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# Enhancing epoxy nanocomposites with mineral nanofillers: A comprehensive review of particle size, shape, and concentration

## ABSTRACT

*Mineral nanofillers have emerged as promising reinforcements for epoxy nanocomposites, offering enhanced mechanical, thermal, and physical properties of the latter. This review comprehensively explores the influence of mineral nanofiller morphology, surface modification, and loading on the performance of epoxy nanocomposites. The size and shape of nanoparticles significantly affect the interfacial interactions and dispersion within the epoxy matrix, with smaller particles and higher aspect ratios generally leading to improved properties. Surface modification techniques, such as the use of coupling agents, polymer grafting, and functionalization, are crucial for optimizing the compatibility and bonding between nanofillers and epoxy matrix. The loading concentration of mineral nanofillers plays a critical role in determining the final properties of nanocomposites, with optimal loadings varying depending on the specific nanofiller and the desired enhancements. Key mineral nanofillers, including nano-silica, nano-clay, nano-calcium carbonate ($CaCO_3$), and nano-talc, have demonstrated remarkable improvements in mechanical strength, thermal stability, and barrier properties when incorporated into epoxy matrix. This review provides valuable insights into the complex interplay between nanofiller characteristics and epoxy nanocomposite performance, guiding the design and development of advanced materials for various applications.*

***Keywords:****Epoxy composites, Mineral nanofillers, Particle size, Particle shape, Morphology, Surface modification, Filler Loading, Nano-silica, Nano-clay, Nano-$CaCO_3$, Nano-Talc*

## 1. INTRODUCTION

Epoxy composites are renowned for their exceptional strength and resistance properties; however, enhancements are needed for high-performance applications, where mechanical strength, dielectric properties, thermal resistance, optical properties, and barrier properties are crucial. Recent studies have concentrated on integrating advanced materials, especially nanomaterials, into epoxy composites to enhance their characteristics; particularly in terms of mechanical, thermal, and electrical properties. The integration of carbon nanofillers, such as graphene and carbon nanotubes (CNTs), can enhance the mechanical strength, fatigue resistance, and electrical conductivity of these materials, particularly in the aerospace and automotive industries[1].

Researchers have noted that the inherent electrical conductivity of graphene and CNTs limits their use in dielectric applications [2,3]. Although grafting and surface modification may help, these processes are cost-prohibitive for large-scale production [4,5]. Carbon nanotubes (CNTs) are associated with a range of health risks, including cytotoxicity, inflammation, fibrosis, genotoxicity, tumorigenesis, and immunotoxicity [6,7]. Additionally, the propensity of graphene to form agglomerates [8,9] introduces further challenges. Researchers are seeking alternatives to carbon-based materials that are cost-effective, safe, and easy to process while maintaining mechanical strength, dielectric properties, and other essential characteristics. Mineral nanofillers offer advantages over carbon-based materials, addressing limitations such as dielectric properties and costs, while forming stronger bonds with polymer matrices [10,11]. The ability of mineral nanofillers to adjust their surface properties, including surface energy[12], surface functionalization[13], and morphology through variations in shape and size[14], enhances their dispersion within polymer

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matrices. This leads to improved mechanical reinforcement without the extensive modification techniques required for carbon-based fillers. While carbon materials, such as carbon nanotubes, offer exceptional properties, including high tensile strength and electrical conductivity, these benefits are often compromised by the need for functionalization to ensure matrix interaction and dispersion[15,16]. While carbon nanofillers, such as carbon nanotubes (CNTs) and graphene, are acclaimed for their multifunctionality in electronics and structural materials[17], mineral nanofillers enhance ductility and toughness[18], improve biocompatibility[19], reduce toxicity[20], and facilitate the development of hybrid formulations[21]. These materials are more sustainable and readily available, aligning with trends towards environmentally friendly materials [22]. This sustainability makes them appealing for applications that prioritize ecological concerns. Their lower cost compared to carbon-based fillers provides a financial advantage, which is crucial for broadening their production and application across sectors[23,24].

