

The Effect of Nano Nickel Oxide(n-NiO) on the Hardness properties of As-Cast and Cured Nano Composite

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Abstract: In recent advancements in material science, there is a trend toward replacing existing single-phase materials with composites for enhanced control over properties. This study focuses on developing a reinforced composite using Nano Nickel Oxide(n-NiO) within an epoxy resin matrix to enhance its mechanical properties. The aim is to investigate how the matrix material affects the density, hardness of the developed composites. Composites were fabricated using a stirred dispersion with resin casting, adhering to ASTM standards for specimen preparation and testing. Tests were conducted at room temperature and under Cured conditions. Results indicate that incorporating n-NiO significantly improves the mechanical properties of epoxy resin. Magnetic stirring facilitated the homogeneous distribution of n-NiO within the epoxy matrix, followed by curing at ambient temperature. For comparative analysis, some composites underwent additional thermal curing at 80°C for 40 minutes. The impact of varying n-NiO concentrations (0.5%, 1%, 1.5%, and 2%) on the composite's properties was assessed. The curing process employed K6 hardener in a weight ratio of 1:10. Mechanical attributes such as Density, Hardness were evaluated. The composite with 1.5% n-NiO exhibited optimal enhancement in all measured properties compared to neat epoxy resin. This study concludes that the inclusion of n-NiO significantly augments the mechanical performance of the epoxy-based composite material.

Keywords: Mechanical, Nano Nickel Oxide(n-NiO), Epoxy Resin, Composite, Stirred Dispersion, Room Temperature, Oven Curing, ASTM

I. INTRODUCTION

1.1 Composite Materials

composites materials consist of two or more different components with varying chemical or physical characteristics. The matrix, which acts as a binder, holds the reinforcements or fibers together, allowing the composite to exhibit a combination of properties from the individual components, often resulting in enhanced properties that surpass those of the original materials. For example, in mud bricks, mud acts as the matrix while straw or aggregate serves as the reinforcement. Similarly, in wood, cellulose and lignin fulfill these roles, and in concrete, small stones or gravel (aggregate) are bound together by cement, demonstrating good strength under compression. Concrete can be further reinforced under tension by incorporating metal rods, wires, mesh, or cables, forming reinforced concrete.

Recent advancements have led to the development of nano composites, where nanoscale materials are incorporated into the matrix to achieve superior properties. By carefully selecting the reinforcement, matrix, and manufacturing process, properties can be tailored to match specific requirements. The choice of matrix material can also alter characteristics such as weather durability, chemical resistance, and heat resistance. Nano composites, specifically, offer significant improvements in mechanical strength, dimensional stability, barrier properties, and thermal resistance due to the incorporation of nano-structured materials.

Nano Nickel Oxide (n-NiO) is one such nanostructured material that has garnered attention for its potential applications in various fields. The incorporation of n-NiO into composite materials can enhance their hardness, mechanical strength, and thermal stability. This makes n-NiO composites highly suitable for use in industries such as aerospace, automotive, marine, electronics, and construction, where materials with exceptional performance characteristics are crucial. By leveraging the unique properties of nano-scale reinforcements like n-NiO, modern composite materials can achieve a balanced set of qualities tailored for specific, high-demand application

1.2 Stirred Resin Casting

Stirred resin casting is a method employed to create composites by mixing a liquid resin with various additives, such as nanoparticles, to enhance material properties. This process involves the careful dispersion of nanoparticles within the resin using techniques like ultrasonication and mechanical stirring to achieve a homogeneous mixture. The mixture is then degassed under vacuum to eliminate air bubbles that could compromise the integrity of the final product. The degassed resin is poured into molds and allowed to cure at room temperature or under elevated temperatures, depending on the specific resin system used. This curing process solidifies the resin, embedding the dispersed nanoparticles uniformly throughout the matrix, resulting in a composite material with improved mechanical, thermal, or electrical properties. Key factors in stirred resin casting include achieving uniform dispersion of nanoparticles, controlling mixing parameters to prevent damage to the nanoparticles, and ensuring strong interfacial bonding between the resin and additives. This method is widely used in various industries, including aerospace, automotive, electronics, construction, and biomedical devices, due to its ability to produce high-performance composites with tailored properties.

