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ENHANCED THERMAL, MECHANICAL, AND ELECTRICAL PROPERTIES OF EPOXY COMPOSITES REINFORCED WITH CDS/MWCNT NANOHYBRIDS SYNTHESIZED VIA HYDROTHERMAL METHOD

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Abstract: In this research, the effects of incorporating a hybrid nanocomposite consisting of cadmium sulfide (CdS) nanoparticles and multi-walled carbon nanotubes (MWCNTs) synthesized by hydrothermal method into an epoxy resin system on the bulk density, Shore D hardness, thermal conductivity coefficient, thermal stability, and dielectric properties have been investigated. In addition, the structural and physical properties of these nanocomposites aimed to determine their potential applications as lightweight and thermal insulation materials. The study included synthesizing CdS/MWCNT nanohybrid structures using $CdCl_2{\cdot}H_2O$ and $Na_2S_2O_3{\cdot}5H_2O$ precursors under specific time and concentration parameters. Then, these nanohybrids were integrated into the epoxy matrix to form innovative composite materials. The prepared composite samples were characterized using various methods to evaluate their mechanical, thermal, structural, and electrical properties. Techniques such as tensile tests, Shore D hardness measurements, microscopy, Fourier transform infrared spectroscopy (FT-IR), thermal conductivity, and dielectric measurements were used. The findings revealed that incorporating certain amounts of CdS/MWCNT nanohybrids significantly affected the density, hardness, thermal conductivity, mechanical strength, and dielectric properties of epoxy composites. In particular, the high surface area and effective distribution of CdS/MWCNT nanohybrid increased the mechanical strength and improved the thermal and electrical conductivities. The bulk density measured as 1133.5 kg/m^3 in the first experimental group reached 1145.1 kg/m^3, showing a steady increase until the 5th group. Shore D hardness measurements, which were initially measured as 77.6, increased to 79.8 in the last experimental group with the addition of nanohybrid structures. The thermal conductivity measured as 0.112 W/m·K in the first experimental group reached 0.136 W/m·K in the last group. Dielectric measurements showed that the dielectric coefficient increased from 3.86 in the initial sample to 5.67 in the nanoparticle-reinforced epoxy composites, indicating that the additive significantly improved the electrical properties, leading to a higher dielectric constant and enhanced energy storage potential. Microscopy images confirmed the homogeneous distribution of the nanohybrid within the epoxy matrix and strong interfacial interactions. FT-IR analysis confirmed the chemical bonds present in the hybrid composite structure. These results highlight the significant potential of incorporating CdS/MWCNT nanohybrid structures into epoxy composites to develop functional materials with advanced technological applications. This comprehensive study provides valuable insights into nanocomposite technology and highlights the promising role of CdS and MWCNT-based hybrid systems in future material designs.

Keywords: Cadmium sulfide, MWCNT, Hydrothermal method, Thermal Conductivity, Dielectric constant, Hybrid nanocomposite

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1. Introduction

Since the desired qualities of nanoparticles vary depending on the field in which they are generated, many manufacturing techniques have been developed. Several factors, including the synthesis techniques used, the precursor concentration, the reaction environment, temperature, pressure, reaction time, and stabilizing agents, affect the degree of production at the nanoscale. Variations in a product's nanosize often result in significant changes to the composition's properties. Nanoparticles, both single and aggregate, find use in solar systems (Bastianini et al., 2024), thermoelectricity (Hussain et al., 2024), catalysis (Shaban et al., 2024), and health diagnostics (Ndlovu et al., 2024; Stiufiuc et al., 2024; Manoharan et al., 2024; Abid et al., 2024). It is used in many different industries, including the pharmaceutical (Bharmal et al., 2024), solar cell manufacturing (Holmes et al., 2024), and other industries (Solomon et al., 2024; Majeed et al., 2024; Jan et al., 2024).

The variations in their electrical, optical, magnetic, and catalytic properties can be attributed to the fact that many nanoparticles differ from their macroscopic counterparts with the same composition in terms of size, shape, surface chemistries, and quantum effects (Ozcan et al., 2024). To produce nanoparticles with precise morphologies, sizes, forms, and surface chemistries, control over the synthesis process is required (Eikey, 2024). This is a difficult, demanding, and drawn-out procedure. Because it usually calls for many chemicals and dependent experimental parameters including reagent concentrations, reaction periods, temperatures, and mixing efficiency, the precise synthesis of nanoparticles is difficult. Completing the synthesis successfully also depends on the order in which the chemicals are added to the reaction mixture. To ascertain the exact amount that each experimental variable contributes to the outcome, experience, intuition, and trial and error are usually combined. This makes it difficult and time-consuming to determine the ideal recipe and reaction conditions. As a result, more precise and effective techniques are needed for the synthesis of nanoparticles with specific characteristics (Nanda et al., 2024).