Mineral nanofillers, including clay[25,26] Wollastonite[27], alumina[28], calcium carbonate ($CaCO_3$), talc [29], and halloysite[30], have been researched extensively with materials such as polypropylene, polyethylene, polyamides, polyvinyl chloride, and polylactic acid for developing thermoplastic composites. The integration of thermoset materials with mineral nanofillers is a significant trend in nanocomposite research. Examples include calcium carbonate in unsaturated polyesters and phenolics[31], aluminium hydroxide in polyurethane[32], silica in phenolics[33,34] and talc in unsaturated polyesters[35]. Epoxy is a prominent matrix resin in nanocomposites owing to its high mechanical strength, chemical resistance, and adhesive properties, and has been widely used in the automotive and aerospace industries[1,36,37]. The increasing demand for high-performance applications has prompted researchers to explore methods to enhance the sustainability and cost-effectiveness of epoxy nanocomposites. The incorporation of mineral nanofillers into epoxy resins may represent a promising solution that has attracted the interest of researchers. Although carbon-based nanofillers are renowned for their exceptional performance characteristics, this review does not compare them with other nanofillers. Instead, it focuses on examining the morphological characteristics of mineral nanofillers to enhance their performance in epoxy resins. This review analyzes the influence of the morphology of key mineral nanofillers, such as nano-silica, clay, calcium carbonate, and talc, on the mechanical durability, electrical properties, thermal stability and flame resistance of epoxy composites.

## 2. EPOXY RESIN

Epoxy resins, frequently referred to as polyepoxides, are a category of thermosetting polymers distinguished by the inclusion of two or more three-membered epoxide (oxirane) rings in their molecular structure (Fig. 1).

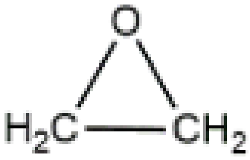


*Figure 1. Epoxide Ring*

Epoxy resins have become significant polymeric matrices for composites because of their exceptional mechanical properties, chemical resistance, and adaptability. These thermosetting polymers form strong crosslinked networks during curing, ensuring excellent adhesion to the reinforcing fibers and fillers. Composites made from epoxy exhibit high strength-to-weight ratios, dimensional stability, and resistance to environmental degradation, making them suitable for various applications[38]. The ability to customize epoxy formulations allows the modification of composite properties, such as toughness, thermal stability, and electrical conductivity. Additionally, epoxy matrices provide advantageous processability and support various manufacturing methods, including resin-transfer molding, filament winding, and autoclave curing[39]. As advancements in epoxy chemistry continue, new formulations with improved performance characteristics are being developed, thereby broadening the applications of epoxy-based composites in advanced fields.

Standard epoxy resin systems typically exhibit tensile strengths between 90 and 120 MPa, tensile moduli ranging from 3100 to 3800 MPa, and glass transition temperatures (Tg) varying from 150 to 220 °C [40]. These characteristics render them suitable for various high-performance applications. The benefits of epoxy resins include their remarkable mechanical strength, superior adhesion to fibers, resistance to chemicals and moisture, minimal curing shrinkage, and adaptable processing options. However, they may exhibit brittleness, necessitating the incorporation of modifiers to enhance toughness. Curing duration can occasionally be prolonged, and certain epoxy systems may pose a risk of skin sensitization [41].

Epoxy matrix composites are frequently employed in aerospace applications, particularly in aircraft components, where a high strength-to-weight ratio is essential [42]. They are also widely utilized in automotive manufacturing for structural and body components, aiding in weight reduction initiatives [43,44]. In the marine sector, epoxy composites are used in boat hulls and various marine structures because of their durability and water resistance [45]. These materials are used in the construction industry for structural reinforcement and as protective coatings [46]. Furthermore, epoxy resins play a crucial role in electronics by encapsulating delicate components and producing circuit boards [47]. They are also used in high-performance sporting goods that require durability[48].

Recent developments in nanotechnology have facilitated the integration of nanomaterials into epoxy matrices, resulting in nanocomposites with enhanced mechanical and functional properties of epoxy matrices. These nanocomposites exhibit increased fracture toughness, thermal conductivity, and barrier properties, which expand their potential for use in extreme conditions and high-performance applications [49,50]. The rise of bio-based epoxy resins sourced from renewable materials is also gaining momentum, addressing environmental issues and potentially lowering the carbon footprint of composite materials [51,52].

