNTs, as shown by the UV-vis absorption in the 200–1200 nm range. The stability of dispersed solutions is indicated by the absorbance of raw MWCNTs and supernatant solutions of centrifuged MWCNTs.

The MWCNT particle size and average size of dispersed MWCNTs, as revealed by the DLS investigation, offer a clear explanation for the very small particle sizes that denote good dispersion with the backing of absolute zeta potential value. Although the morphological examination of MWCNTs by TEM is an important technique, the limited amount of sample used during analysis shows the need for several pictures per sample. A TEM investigation of MWCNTS distributed in various solutions in the current work reveals sizes ranging from 5 to 50 nm in diameter. All of the characterization methods mentioned above were related to the hydrothermally dispersed MWCNTs' Raman study; this analysis showed debundling by moving the G and D bands and also showed that the diameter of the MWCNTs had not altered.

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