Epoxy resins are exceptionally durable, versatile, and customizable synthetic polymer materials. Because of their chemical resistance, adhesive strength, and mechanical strength, these materials are widely used in a variety of industries (Singla et al., 2010; Wang et al., 2011; Hsissou et al., 2019). Epoxy resin and hardener are the two main ingredients of epoxy resins. When these two ingredients are combined, a chemical reaction takes place that causes the resin to cross-link and solidify (Unnikrishnan et al., 2008). Epoxy resins are very resistant to impacts, abrasion, acids, bases, oils, and solvents after curing (Anwar et al., 2024). They are therefore usually chosen in the aircraft sector (Xavier, 2024), floor coatings (Ruf et al., 2024), tank coatings (Dong et al., 2024), pipelines (Samardžija et al., 2024), and industrial coatings (Yu et al., 2022). Nevertheless, because of their waterproof and moisture-resistant qualities, epoxy resins are widely used in marine (Şomoghi et al., 2024) and other areas that come into contact with water, as well as in strengthening structures (Brandtner-Hafner, 2024), improving the properties of concrete (Ahn et al., 2024), installing floor coverings (Al-kawaz et al., 2024), and bonding different materials like metal, wood, and glass (CG et al., 2024). Because of their electrical insulating qualities, they are also employed to cover circuit boards and safeguard electrical components (Tian et al., 2024; He et al., 2024).

Because of their energy band structures, metal sulfides including zinc sulfide (ZnS), copper sulfide (CuS), lead sulfide (PbS), and cadmium sulfide (CdS) offer a compromise between electrical insulation and conductivity. They become a technical system that can be employed in sensors (Karikalan et al., 2017), photodetectors (Duan et al., 2024), and electronic gadgets (Karikalan et al., 2017; Fang et al., 2020). Because of its narrow band gap, lead sulfide (PbS) is used in infrared sensors and photodetectors (Halge et al., 2020), and zinc sulfide (ZnS) is used in optoelectronic applications (Vijai Anand, 2021), because it can be made conductive under certain conditions despite showing insulating properties, and copper sulfide (CuS) is used as materials that absorb electromagnetic waves (Han et al., 2024). The primary factor influencing the electrical characteristics of metal sulfide nanoparticles is their band gap, or quantum size effect, which varies with particle size. For instance, reducing the particle size in semiconductors like zinc sulfide (ZnS) and cadmium sulfide (CdS) increases the band gap, altering the material's electrical characteristics (Heiba et al., 2020). The characteristics of light transmission and absorption are likewise impacted by this attribute. Smaller nanoparticles may differ in their electrical conductivity characteristics due to their greater band gap.

Thanks to its special optical, electrical, and chemical characteristics, nanocadmium sulfide is a semiconductor material with a wide range of uses (Heiba et al., 2024; Azab et al., 2024). With a straight band gap of 2.42 eV at 300 K, CdS has a variety of beneficial chemical and physical characteristics. This results in appropriate and potential uses of CdS in several devices related to contemporary needs, such as solar cells, photochemical devices, sensor devices, detector devices, optoelectronic devices, luminescence devices, and many more (Erra et al., 2007). Numerous methods for creating CdS nanoparticles have been published during the last few decades (Chatterjee et al., 2001). The potential to develop novel experimental techniques facilitates the production of nanoparticles with a restricted size and shape distribution at an extremely cheap cost. In recent years, scientists have engaged themselves in the creation of high-quality CdS nano-particles and the investigation of their many qualities (Dabhane et al., 2021). Semiconducting metal sulfides such as cadmium sulfide (CuS) increase the electrical conductivity of epoxy composites, enabling their use in electronic applications (Rahimi-Ahar et al., 2024). Such composites can be used in applications such as electronic circuits (Panda et al., 2021), antistatic materials (Yudaev et al., 2023), and electromagnetic shielding (Bheema et al., 2024).

Researchers investigated the feasibility of incorporating MWCNT/CdS nanohybrid structures as additives into epoxy composites. In the literature, it has not yet been fully addressed how various variables during the synthesis of metal sulfides do not change the overall properties of the composite. In this case, further research is required to create CdS/MWCNT doped composite components fabricated under different conditions. The main objective of this study was to better study the strength, heat resistance, and dielectric properties of doped nanocomposite materials and also the potential of CdS/MWCNTs as reinforcements for composite properties under various manufacturing process conditions. This was achieved by looking at potential applications, evaluating their strength, and investigating processing methods for these methods. The study also seeks to pinpoint the difficulties faced and offer perspectives on the subject of materials science attained through environmentally friendly synthesis.