1.3 Hardness

A material's hardness is determined by how resistant it is to permanent deformation, indentation, or scratching from mechanical forces. This property is crucial in many fields, including engineering, metallurgy, and materials science, since it indicates if a material is appropriate for a given application where durability and mechanical strength are critical. To precisely measure mechanical hardness, a variety of standardized tests are used, including the Vickers, Rockwell, and Brinell hardness tests. One technique for determining the hardness of materials, especially thin sections or small areas, is the micro-Vickers hardness test. This is a variant of the classic Vickers hardness test that is intended for use with microstructures or tiny components where the standard testing method would not be feasible.

The micro-Vickers hardness test is a method used to measure the hardness of materials, particularly metals or ceramics at a very small scale. The micro-Vickers hardness test is the most adaptable and popular technique for analyzing the mechanical characteristics of materials at a microscopic size, offering insightful information on their functionality and applicability for a range of uses. It can be applied to evaluate the hardness of thin or small samples, like microstructures, thin films, or coatings. The test yields repeatable, incredibly accurate findings. The test provides for fine control over the applied force, making it useful for analyzing materials with a wide range of hardness values and it is applicable for a wide range of materials, including metals, ceramics, polymers, and composites.

1.4 Density Test

A Density assessment, often referred to as a specific gravity examination, is conducted to ascertain the weight per volume unit of a substance. Within the scope of composite materials like Epoxy based nano particles and fibre-reinforced polymers (FRPs), such density evaluations are crucial for ensuring quality, characterizing materials, forecasting performance, refining processes, and upholding the quality of the composites. The density for each composite variant is determined in accordance with the ASTM D792 guidelines

This is an important parameter in materials science and engineering because it affects various properties of the composite, including its strength, stiffness, and performance in different applications. By accurately determining the density of composites, engineers and manufacturers can make appropriate decisions in the selection of material, process optimization, and product performance. The determination of density involves contrasting the mass of a specimen in air with its mass when immersed in water. This process includes recording the specimen's mass in air and then noting the reduction in apparent mass when it is submerged in water. The material's density is derived using the equation as seen in below lines :

This method utilizes the concept of Archimedes' principle, which posits that an object submerged in a fluid is subject to a buoyant force equivalent to the weight of the fluid it displaces. By assessing the variance in the sample's weight when measured in air versus submerged in water, one can deduce the volume of the displaced water. Consequently, this allows for the calculation of the sample's density

II. MATERIALS AND METHODOLOGY

2.1 Material selection

Compositions of composites include the following materials:

1. THF
2. Epoxy Resin
3. Nano Nickel Oxide.

THF : Tetrahydrofuran (THF) is a versatile organic solvent frequently employed in the preparation of polymer nanocomposites due to its excellent solvency properties and low boiling point. In the stirred resin casting process, THF is utilized to dissolve polymethyl methacrylate (PMMA), creating a homogeneous polymer solution that facilitates the uniform dispersion of nanoparticles. The process begins with the dissolution of PMMA in THF, followed by the addition of nanoparticles, which are evenly distributed throughout the polymer matrix using mechanical stirring or ultrasonication. This step is crucial for achieving a homogeneous mixture, preventing agglomeration, and ensuring the optimal performance of the nanocomposite. The resultant PMMA-nanoparticle solution is then poured into molds, and the THF is allowed to evaporate, leaving a solidified composite material. Degassing under vacuum may be employed to eliminate air bubbles and defects. The evaporation of THF is typically performed at room temperature or with gentle heating to accelerate the process. Due to its flammability and volatility, THF must be handled with appropriate safety precautions, including the use of fume hoods and personal protective equipment. The use of THF in stirred resin casting enables the creation of PMMA-based nanocomposites with enhanced mechanical, thermal, and optical properties, making it an invaluable solvent in materials science and polymer chemistry.

Nano Nickel Oxide: Nickel, classified as a block D, period 4 element, combines with oxygen, a block P, period 2 element, to form nickel oxide nanoparticles (NiO NPs). These metal oxide nanoparticles are available in both powder and dispersion forms. Nano Nickel Oxide exhibits a wide array of properties, including high electrical conductivity, thermal stability, catalytic activity, and magnetic properties. These attributes make NiO NPs highly valuable in a variety of applications, such as catalysis, energy storage, sensors, electronic devices, and environmental remediation. Proper dispersion of Nano Nickel Oxide in a suitable medium, such as THF/ethanol, is crucial for maximizing their functionality and ensuring uniform distribution within the host material. Achieving a stable and homogeneous dispersion of NiO NPs is essential for unlocking their full potential and leveraging their unique properties in diverse fields, from advanced materials to biomedical and environmental technologies. The optimization of dispersion techniques is critical for achieving the desired performance of nanoparticle-based products and formulations, thereby ensuring their success in various industrial and research applications.

