



Study the Effect of Adding MWCNTs on the Hardness, Impact Strength, and Structural Properties of Composite Materials based on Epoxy Polymer



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Abstract

This research focuses on using Multiwalled carbon nanotubes to successfully produce MWCNTs/Epoxy nanocomposite with various MWNT weight loadings (0.25, 0.50, 1 and 2 % wt.). The samples were manufactured by utilizing an ultrasonic machine and a solution mixing method (MIT). The characterization, mechanical characteristics, and morphology of the resulting MWCNTs/Epoxy nanocomposites were investigated using FTIR, hardness, impact testing, and FE-SEM. According to the findings, different MWCNT loadings enhanced the mechanical performance of epoxy nanocomposites. Compared to pure epoxy, the impact result of MWCNTs/epoxy nanocomposite was recorded to increase as 33 %, 46 %, 75 %, and 108 %, respectively. Furthermore, increasing the amount of MWCNT nanofillers in nanocomposites samples improved their hardness to be as 0.4% ,2.20% ,2.89%. Finally, the FTIR and FE-SEM were also tested to find the structure of MWCNTs and dispersion quality.

Keywords: Epoxy, MWCNTs, mechanical properties, FTIR, Impact strength, hardness, SEM.

1. Introduction

As a matter of fact, the world is created from moving carbon atoms. Epoxy-based materials are increasingly being used in a number of industrial applications. One of the primary disadvantages of polymers in industrial applications is their poor surface properties. The purpose of incorporating various forms of filler into a polymer matrix was to improve the required physical and mechanical characteristics of polymer composites [1-3]. Many researchers utilized tensile, hardness, and impact tests to determine the different mechanical properties of polymers [4, 5].

Iijima's discovery of carbon nanotubes in 1991 garnered a lot of interest in improving their characteristics [6]. Other nanoparticles, such as carbon nanotubes, have the extraordinary powers of carbon atoms, making it even more unexpected, especially with the nano-size characteristic. Carbon nanotubes (CNTs), single graphene layers (SWNTs), and multiple graphene layers wrapped onto themselves (MWCNTs) are crystalline carbon structures that have only recently been discovered [7]. Multiwalled carbon nanotubes (MWCNTs) are generally described as hollow and lengthy concentric

cylinders ranging from 6 to 25 or more graphite sheets on a nanoscopic level [8]. Because of the existence of multiple layers of graphene, MWCNTs have a higher mechanical strength, making them useful in composite materials [9]. The addition of small quantities of MWCNTs to thermoplastic polymers, on the other hand, has resulted in considerable increases in mechanical properties. Epoxy resin is the most common form of thermo setting resin. The Epoxy resins were used in a wide range of applications because of their high tensile strength and modulus, as well as their low cure time. Shrinkage resistance, chemical and corrosion resistance, excellent adhesion, and dimensional stability are all features of this material [10]. What would happen if we combined these two materials, each of which has complimentary properties? The nanoscale filler's dispersion in the polymer matrix is crucial in defining the composite's characteristics [9, 10]. Fidelus et al. studied the nanocomposites of MWCNTs/epoxy with 0.5 wt.% addition. Their results showed a considerable increase in impact strength (70 percent reinforcement) [11]. Albozahid also looked into the impact of adding graphite filler

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to epoxy composites, and found that mechanical properties were enhanced [12]. The goal of this research is to investigate the effect of MWCNTs addition on the epoxy matrix's mechanical characteristics. The mechanical characteristics of the epoxy composites include impact, test, and the hardness test. Morphology characteristics of MWCNTs were further studied using Fourier transform infrared spectroscopy (FTIR) and the SEM to reveal dispersion of MWCNTs inside epoxy resin.

2. Experimental

2.1. Materials

Multi-walled carbon nanotubes (MWCNTs) industrial-grade from (Nanjing XFNANO Materials Tech Co. Ltd) with outer diameter (8-15nm), purity (>90 wt.%); inner diameter (3-6nm) (Tube length (30-50 μ m), apparent density (0.1 g/cm³); tap density (~2.1g/cm³), and conductivity (>100s/cm). The epoxy type used in this investigation was Sikodor-52, which was provided by Sika Turkey. Sikodor-52 is a two-component, low-viscosity liquid based on a high-strength epoxy resin that offers solvent-free hardening, shrinkage-free hardening, high mechanical and adhesive strength, and low viscosity.