2. Materials and Methods

All chemicals used for the synthesis and characterization of the nanocomposite were supplied and used directly from Merck ($CdCl_2{\cdot}H_2O$), Zag Chemical ($Na_2S_2O_3{\cdot}5H_2O$), Nanography (MWCNT), and Polisan (epoxy resin). The epoxy resin used here is a solvent-free, two-component reaction-curing epoxy resin. Nanoparticle synthesis components are used without any additional processing, such as drying. Epoxy resins are directly cured at room temperature without the use of any additional materials.

2.1. Production of CdS Nanoparticles

In this study, cadmium sulfide nanoparticles were synthesized using an environmentally friendly hydrothermal process, a green synthesis technique. $CdCl_2{\cdot}H_2O$ and $Na_2S_2O_3{\cdot}5H_2O$ were used as precursor chemicals. The specified amounts of $CdCl_2{\cdot}H_2O$ were taken into a beaker and dissolved in 50 mL of pure water. $Na_2S_2O_3{\cdot}5H_2O$ was dissolved in 30 mL of pure water in another beaker. $Na_2S_2O_3{\cdot}5H_2O$ precursor solution was added dropwise to $CdCl_2{\cdot}H_2O$ precursor solution in an ultrasonic bath environment at room temperature for 20 min. At the end of the period, the mixture obtained was transferred to a 100 mL hydrothermal reaction vessel made of Teflon to complete the nanoparticle formation process. At the end of the period, the obtained nanoparticles were centrifuged at 4000 rpm to remove the liquid part. The obtained nanoparticles were washed with distilled water three times, then the precipitate was collected and dried in a 105 °C oven. Finally, they were manually crushed in a mortar and dried again to obtain cadmium sulfide nanoparticles (CuS-NPs) in the form of powder samples. Table 1 shows the concentration and time programming for nanoparticle synthesis.

Table 1. Concentration and time scheduling scheme for CdS-NPs

Sample No	Conc. of $CdCl_2$ (mM)	Conc. of $Na_2S_2O_3$ (mM)	Time (h)
1st	57.5	115	21
2nd	137.5	275	15
3rd	217.5	435	9
4th	250.0	500	15

2.2. Production of CdS/MWCNT Hybrid Nanocomposite

For each hybrid nanocomposites, the same amount of CdS-NP and MWCNT were 0.25 wt.% for the composite 1, 0.50 wt.% for the composite 2, 0.75 wt.% for the composite 3, and 1 wt.% for the composite 4 of epoxy resin. The indicated amounts were taken into a beaker and 10 mL of ethanol was added. This mixture was sonicated in an ultrasonic bath for 30 min. At the end of the period, the obtained hybrid nanocomposites were centrifuged at 4000 rpm to remove the liquid part. The obtained hybrid nanocomposites were dried in an oven at 60 °C. Finally, the CdS/MWCNT nanocomposites were manually pounded in a mortar and dried again to obtain the powder sample form.

2.3. Production of CdS/MWCNT/epoxy Resin Nanocomposite

This production was carried out in two stages. In the first stage, the epoxy primary component (A) and the CdS/MWCNT hybrid nanocomposite component were combined in a beaker. It was mixed with a mechanical mixer at 500 rpm for 30 min to ensure homogenization. This pretreatment was performed to homogeneously distribute the nanomaterial before the reaction of the epoxy components began. In the second stage, the hardener component (B) was added to the medium and mixed at the same speed for another 15 min. When the time was up, the composite paste was immediately transferred to the silicone molds. It was left to cure at room conditions for one day. Commercial epoxy resin consists of approximately 5/8 primary component and 3/8 hardener component by mass.

The numbering of the produced epoxy composites was made by considering the studies in Table 1. Experiment 1 (pure epoxy resin-based polymer), Experiment 2 (1st sample reinforced composite), Experiment 3(2nd sample reinforced composite), Experiment 4 (3rd sample reinforced composite), and Experiment 5(4th sample reinforced composite) are expressed.