*Table 1. Properties of epoxy resin and its significance in composites [1,53,54].*

| Property | Description | Significance in Composites |
|---|---|---|
| Strength | It has excellent tensile, compressive, and flexural strength. | It provides structural integrity to composite materials, thereby enabling load-bearing applications. |
| Modulus | It has high stiffness and rigidity. | This contributes to the overall stiffness and reduces deformation under load. |
| Adhesion | It bonds well with various reinforcing fibers (glass, carbon, aramid, and natural fibers). | This ensures efficient stress transfer between the fiber and matrix, maximizing the strength and durability of the composite material. |
| Chemical Resistance | It has resistance to a wide range of chemicals, solvents, and moisture. | It is suitable for application in harsh environments and provides long-term durability. |
| Thermal Resistance | It has good performance at elevated temperatures (achievable using curing agents). | This allows its use in applications involving thermal cycling and high operating temperatures. |
| Electrical Insulation | It has excellent dielectric properties | This is ideal for electrical and electronic applications that require insulation. |
| Shrinkage | Minimal shrinkage occurs during curing. | This reduces the internal stresses in the composite, leading to better dimensional stability and a reduced risk of cracking in the final product. |
| Processing | Various composite manufacturing techniques can be employed | It offers flexibility in terms of component design and production. |
| Durability | It provides long-lasting performance and resistance to fatigue and wear. | Extending the service life of composite components. |
| Strength-to-weight ratio | When combined with lightweight fibers, a high strength-to-weight ratio was obtained. | This is crucial for aerospace, automotive, and sports equipment, where weight reduction is essential for their performance. |

## 3. MINERAL NANOFILLERS

Mineral nanofillers are inorganic particles ranging from 1 to 100 nm in size that, when evenly distributed in a polymer matrix, greatly improve the mechanical strength, thermal stability, flame resistance, and barrier properties of the matrix [55].Their diverse shapes, such as platelets, fibers, tubes, and spheres, along with their high surface-to-volume ratio, form extensive interfaces with the host polymer. This interaction limits the chain mobility and enhances the load transfer [56]. Commonly used mineral nanofillers include montmorillonite, talc, calcium carbonate, silica, halloysite, aluminium hydroxide, and magnesium hydroxide, which have been studied and are listed in Table 2 with their properties and applications.

*Table 2. Properties andapplications of common mineral nanofillers integrated polymeric nanocomposites*

| Mineral Nanofiller | Key Properties Imparted to Nanocomposites | Common Applications in Nanocomposites | Ref. |
|---|---|---|---|
| Nano-clays | | | |
| Montmorillonite (MMT) | Improved mechanical strength, stiffness, thermal stability, barrier properties (gas/moisture) | Packaging, automotive parts, coatings, flame retardants | [57,58] |
| Halloysite Nanotubes (HNTs) | Improved mechanical properties, thermal stability, potential for controlled release/self-healing, reduced flammability | Coatings, drug delivery systems, tissue engineering | [56,59] |
| Talc | Improved stiffness, dimensional stability, heat resistance, nucleation agent | Automotive parts, appliances, packaging | [60,61] |
| Metal Oxides | | | |
| Silica ($SiO_2$) | Enhanced mechanical strength, hardness, scratch resistance, rheology control, thermal stability | Coatings, adhesives, dental composites, electronics packaging | [62,63] |
| Titanium Dioxide ($TiO_2$) | UV absorption, photocatalytic activity, improved mechanical properties, thermal stability | Self-cleaning surfaces, sunscreens, catalysts, protective coatings, sensors | [64,65] |
| Aluminium Oxide ($Al_2O_3$) | Improved thermal conductivity, electrical insulation, hardness, wear resistance | Electronics packaging, thermal interface materials, cutting tools, wear-resistant coatings | [66,67] |
| Zinc Oxide (ZnO) | UV filtering, antimicrobial activity, improved mechanical/thermal properties, anti-corrosion | Coatings (anti-corrosion, antimicrobial), cosmetics, sensors, varistors | [68,69] |
| Magnesium Oxide (MgO) | Improved thermal conductivity, mechanical properties, flame retardancy | Electronics, high temperature applications, biomedical application | [70,71] |
| Carbonates | | | |
| Calcium Carbonate ($CaCO_3$) | Improved stiffness, impact strength, processability, cost reduction | Packaging films, pipes, profiles, automotive interiors, biomedical applications | [72,73] |
| Sulfates | | | |
| Barium Sulfate ($BaSO_4$) | Increased density, improved stiffness, chemical resistance, corrosion resistant | Automotive parts, medical implants, tissue engineering, coatings, radiation protection application | [74,75] |
| Other Minerals | | | |
| Mica | Improved stiffness, thermal stability, electrical insulation | Electrical insulation, coatings, automotive parts, | [76,77] |
| Wollastonite | Improved mechanical properties (stiffness, strength), dimensional stability, anticorrosive | Automotive parts, coatings, medical application | [27,78] |