Epoxy Resin : Epoxy resin is a widely utilized thermosetting polymer known for its remarkable mechanical properties, superior adhesion, chemical resistance, and thermal stability. Formed through the reaction of epoxide groups with curing agents, epoxy resin results in a rigid, durable material ideal for a broad spectrum of applications, including coatings, adhesives, composites, and electronics. Its high tensile and compressive strength make it suitable for structural uses, while its excellent adhesion ensures effective bonding with a variety of substrates, such as metals, ceramics, and plastics. Furthermore, epoxy resin's resistance to chemicals and solvents, along with its ability to maintain properties at elevated temperatures, enhances its functionality in harsh environments. Additionally, its exceptional dielectric properties make it an essential material in the electrical and electronics industries. These characteristics collectively contribute to the extensive application and ongoing research into epoxy resin, particularly in developing advanced composites and high-performance materials.

2.2 Methodology: Nano Nickel Oxide was acquired to serve as a reinforcing agent in the preparation of composites, with a specific ratio carefully selected to achieve targeted improvements. The nanoparticles were dispersed in Tetrahydrofuran (THF) using an ultrasonicator over a defined duration at a chosen frequency to ensure consistent distribution. Epoxy resin, quantified by weight, was blended with the dispersed Nano Nickel Oxide using a magnetic stirrer set at specified revolutions per minute (rpm) to attain a homogeneous mixture. A hardener, in a predetermined ratio, was selected for the subsequent curing process. Through thorough integration using a magnetic stirrer at a controlled speed, the uniformly dispersed nanoparticles were uniformly incorporated into the resin blend, ensuring a uniform foundational solution for the composite. To facilitate easy extraction, the mold was initially treated with a wax layer to prevent bonding between the interior of the mold and the sample. The resin mixture was then carefully transferred into the pre-treated mold, temporarily adjusted using spacers to achieve the desired thickness and dimensions. The material underwent natural curing (As-Cast) within the mold cavity for the specified duration. Following this initial curing phase, the specimen was removed from the mold and subjected to additional post-curing. After the natural curing phase, the material underwent an additional step involving heating in a hot oven up to 80°C for 40 to 45 minutes, followed by storage in a controlled environment to ensure complete curing. Subsequently, samples were segregated based on whether they underwent natural or oven curing and then prepared to ASTM standards using a water jet cutting machine for subsequent mechanical testing procedures. This systematic approach ensures the effective dispersion and curing of Nano Nickel Oxide in epoxy resin, facilitating the production of high-quality composites suitable for comprehensive mechanical property analysis.



III. EXPERIMENTATION

3.1 Material composition

The research focuses on varying the concentration of Nano Nickel Oxide particles in an epoxy resin as shown in Table 1 while maintaining a consistent formulation. The epoxy and hardeners are fixed in a 1:10 ratio by weight, ensuring stability across different compositions.

Guidelines limit the total nanoparticle content to 2% by weight, prompting meticulous control over composite composition. This results in a hybrid composite predominantly composed of 98% epoxy blend, with the remaining 2% allocated to nanoparticles. Essential components like hardeners and Tetrahydrofuran (THF) are integrated into the composite to aid in the curing process.

The study explores a range of Nano Nickel Oxide concentrations from 0.5% to 2% within the confines of the 2% total nanoparticle content, excluding THF and hardeners. This systematic variation allows for a detailed assessment of how nanoparticle proportions affect composite properties. By treating the entire 2% as a unified range for nanoparticle variation, the research aims to provide comprehensive insights into optimizing the performance of nano-enhanced epoxy composites

Table 1 Specimen Composition of Different Composition

Sl. No.	Resin (Epoxy) ((Excluding Hardener Weight)	Nano Nickel oxide Reinforcement (Excluding THF Solvent Weight)
1.	99.5%	0.5%
2.	99%	1%
3.	98.5%	1.5%
4.	98%	2%
5.	100%	---

3.2 Testing

The prepared composite specimen is subjected to Hardness test conducted using micro-Vickers hardness tester as per ASTM E384 standards and Density test performed as per ASTM D792

Density Test

The geometry of Density test specimen is shown in Figure1 as per ASTM D792 standards. Specimen length is 25mm, breadth is 25mm and thickness is 10mm. ASTM D792 is a widely recognized standard test method used to determine the density and specific gravity of plastics by the displacement method. This test is crucial for characterizing plastic materials in research and quality control. According to ASTM D792, the density of a plastic specimen is determined by weighing the sample in air and subsequently in a liquid of known density, typically distilled water.