3. Results and Discussion

In Figure 1, the bulk density values (kg/m^3) of the composites belonging to different experimental groups are given and the results show that the bulk density increases regularly with the increase in the amount of CdS/MWCNT hybrid nanostructure added to the composite structure. The bulk density measured as 1133.5 kg/m^3 in the first experiment (1st group) showed a steady increase until the 5th group reached 1145.1 kg/m^3. This increase reveals that the hybrid nanostructures are homogeneously distributed in the epoxy matrix and the nanocomposite gains a more compact and dense structure. The high density of the nanohybrids and the increase in the bonding surfaces in the epoxy resin matrix play a key role in the increase in density. The significant increase seen especially in the 4th and 5th groups indicates that the added nanostructure amount provides an effective interface interaction with the matrix, fills the voids, and improves the microstructural integrity. These findings show that optimizing the amount of nanostructures can also make positive contributions to other properties of composites such as mechanical strength and thermal conductivity (Aydoğmuş et al., 2022).

Figure 2 shows the Shore D hardness values of epoxy-based composites belonging to different experimental groups. It is seen from the graph that the hardness values increase regularly with the increase in the amount of CdS/MWCNT nanostructure in the hybrid nanocomposite. The Shore D hardness value, which was initially measured as 77.6, increased to 79.8 in the 5th experimental group. This increase can be associated with the homogeneous distribution of nanohybrids in the epoxy matrix and the increase in the mechanical strength of the composite by providing a strong interface interaction with the matrix. Nanohybrids increased the compactness of the material and improved the load-carrying capacity by filling the micro-voids in the composite structure. These findings show that optimizing the amount of nanostructures provides positive contributions to the mechanical performance of the composite (Dağ et al., 2023).

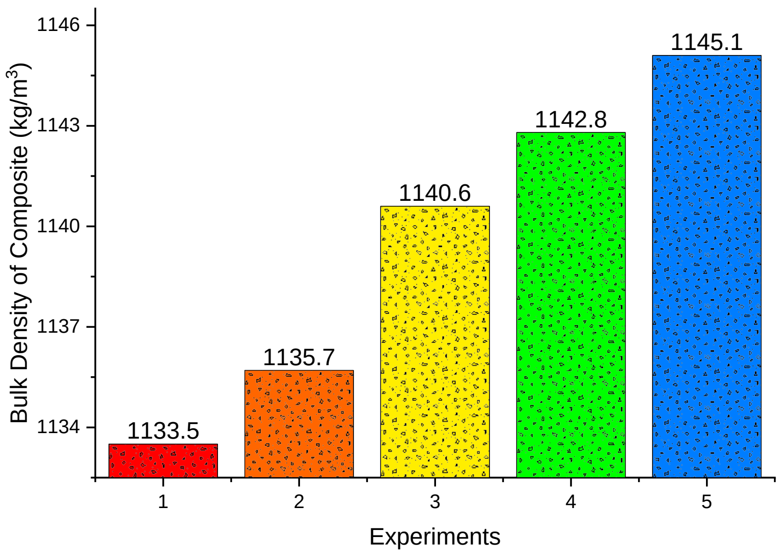

Figure 1. Bulk densities of the obtained epoxy-based composites.

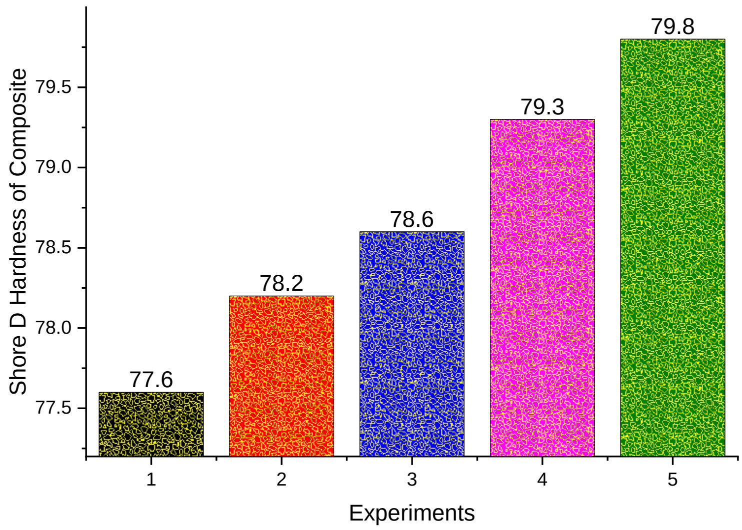

Figure 2. Shore D hardness of the produced composites.