## 4. MINERAL NANOFILLERS INTEGRATED EPOXY COMPOSITES

The integration of mineral nanofillers into epoxy composites significantly enhances their mechanical, thermal, and physical properties, making them suitable for various advanced applications. Different types of mineral nanofillers have been studied for their effectiveness in improving the performance of epoxy composites. When incorporated into carbon-fiber-reinforced epoxy composites, zinc oxide (ZnO) nanofillers significantly improve the mechanical and thermal properties. The maximum tensile and flexural strengths increased at higher ZnO concentrations, along with a significant improvement in thermal stability. This can be attributed to the enhanced fiber-matrix adhesion, reduced void content, and improved load-transfer capabilities [79]. Additionally, ZnOnanofillers have shown promise in enhancing the mechanical and anti-corrosion properties of epoxy composites, making them valuable for applications such as UV-protective coatings and in the chemical industry [80]. MXenes, known for their high electrical and thermal conductivities, are becoming popular nanofillers in epoxy resin systems. Their chemical compatibility with epoxy matrices enhances the performance of composites in applications such as electromagnetic interference shielding and protective coating [81]. The incorporation of nanofillers, such as silica ($SiO_2$) and cobalt ferrite ($CoFe_2O_4$) Hybrid Nanofillers,resulted in superior mechanical properties in glass fiber-reinforced composites. This is due to the good fiber/matrix interfacial bonding, as observed in the hybrid nanocomposites [82]. Incorporating Polyhedral OligomericSilsesquioxane (POSS) nanoparticles into carbon fiber-reinforced epoxy composites significantly enhances their mechanical properties owing to the homogeneous distribution of the nanofillers within the matrix [83]. Aluminum Oxide ($Al_2O_3$) fillers contribute to the mechanical strength, wear resistance, and hardness of basalt/epoxy composites. The addition of these nanofillers led to a reduction in wear loss and an improvement in certain mechanical properties, although with varying effects on the tensile strength and hardness, depending on the filler percentage [84]. Each type of nanofiller uniquely contributes to the overall enhancement of the epoxy composite properties, offering tailored solutions depending on specific application requirements. From a logical standpoint, the improvement in the properties is linked to factors such as morphology, interfacial interactions, dimensionality, and filler loading content. These aspects should be prioritized when examining the performance characteristics of mineral-nanofilled epoxy composites. Sections 4.1, 4.2, and 4.3 explore the impact of the morphology of essential mineral nanofillers, including nano-silica, clay, calcium carbonate, and talc, on the properties of epoxy composites.

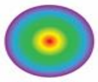
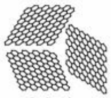
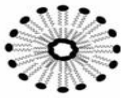
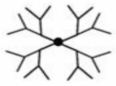
### *4.1. The influence of mineral nanoparticle size and shape on the characteristics of epoxy nanocomposite*