The test involves immersing the specimen in the liquid and measuring the apparent loss in weight due to buoyancy, which corresponds to the volume of the displaced liquid. By accurately measuring the mass of the specimen in air and in the liquid, and knowing the density of the liquid, the density of the specimen can be calculated using the principle of Archimedes.

This method provides precise and reproducible measurements of density, essential for identifying material compositions, assessing material quality, and ensuring compliance with material specifications. The standard specifies detailed procedures for sample preparation, conditioning, and measurement, ensuring consistent and reliable results across different laboratories and applications



Figure 1 Geometry of Density Test Sample (All Dimensions are in mm)

Hardness Test

The geometry of hardness test specimen is shown in Figure 2 as per ASTM E384 standards. Specimen length is 25mm, breadth is 25mm and thickness is 10mm. Micro Vickers hardness test is performed for characterizing the mechanical properties of materials at a microscopic scale, providing valuable insights into their performance and suitability for various applications

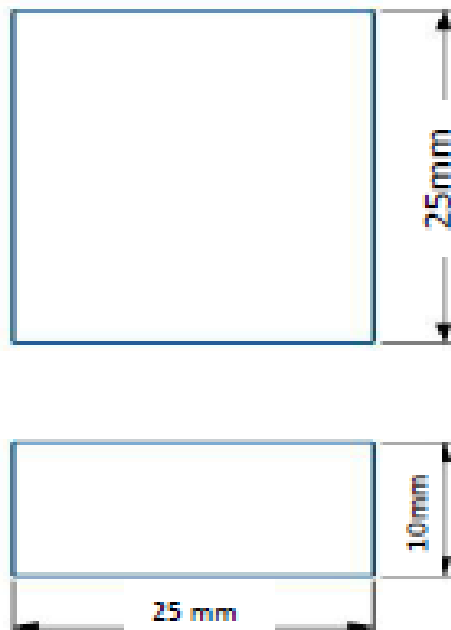


Figure 2 Geometry of Hardness Test Sample (All Dimensions are in mm)

IV. RESULTS AND DISCUSSIONS

4.1 Hardness Test Results

The hardness test results of the developed composites are shown in the Table 2 and Figure 3.1,3.2

Table 2 Hardness Result at As-Cast and Cured condition

Sl no.	Composite Code	% of Nano Nickel Oxide	VHN (As-Cast)	VHN (Cured)
1	PURE EPOXY(N0)	--	24.8	28.3
2	A(N0.5)	0.50%	26	28.6
3	B(N1)	1%	29.9	30.1
4	C(N1.5)	1.50%	30.8	34
4	D(2)	2%	26.4	28.86

The micro-Vickers hardness value (VHN) of the developed composite materials with different concentrations of nano nickel oxide is shown in Table 2. Based on the Vickers hardness test results obtained for the specimens, it is evident that there exists a significant diversity in hardness levels among the samples. The base material, pure epoxy (Composite Code N0), exhibited a VHN of 24.8 when As-Cast and 28.3 when Cured.

Adding 0.5% nano nickel oxide (Composite Code N0.5) resulted in a slight increase to 26 VHN (+4.84%) for As-Cast specimens and 28.6 VHN (+1.06%) for Cured specimens. A more substantial increase in hardness was observed with a 1% additive concentration (Composite Code N1), reaching 29.9 VHN (+20.56%) for As-Cast and 30.1 VHN (+6.36%) for Cured specimens.

The highest hardness among As-Cast specimens was achieved with a 1.5% additive concentration (Composite Code N1.5), yielding a VHN of 30.8 (+24.19%), while the Cured specimens at the same concentration showed the highest overall hardness with a VHN of 34 (+20.14%). Interestingly, increasing the additive concentration to 2% (Composite Code N2) led to a decrease in hardness to 26.4 VHN (+6.45%) for As-Cast and 28.86 VHN (+2.05%) for Cured specimens.