Figure 3 shows the thermal conductivity coefficients of the composites in different experimental groups. It is observed from the graph that the thermal conductivity values increase significantly with the increase in the amount of CdS/MWCNT nanohybrid. The thermal conductivity measured as 0.112 W/m·K in the first experimental group reached 0.136 W/m·K in the 5th group. This increase is related to the high thermal conductivity capacity of the nanohybrids and their homogeneous distribution in the epoxy matrix. The nanohybrids increased the heat conduction in the matrix by acting as a bridge during heat transfer. In addition, the decrease in the thermal barriers in the matrix with the increase in the interfacial area of the nanostructures strengthened the thermal performance of the composite. These results show that hybrid nanocomposites are a potential candidate in applications requiring high thermal conductivity (Buran et al., 2023).

Figure 4 shows the dielectric coefficients of the epoxy composites without additives and nanoparticle-reinforced. It is understood that the dielectric coefficient of the epoxy matrix-based composite increases with the addition of nanoparticles. As seen in the figure, the dielectric coefficient of the pure epoxy-based polymer without additives is 3.86, while it increases to 5.67 in the epoxy composites with nanoparticle reinforcement. Accordingly, an increase in electrical conductivity is observed depending on the amount of additives, which can increase dielectric losses. The change in the dielectric coefficient exhibits a complex relationship depending on the amount of additives, frequency range, and the internal structure of the composite. The dielectric coefficient of pure epoxy-based polymer without additives is 3.86 (Torğut et al., 2024).

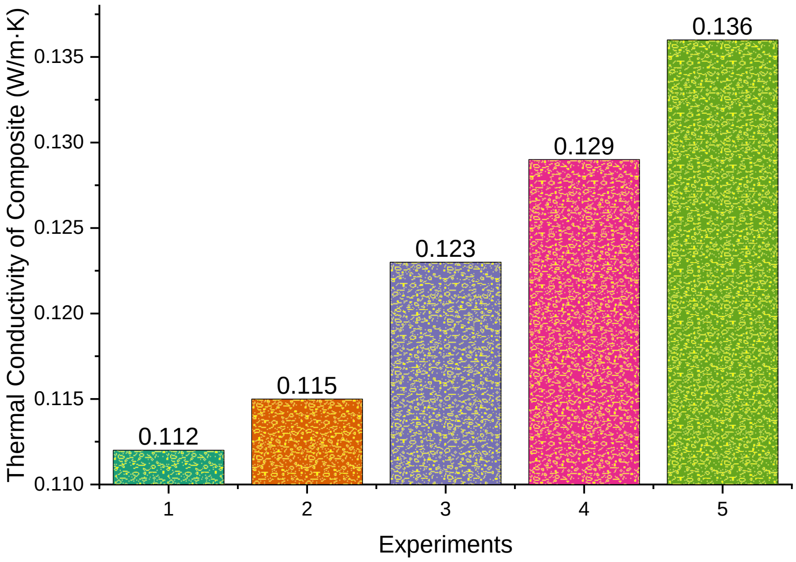

Figure 3. Thermal conductivity coefficients of epoxy-based composites.

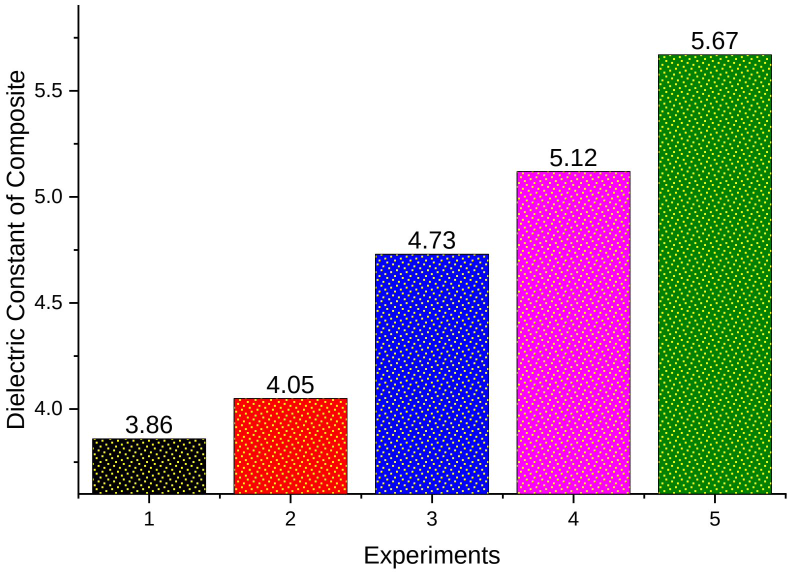

Figure 4. Dielectric coefficients of the samples produced in the experiments.

Figure 5 and Figure 6 show the tensile stress and strain (%) values as a result of the tensile test. As the tensile stress values of the samples increase, the strain rates decrease. When the experimental studies are evaluated, the elastic behavior decreases as the mechanical strength of the composites increases.