2.2 Mold Preparation

Using a Computer Numerical Control (CNC) milling machine, an acrylic plastic sheet produces impact test molds, as shown in figure (1). An impact test mold with dimensions of (20 * 150) mm and a thickness of (5mm) is created in two layers. The ASTM (D256-04) impact specimen is the industry-standard ASTM [13].



Fig.1. Molds of impact test by using a Computer Numerical Control (CNC)

2.3 Preparation of MWCNT/epoxy nanocomposite casting samples

MWCNT weight fractions of 0.25, 0.50, and 1.2% were used in this investigation. The mixing process of epoxy with various weight percentages of nanofiller was conducted, followed by dispersing the combination in ethanol. The mixture was then filtered and washed with distilled water. The powder was dried in an oven at 60 degrees Celsius for 20 minutes. After washing the MWCNTs sample for four days, it must be allowed to dry in the air for a few hours. The mechanical stirrer is used to mix epoxy resin with various weight percentages of MWCNTs nanofiller

for 15 minutes. Optimum dispersion of MWCNTs with epoxy was continued with an ultrasonic homogenizer (MTI, 1200w) for 20 minutes. The hardener is then added, and the mixture is finally mechanically mixed for 10 minutes to achieve homogeneity before being poured into the molds at room temperature in a vacuum furnace (-80 kpa) until the bubbles are eliminated. It was then poured into the molds and left to cure for 48 hours.

3. Characterization Techniques

3.1. Hardness test

Hardness is a characteristic that expresses a material's resistance to deformation when subjected to a concentrated force on its surface. Tests established shore D hardness. TIME Group Inc, Beijing, China, provided the Digital Shore D Hardness Tester TIME@5431 for the test. According to the (ASTM D-2240) at Materials Engineering College, the University of Babylon was carried out by taking an average of five measurements for each specimen at different points on the surface of specimens. As shown in Figure 2, three samples were evaluated for each mass fraction of MWCNTs (0.25, 0.50, 1 and 2% wt).



Fig.2. Pure epoxy and their nanocomposite samples were tested for each mass fraction of MWCNTs

3.2. Impact strength

The test was carried out using a device (WP 400 Impact test, 25Nm) pendulum impact tester of gunt Hamburg CO / Germany at Materials Engineering College, the University of Babylon. According to the standard impact specimen ASTM D256. Three samples were testing, and the average was calculated for each composite. The test was performed at room temperature. Figure (3) showed impact specimens after the test. Impact strength (I.S) is calculated by applying the equation

$$I.S = \frac{Uc}{A} \text{ (KJ/m}^2\text{)}$$

Where

Uc: the fractured impact energy (K Joule), which is determined from the Charpy impact test device.

A: the cross-sectional area of the samples.



Fig.3. shows impact specimens after the test. Impact strength

Materials type	Improvement Percentage of Impact Strength (%)
Epoxy-MWCNTs-0.25%	33
Epoxy-MWCNTs-0.50%	45
Epoxy-MWCNTs-1%	75
Epoxy-MWCNTs-2%	108

3.3. FTIR test

To investigate the chemical composition for the powdered MWCNTs, spectra is acquired at room temperature of 25 C using an Alpha FTIR spectrometer by Shimadzu equipment at the College of Science, Department of Chemistry at UOK. The chemical structures of organic compounds and the behavior of functional groups throughout the polymerization process are revealed by FTIR. It works in the infrared region (IR), which ranges from 14,000 cm^{-1} to 10 cm^{-1} and correlates to changes in molecular vibration energy.

3.4. SEM test

The morphological features MWCNTs powder were observed using SEM (VEGA/TESCAN-XMU type at Razi metallurgical research center in the Islamic Republic of Iran). This nanopowder's cryogenically surface was covered with a thin coating of gold or carbon to make it conductive to incident electron beams, resulting in a better SEM image.