This disparity in hardness values indicates varying degrees of resistance to indentation or deformation across the specimens. The observed variations in hardness may stem from differences in material composition, micro structural characteristics, or manufacturing processes utilized during fabrication. The results clearly demonstrate that the hardness of epoxy composites is significantly influenced by both the concentration of nano nickel oxide additives and the curing method. Cured specimens generally exhibited higher hardness values compared to As-Cast ones, with the optimal hardness achieved at a 1.5% additive concentration for both curing methods. These findings underscore the essential role of hardness testing in evaluating material properties and assessing their suitability for specific applications, particularly in scenarios where mechanical strength and durability are paramount.

4.2 Density test

The Density test results of the composite specimen are shown in Table 3 and Fig 4.1,4.2

Table 3 Density Test at As-Cast and Cured condition

Sl no.	Composite Code	% of Nano Nickel Oxide	Density (As-cast) in kg/m ³	Density (Cured) in kg/m ³
1	PURE EPOXY(N0)	----	1154.158	1159.679
2	A (N0.5)	0.50%	1174.258	1178.775
3	B (N1)	1%	1180.163	1182.487
4	C (N1.5)	1.50%	1183.153	1184.624
4	D (2)	2%	1164.48	1167.632

The density of composite materials with varying concentrations of nano nickel oxide was investigated, and the results are presented in Table 3. The data reveal significant variation in density among the samples, indicating the impact of nano nickel oxide concentration and curing methods. Pure epoxy (Composite Code N0) exhibited a density of 1154.158 kg/m³ when As-Cast and 1159.679 kg/m³ when Cured.

Adding 0.5% nano nickel oxide (Composite Code N0.5) increased the density to 1174.258 kg/m³ for As-Cast specimens and 1178.775 kg/m³ for Cured specimens, representing a 1.74% and 1.64% increase, respectively. Increasing the concentration to 1% (Composite Code N1) resulted in densities of 1180.163 kg/m³ and 1182.487 kg/m³, demonstrating a 2.25% and 1.97% increase.

The highest density was observed with a 1.5% additive concentration (Composite Code N1.5), yielding 1183.153 kg/m³ for As-Cast and 1184.624 kg/m³ for Cured specimens, corresponding to a 2.51% and 2.15% increase. However, a 2% concentration (Composite Code N2) led to a decrease in density to 1164.48 kg/m³ for As-Cast and 1167.632 kg/m³ for Cured specimens, showing a 0.89% and 0.68% increase, respectively.

The reduction in density at higher additive concentrations suggests potential agglomeration or other micro structural changes that negatively affect material compactness. These variations underscore the critical role of nano nickel oxide concentration and curing methods in optimizing composite properties.

The results indicate that a 1.5% nano nickel oxide concentration enhances the density of epoxy composites for both curing methods. However, the observed decrease at the 2% concentration warrants further investigation. This study highlights the importance of density testing in evaluating material properties and their suitability for specific applications, particularly where material compactness and integrity are paramount.