FT-IR spectra provide important information to evaluate the chemical bond structures and interactions between the components of epoxy resin-based CdS/MWCNT hybrid nanocomposite. In the pure epoxy resin spectrum, a broad peak around 3400 cm^{-1} generally indicates hydroxyl groups (O–H), while peaks belonging to C–H stretching vibrations are observed in the range of 2920-2850 cm^{-1}. In addition, the characteristic peaks of epoxy are C–O–C asymmetric and symmetric stretching vibrations in the range of 1250-1050 cm^{-1} and vibrations confirming the presence of an epoxy ring at a wavelength of approximately 910 cm^{-1}. The preservation of the characteristic peaks of the epoxy matrix in FT-IR spectra indicates that the nanohybrid does not cause structural changes by reacting with epoxy but instead forms interfacial interactions such as physical or hydrogen bonds. However, the decrease or disappearance of the epoxy ring peak around 910 cm^{-1} indicates that the epoxy cross-links with the hardener. The decrease or disappearance of the O–H broad peak around 3400 cm^{-1} with the inclusion of CdS/MWCNT may indicate that the free hydroxyl groups on the surface interact with the epoxy matrix and contribute to the formation of a hydrophobic structure. This indicates that the nanohybrid provides a homogeneous distribution and the binding areas within the matrix increase. In addition, characteristic vibrations belonging to metal-sulfur (Cd–S) bonds can be observed in the FT-IR spectra of the CdS/MWCNT nanohybrid (400-600 cm^{-1}). These peaks confirm that the nanohybrid is successfully dispersed in the epoxy matrix and integrated into the material structure. In addition, the increases in the intensity of the C–O–C and C–H peaks with the addition of the nanohybrid support that the nanohybrids reinforce the epoxy matrix. Overall, FT-IR results reveal that the hybrid structure is homogeneously distributed within the epoxy matrix and the observed improvements in physical properties are due to effective interfacial interactions through chemical bonds (Lu et al., 2024; Qin et al., 2024).

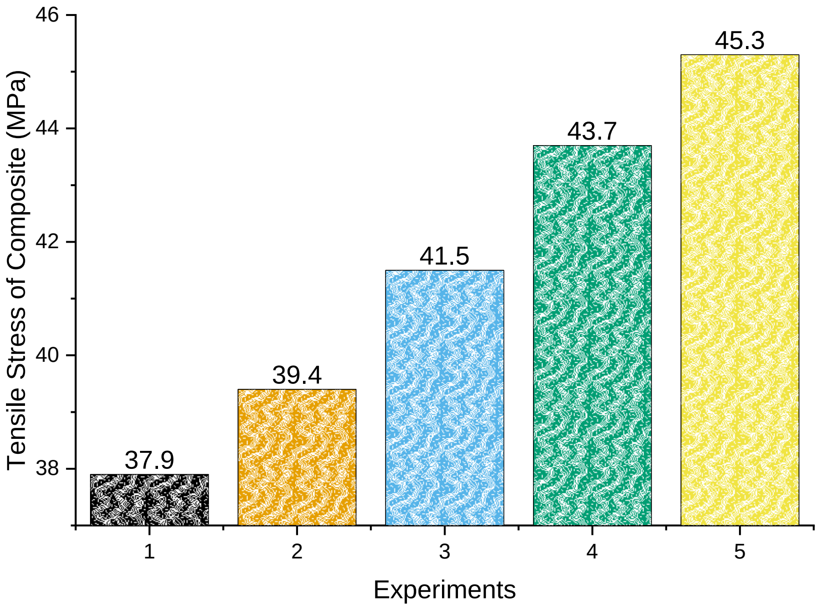

Figure 5. Tensile strength values of the obtained composite samples.