4. Results and Discussion

4.1 Impact Strength

Figure(4) illustrates the impact strength of MWCNTs as a function of filler loading. It shows that adding MWCNTs into epoxy has a substantial effect on the impact strength of MWCNT nanocomposites when MWCNTs are loaded. MWCNTs/epoxy composites demonstrated higher impact strength, with an increase of about (33.3%,45.8%,75%,107.5%), respectively compared to pure epoxy as shown in Table1. This is due to the MWCNTs/epoxy

nanocomposite's higher dispersion of carbon nanotube particles in the matrix, providing a significant toughening effect compared to pure epoxy. The debonding of the chain segments from the filler surface allows the matrix entanglement structure to relax when the load is communicated to the matrix-filler physical network, resulting in improved impact toughness[12]. The filler content has an impact on low impact energy—the composites' ability to absorb and decrease energy during fracture propagation. In the case of a thermoset toughened polymer, however, the presence of the thermoset essentially induces stress redistribution in the composite, resulting in micro-cracking or crazing at several locations, resulting in a more effective energy dissipation process. Other studies that are similar to the present findings are in agreement with it [12, 14].

Table 1:Improvement Percentage of Impact Strength of MWCNTs/epoxy composites.

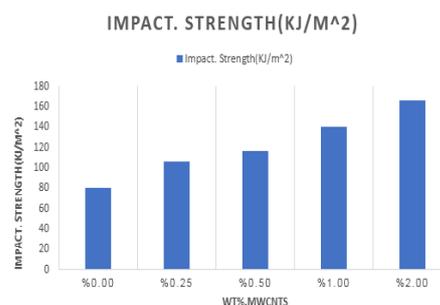


Fig.4.Impact strength of MWCNTs filled Epoxy composites.

4.2. Hardness test

Figure (5) displays the hardness of different epoxy nanocomposites (shore – D). When the concentration of microparticles was increased, the findings revealed a modest improvement in hardness, as shown in Table 2. MWCNTs/epoxy composites, on the other hand, have a greater hardness than net epoxy. The best percentage increases at a weight fraction of 1% and 0.5%, respectively, for MWCNTs nanocomposites. Due to nanoparticle dispersion in epoxy, there is a minor increase in hardness since few nanoparticles are on the top and lower surfaces of the samples [15]. At 2wt.% hardness values, the development of CNT agglomerates causes the mechanical strength to be reduced, effectively resulting in a porous material. Furthermore, agglomerates function as slip planes and planes where cracks may easily spread, resulting in sample breakage even at modest mechanical stresses[16].

Table2: Improvement Percentage of Hardness of MWCNTs/epoxy composites.

Materials type	Improvement Percentage of Hardness (%)
Epoxy-MWCNTs-0.25%	0.41
Epoxy-MWCNTs- 0.50%	2.20
Epoxy-MWCNTs- 1%	2.89
Epoxy-MWCNTs- 2%	3.12

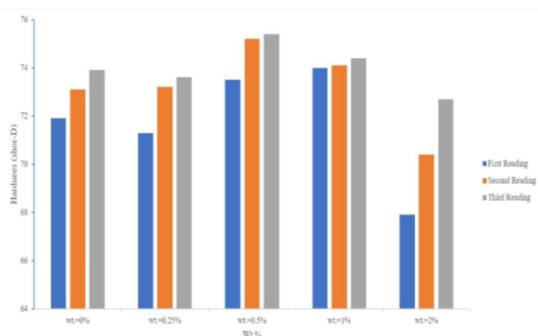


Fig.5: Hardness MWCNTs filled epoxy composites.

4.3. Fourier-Transform Infrared Spectroscopy Infrared (FTIR)

The study of interactions between infrared light and matter is known as infrared spectroscopy. Raman spectroscopy is frequently used to describe structural abnormalities in carbon nanotubes because it is very sensitive to them[17]. The absorption of various infrared light wavelengths by the material is measured using this approach. The infrared absorption bands of the MWCNTs identify distinct molecular components and structures. The surface functional group of carbon nanotubes Fourier transform infrared spectroscopy was used to confirm the formation of chemical functional groups on MWCNTs. (FTIR). The presence of the characteristic band (the O–H stretch of terminal carboxyl groups) at (3735-2670) is O-H. The peaks located at (2112-1503) is C=C, indicating that carboxyl group caused the adhesion of polar functional groups such as (OH) on the surface of MWNT. The FTIR spectrum of the chitosan is in the range (4000-500) cm^{-1} .