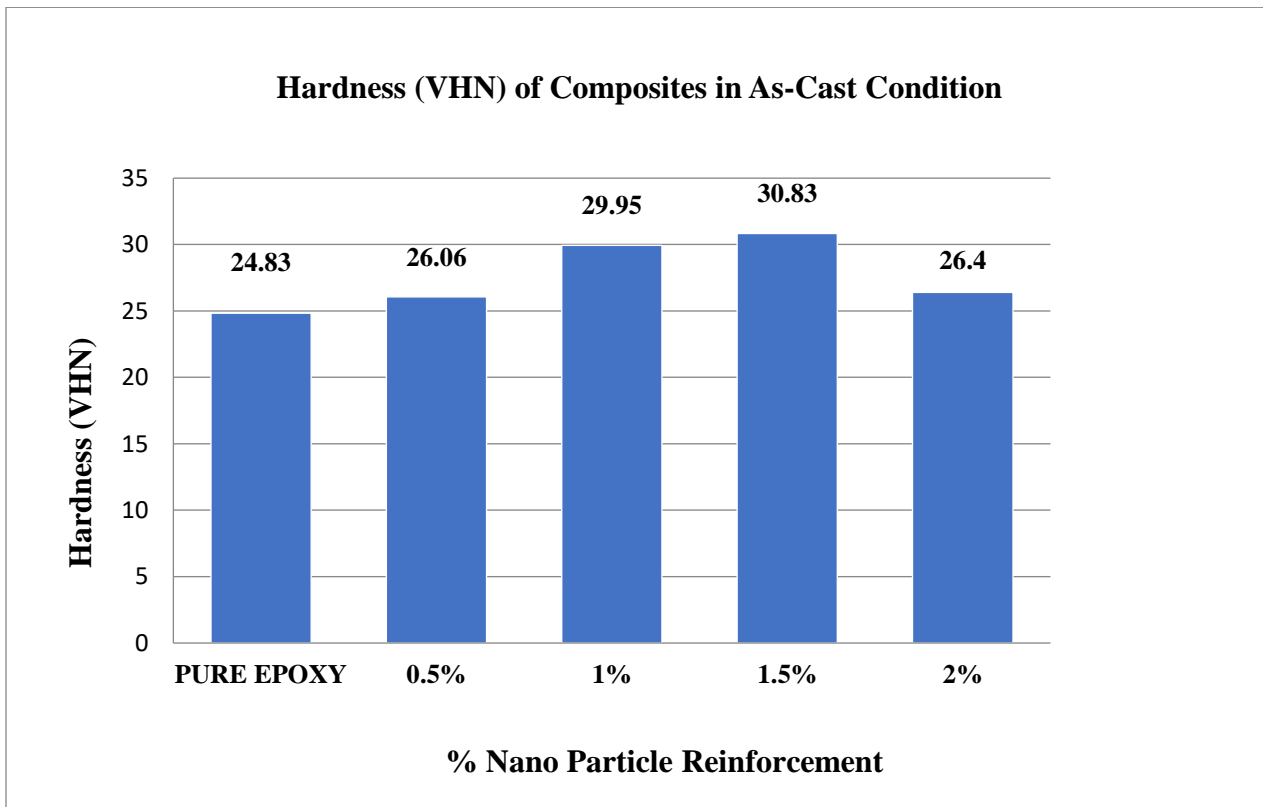


Figure 3.1: Effect of Nano Nickel Oxide on the Hardness (VHN) of Epoxy Composites Reinforced with Different Percentages of Nano Nickel Oxide in As-Cast condition