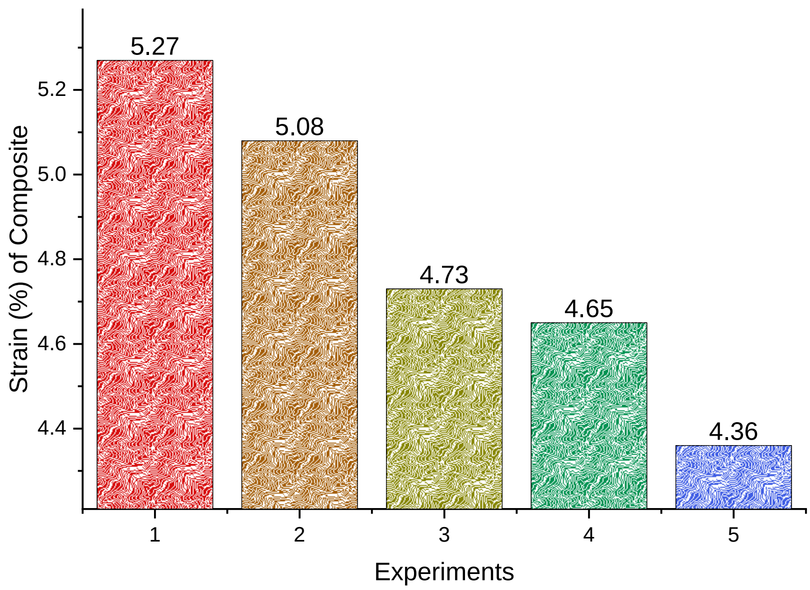

Figure 6. Tensile strain (%) values of samples obtained in experimental studies.

Figure 7 shows the FTIR spectra of the CdS/MWCNT/Epoxy nanocomposite containing CdS-NPs produced by hydrothermal technique immobilized in epoxy resin. In FTIR spectra, it is expressed as pure epoxy resin (Experiment 1), 1st composite (Experiment 2), 2nd composite (Experiment 3), 3rd composite (Experiment 4), and 4th composite (Experiment 5). According to the FTIR spectrum results, there is no chemical bond between the nanocomposite additive and the epoxy resin, but there is a physical interaction. The absence of any shift in the characteristic spectrum peaks supports the idea of physical interaction.

In the microscope images shown in Figure 8, the light-colored sample (left side) is the pure epoxy-based composite. The darker image (right side) is the product of Experimental 3.

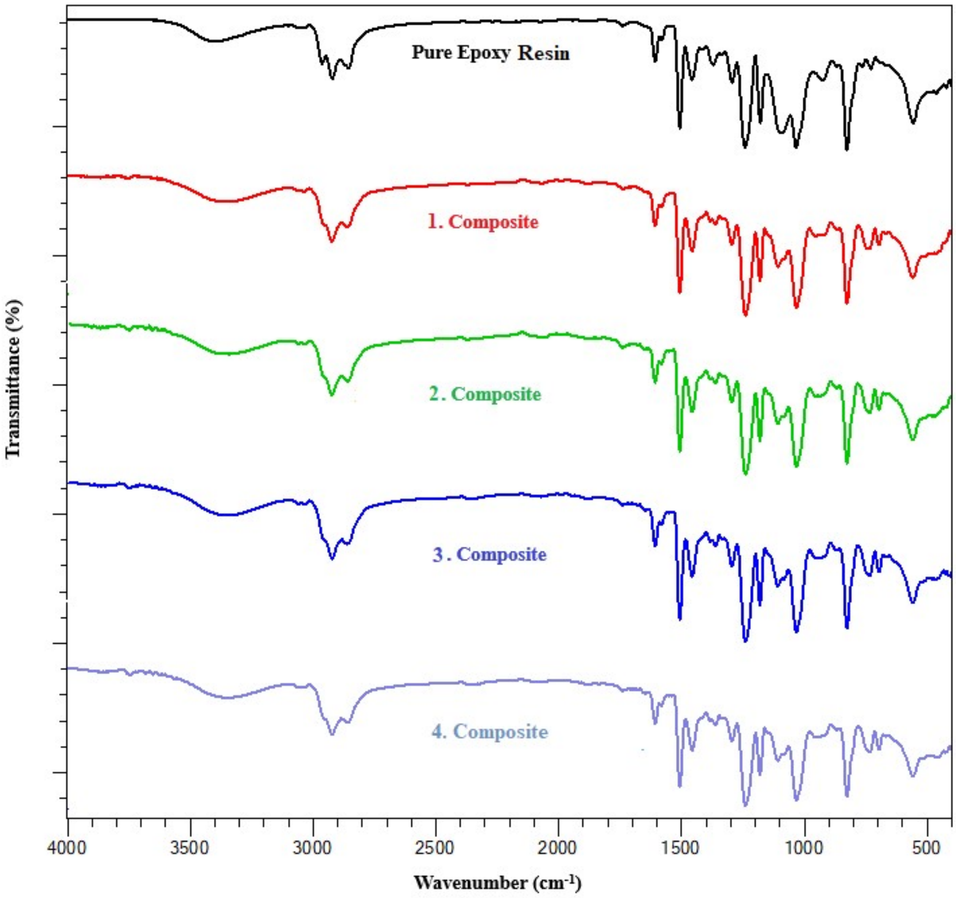

Figure 7. FTIR spectra of the pure polymer and CdS/MWCNT/epoxy nanocomposites.