The impact of nanoparticle size on nanocomposite properties is significant, as smaller nanoparticles improve interfacial bonding and mechanical, thermal, and optical properties due to larger surface area and superior matrix dispersion. Smaller nanoparticles enhance the mechanical characteristics of nanocomposites. Spherical nanoparticles similar in size to polymer monomers significantly enhance the toughness of polymer nanocomposites (PNCs) compared to their larger counterparts [85]. Smaller nanoparticles increase the interfacial area with the polymer matrix, thereby improving the tensile strength. This interaction strengthens the polymer network. The nanoparticle size influences the interphase properties between the particles and the polymer matrix. Smaller nanoparticles enhance the specific surface area, improving interfacial bonding and mechanical properties. However, aggregates or agglomerates may reduce the effectiveness of the interaction, highlighting the importance of particle dispersion and size [86]. The nanoparticle size affects the thermal efficiency of nanocomposites. Smaller nanoparticles enhance the glass transition temperature and storage modulus of epoxy-based nanocomposites, whereas larger particles may reduce these properties owing to less effective matrix interactions [87]. In optoelectronic and magneto-optic applications, the small nanoparticle size influences the optical and magnetic characteristics. Properties such as electrical conductivity and magnetic susceptibility can be improved through appropriate nanoparticle size selection [88].

Bondioli and Cannillo's research[89] investigates the synthesis and evaluation of epoxy–silica nanocomposites, focusing on silica nanoparticles' influence on mechanical and thermal properties. The sol–gel method was used to fabricate composites with well-dispersed silica nanoparticles, enhancing the filler-matrix compatibility. The study showed that silica incorporation improved the stiffness, tensile strength, and thermal stability of the epoxy resin, which was influenced by the particle size and dispersion quality. Smaller silica nanoparticles provided a greater surface area for epoxy interaction, facilitating stress transfer and reducing the risk of agglomeration. Micromechanical and finite element modelling predicted the elastic behaviour of the nanocomposites, with predictions matching the experimental results when the particle dispersion and interfacial bonding were accurately represented. This study highlights the importance of nanoparticle size and distribution in optimizing the performance of epoxy–silica nanocomposites, providing a framework for developing advanced materials for structural and thermal applications.

The shape of nanoparticles in polymeric nanocomposites significantly influences their properties and performance. The diversity of nanoparticle shapes, including spherical, cubic, needle-like, triangular, and rod forms, enables their application in device manufacturing, electronics, optics, and biofuel cells. Spherical nanoparticles, including silica nanoparticles, enhance the thermal, mechanical, physical, and chemical properties of polymer nanocomposites [62]. When uniformly distributed, they improve the overall performance of the composites. One-dimensional nanostructures, such as silver nanowires (AgNWs), exhibit a lower percolation threshold in conductive polymer nanocomposites than spherical silver nanoparticles (AgNPs)[90]. This variation is attributed to the elongated configuration of the nanowires, which promotes conductive networks at reduced filler concentrations. Rod- or tube-like nanoparticles, such as carbon nanotubes, have high aspect ratios. These materials enhance the mechanical strength, stiffness, and toughness through efficient load transfer. They improve the thermal and electrical conductivities at lower concentrations by creating efficient percolating networks, although they increase the viscosity [91]. Plate- or sheet-like nanoparticles, such as graphene and nanoclays, possess high surface areas and aspect ratios. These materials offer outstanding reinforcement and improve mechanical properties, barrier properties, and thermal and electrical conductivity. Similar to rod-like structures, they establish effective networks at low loadings while increasing the viscosity[92]. According to International Union of Pure and Applied Chemistry (IUPAC) definition a nanoparticle is a particle of any shape with dimensions in the 1–100 nm range [93]. Generally, 0D nanoparticles have an aspect ratio close to 1 (making them sphere-like), while 1D nanorods and 2D nanosheets have high aspect ratios significantly greater than 1, reflecting their elongated or plate-like shapes [94].Different dimensions and shapes of nanoparticles are presented in Table 3.