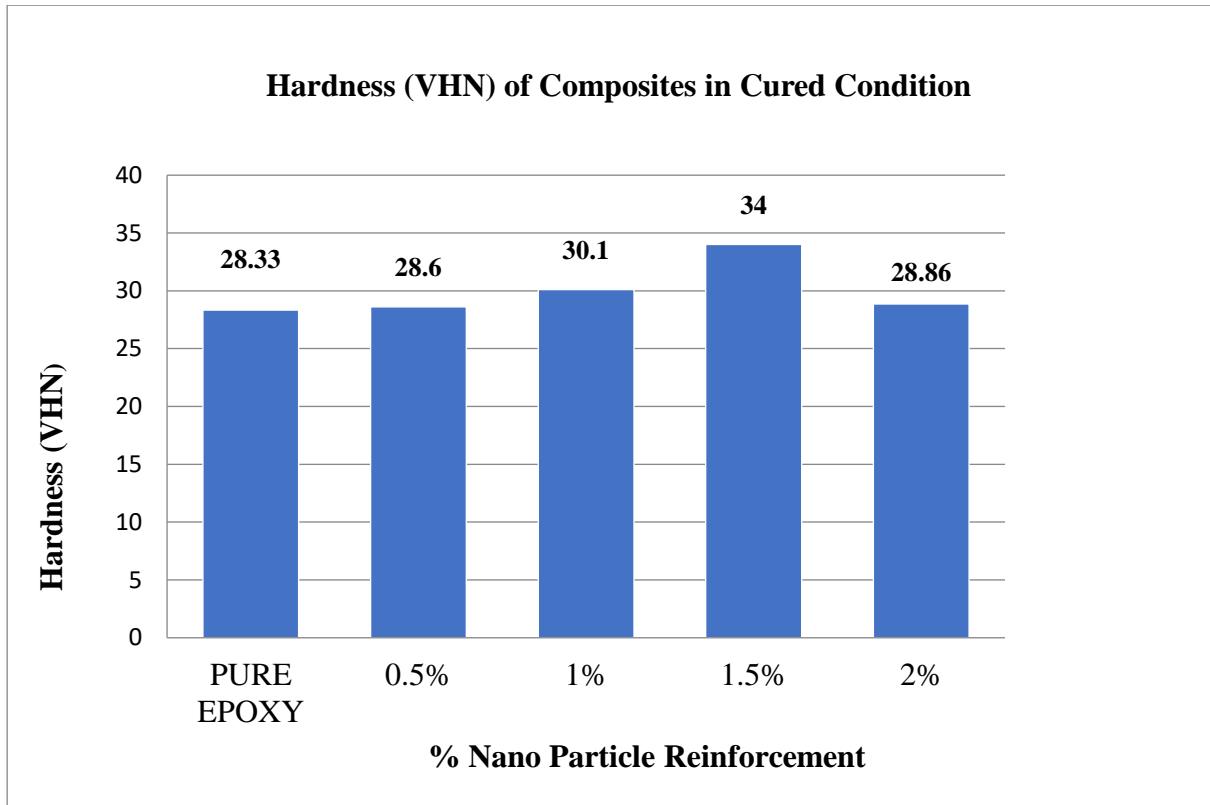


Figure 3.2: Effect of Nano Nickel Oxide on the Hardness (VHN) of Epoxy Composites Reinforced with Different Percentages of Nano Nickel Oxide in Cured condition

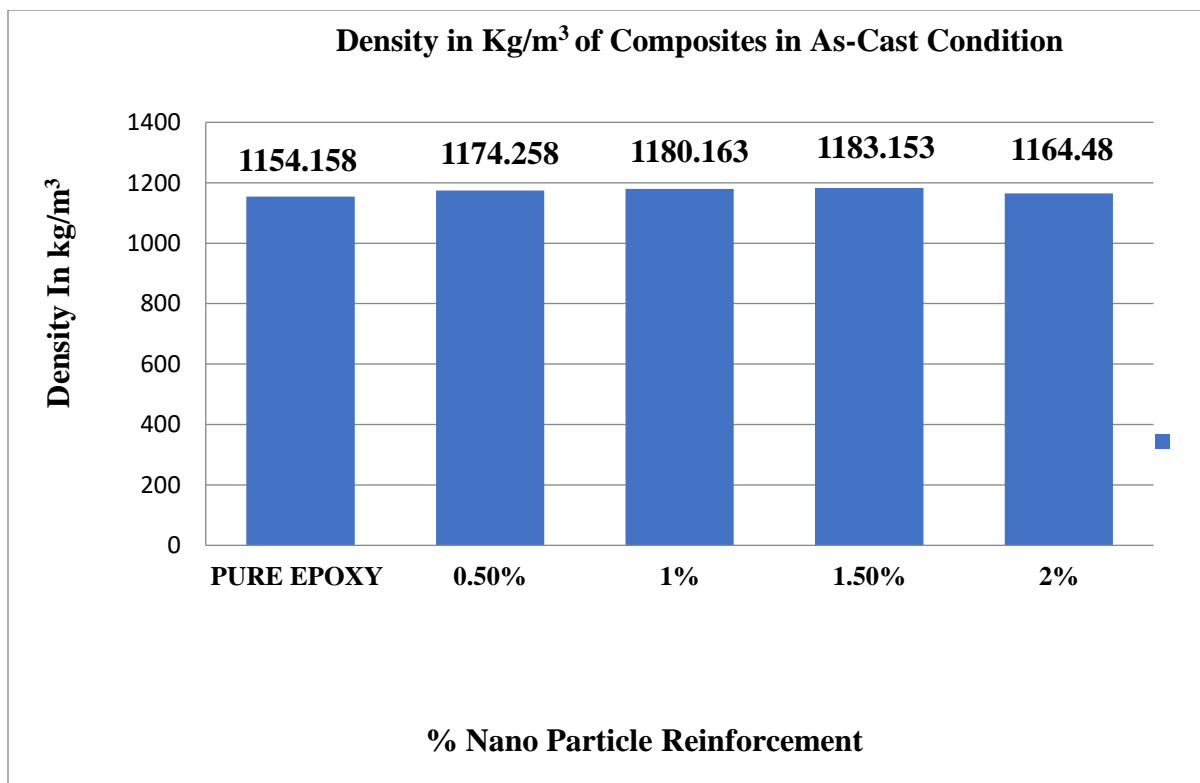


Figure 4.1 : Effect of Nano Nickel Oxide on the Density (kg/m^3) of Epoxy Composites Reinforced with Different Percentages of Nano Nickel Oxide in As-Cast condition