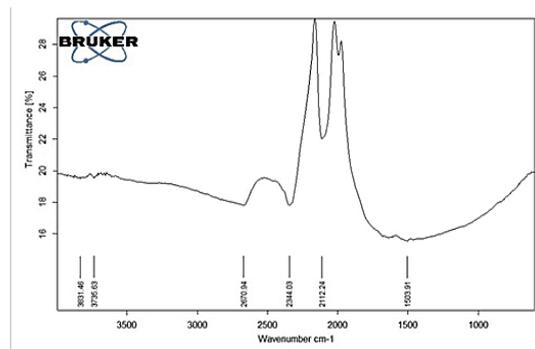


Fig.6. FTIR spectrum of MWCNTs

4.4. FESEM test

FE- SEM was used to analyze the fracture surface of the specimens as shown in Figure7. Micrographs of MWCNTs/Epoxy composites are shown to evaluate the failure mechanism of the composites.

The MWCNTs were completely coated in epoxy resin, as evidenced by micrographs. The fibers were separated from the matrix because of the close connection between the components. The pull-out event was only seen in a few nanofiller in Figure 7A–D. Interface debonding, matrix cracking, or inter-laminar delaminations were not seen. The purpose of this study was to look at the epoxy's interfacial interaction with the nanofillers.

At a resolution of 10 μm , a better resolution picture of MWCNTs/Epoxy composite was created, revealing intriguing details regarding the matrix/nanofillers bonding. The interface that enclosed the filler in the matrix had a high adhesion, resulting in a strong bonding. Smaller arches suggest that the nanofiller-matrix interfacial connection was strong, allowing for efficient stress transfer from the matrix. As a result, the mechanical properties of the composites are improved[18].

The FE-SEM images refer to another characterization procedure used to determine uniform dispersion. The CNT dispersion within the epoxy was visually determined using a scanning electron microscope (FE-SEM). The agglomerated nanotubes (dark strips in Figure 7D))can be visible on the cracked surface of the MWCNTs/Epoxy (2 wt%) composite for dispersion of MWCNTs/Epoxy composite (Figure 7D). The broken ends of MWCNTs are pulled away from the matrix, showing low distribution and interfacial adhesion. The MWCNTs/Epoxy composite, on the other hand, has well-distributed nanotubes throughout the epoxy matrix (Figure7A, 7B, and 7C). This means that MWCNT has high interfacial adhesion in addition to good dispersion.

To prove that MWCNTs are dispersed [19].which is more at higher MWCNTs content (2 wt%). The presence of voids, that are observed in Figure 7D, might be due to improper the mixture of epoxy and CNTs. The accumulation of MWCNTs occurs due to their high surface area, π - π bonding, and van der

Waals forces of attraction [20]. Individual CNTs sticking out of FE-SEM micrographs indicate poor adhesion between polymer and CNTs. FE-SEM data reveal a decent dispersion of MWCNTs in the matrix, but individual CNTs sticking out of FE-SEM micrographs indicate a weak binding between polymer and CNTs.

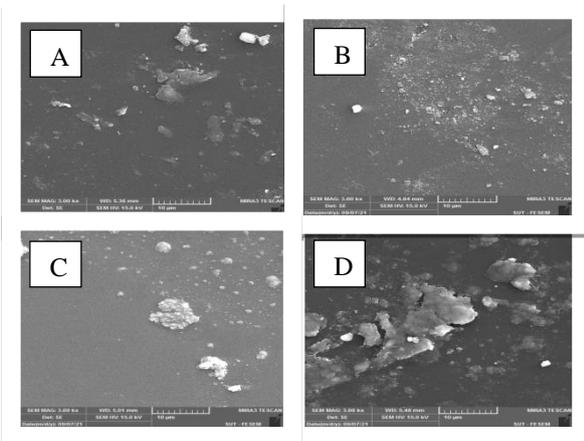


Fig.7. FESEM images of (A) MWCNTs/Epoxy(0.25% wt.), (B) MWCNTs/Epoxy(0.50% wt.), (C) MWCNTs/Epoxy (1% wt.), (D) MWCNTs/Epoxy (2% wt).

5. Conclusion

In this study, various weight fractions of MWCNTs were used to reinforce the epoxy matrix. The solution mixing process was successfully used to accomplish better dispersion using a probe tip. Consequently, the conclusions can be drawn as follows; The impact and hardness findings showed higher values for the composite samples compared with pure epoxy. The chemical groups of the MWCNTs can be identified using the FTIR test. The SEM image showed the tubular nature of the MWCNTs and their clusters. A suitable dispersion was seen at low concentration of MWCNTs. The aggregated MWCNTs was clearly observed at the percentage up to 1% wt..

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