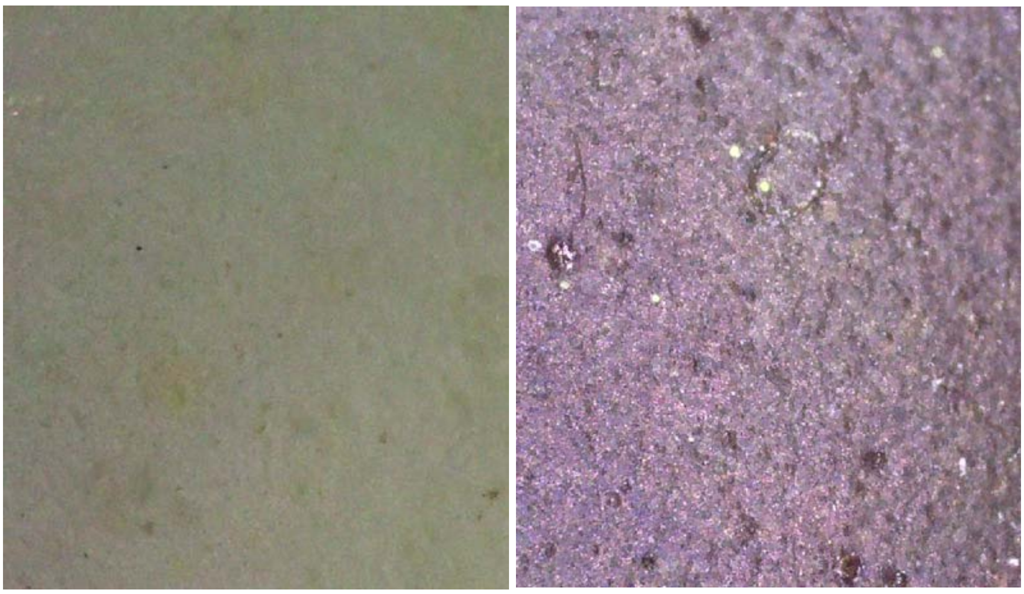

Figure 8. Microscope images of first experiment (left image) and third experiment (right image) samples.

4. Conclusion

This study comprehensively investigated the incorporation of CdS/MWCNT nanohybrids synthesized via hydrothermal methods into epoxy resin composites, focusing on their mechanical, thermal, structural, and electrical properties. The results revealed significant enhancements in key properties due to the synergistic effects of the hybrid nanostructures.

The incorporation of CdS/MWCNT nanohybrids increased the bulk density, Shore D hardness, thermal conductivity, and dielectric coefficient of the composites. These improvements were attributed to the homogeneous distribution of nanohybrids in the epoxy matrix and the strong interfacial interactions between the components. The nanohybrids filled micro-voids within the composite structure, enhancing its compactness, thermal performance, and mechanical strength. Thermal conductivity values showed a noticeable increase from 0.112 W/m·K to 0.136 W/m·K, highlighting the ability of the nanohybrids to act as efficient thermal bridges. Similarly, the dielectric coefficient rose significantly from 3.86 to 5.67, demonstrating the potential of the nanocomposites in energy storage and other dielectric applications. Microscopic analysis confirmed the effective dispersion and interfacial integration of the hybrid structures, while FT-IR analysis supported the presence of strong physical interactions, contributing to the observed property enhancements. These findings underscore the potential of CdS/MWCNT hybrid nanocomposites for advanced technological applications, particularly in fields requiring lightweight materials with high thermal conductivity, dielectric properties, and mechanical strength. Future research should explore further optimization of the hybrid structure and its application in diverse industrial sectors such as electronics, energy, and aerospace. The results provide a solid foundation for developing multifunctional materials leveraging hybrid nanostructures.

Author Contributions

The percentages of the authors' contributions are presented below. All authors reviewed and approved the final version of the manuscript.

	H.Ş.	E.A.
C	60	40
D	50	50
S	50	50
DCP	40	60
DAI	40	60
L	60	40
W	50	50
CR	50	50
SR	60	40
PM	60	40

C=Concept, D= design, S= supervision, DCP= data collection and/or processing, DAI= data analysis and/or interpretation, L= literature search, W= writing, CR= critical review, SR= submission and revision, PM= project management.

Conflict of Interest

The authors declared that there is no conflict of interest.

Ethical Consideration

Ethics committee approval was not required for this study because there was no study on animals or humans.

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