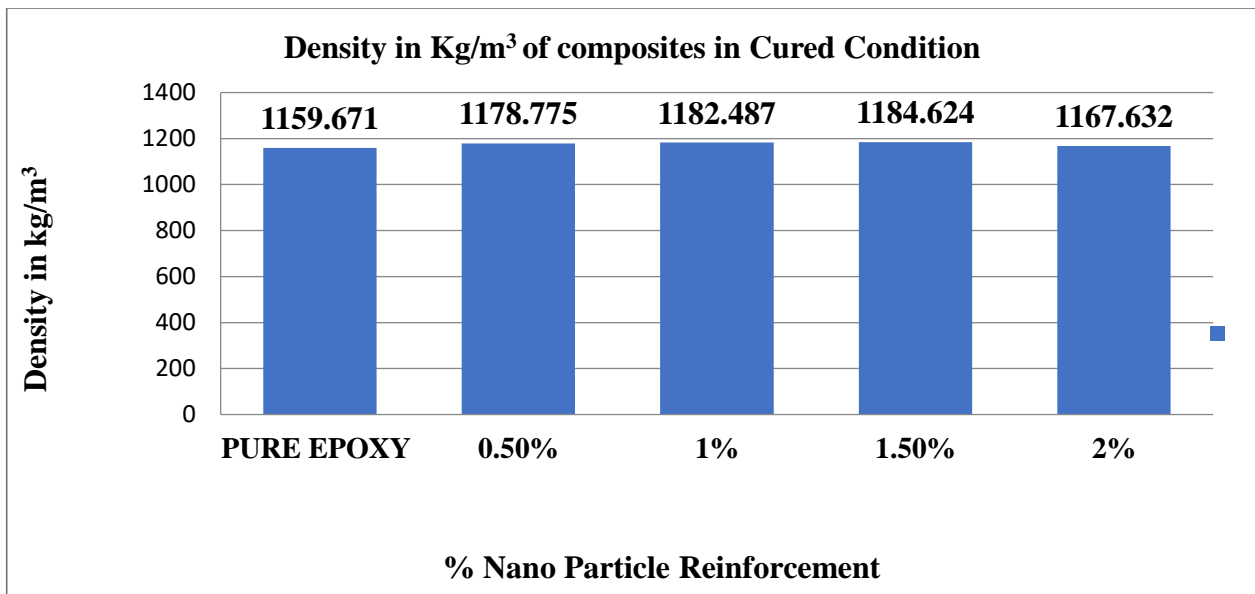


Figure 4.2 : Effect of Nano Nickel Oxide on the Density (kg/m³) of Epoxy Composites Reinforced with Different Percentages of Nano Nickel Oxide in Cured condition

V. CONCLUSION

In conclusion, the study demonstrates the potential and feasibility of Epoxy resin and Nano Nickel Oxide-based composite material by enhancing the mechanical properties. The conclusions drawn from this study are as follows:

- **The hardness** of epoxy composites is influenced by both the percentage of additive and the curing method. The Cured specimens generally exhibited higher hardness values compared to the As-Cast ones.
 - As-Cast: The highest hardness was observed with 1.5% additive. Beyond this concentration, the hardness decreased.
 - Cured: The highest hardness was also observed with 1.5% additive, demonstrating a significant improvement over pure epoxy.
- The results indicate that a 1.5% additive concentration provides the optimal increase in hardness for both curing methods, with the Cured process yielding the highest hardness overall. Further studies could explore the reasons for the decrease in hardness at 2% additive and examine other properties affected by these curing methods.
- **The Density** of epoxy composites is influenced by both the percentage of additive and the curing method. The Cured specimens generally exhibited higher density values compared to the As-Cast ones.
 - Nano Nickel Oxide additives generally increase the density of epoxy resin, except for the 2% concentration in As-Cast specimens, which showed a slight decrease.
 - curing tends to result in slightly higher densities compared to natural curing for both pure epoxy and epoxy with Nano Nickel Oxide additives.
- Nano Nickel Oxide additives can effectively increase the density of epoxy resin, especially at lower concentrations (0.50% to 1.50%), regardless of the curing method. However, the impact on density varies with concentration and curing conditions, suggesting that careful selection of additive concentration and curing method is crucial to achieve desired physical properties in epoxy resin applications.

These findings contribute to the advancement of composite materials, opening new opportunities for innovation and application in various industries

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