*Table 3.Dimensions and Shapes ofNanoparticles[95]*

| Category | Confined Dimensions | Examples of Shapes | Shapes |
|---|---|---|---|
| 0D (Zero-Dimensional) | All three dimensions (length, width, and thickness) are restricted to the nanoscale. | Spheres, Quantum Dots, Fullerenes, Nanoparticles (e.g., $CaCO_3$) | <br>Quantum dots<br><br>Fullerenes<br><br>Gold nanoparticles |
| 1D (One-Dimensional) | Two dimensions (e.g., width and thickness) are confined to the nanoscale, whereas one dimension (length) is extended. | Nanorods, Nanowires, Nanotubes (e.g., silver nanowires, Nanofibers | <br>Metal nanorods,<br>Ceramic crystals<br><br>Carbon nanotubes,<br>Metallic nanotubes<br><br>Gold nanowires,<br>Polymeric nanofibers,<br>Self assembled structures |
| 2D (Two-Dimensional) | One dimension (thickness) is confined to the nanoscale, whereas the other two dimensions (length and width) are extended. | Nanosheets, Nanoplates, Nanolayers (e.g., Graphene Oxide, Clay Nanosheets) | <br>Carbon coated<br>nanoplates<br><br>Graphene sheets<br><br>Layered nanomaterials |
| 3D (Three-Dimensional) | None of the dimensions are confined to the nanoscale; they are bulk materials with nanofeatures. | Bulk materials with nanoscale pores (e.g., mesoporous silica), Nanocrystalline materials (bulk materials made of nanoscale grains), Dendrimers | <br>Liposome<br><br>Polycrystalline<br><br>Dendrimer |

F. N. Ahmed et. al., [96]suggested that the morphology and dimensions of silica particles significantly influenced the properties of epoxy composites. Fig. 2 shows the micrographs depicting the various shapes of silica fillers.Particles with elongated shapes and high aspect ratios enhance mechanical properties owing to their increased surface areas. Particles smaller than 10 µm generally improve performance, whereas angular particles specifically, larger particle sizes (over 10 µm) are associated with reduced mechanical properties.This is likely due to stress concentration factors at the edges of the particles. A uniform distribution of particles enhances the properties, whereas agglomeration can weaken the composites. Elongated silica particles exhibit superior flexural and tensile properties. Although the particle shape had a lesser impact on the thermal properties, silica particles generally improved the thermal stability and reduced the coefficient of thermal expansion. Additionally, the particle shape affects the stress concentration and adhesion at the particle-matrix interface, thereby influencing the overall strength of the composite material.

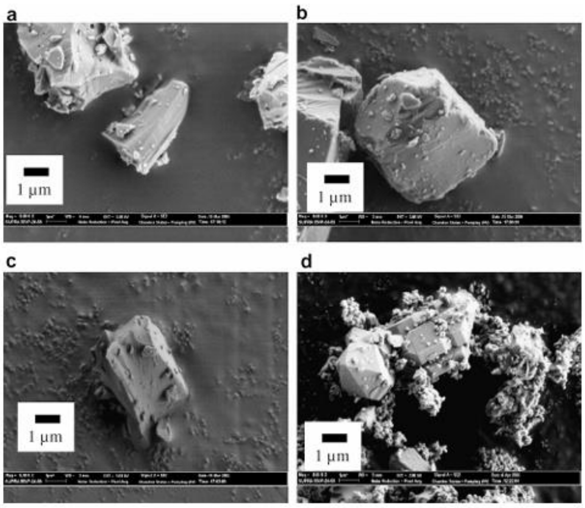


*Figure 2.SEM micrographs depicting various shapes of silica fillers: (a)angular-shaped silica mineral, (b)cubical silica mineral (c)elongated silica mineral, and (d) irregular-shaped fused silica [96]*

V. Hiremath and Shukla [97]reported that the morphology of nanoparticles plays a crucial role in determining the characteristics of epoxy-alumina nanocomposites. Rod-like alumina nanoparticles generally outperform spherical nanoparticles in enhancing the properties of composites. At a concentration of 1 wt%, nanorods increased the storage modulus by 37% compared to a 19% increase with spherical particles, and offered a slightly better enhancement in the flexural modulus (18% versus 17%).The shape of the nanorods allows for greater entanglement, making them more effective at restricting polymer chain movement. However, nanorods also resulted in a marginally lower glass transition temperature and formed larger agglomerates at higher concentrations (1.5 wt. %) than spherical particles. These results, as shown in the Fig. 3 and Fig. 4, indicate that rod-shaped nanoparticles typically provide superior reinforcement and property enhancement, especially at lower concentrations, before significant agglomeration occurs.

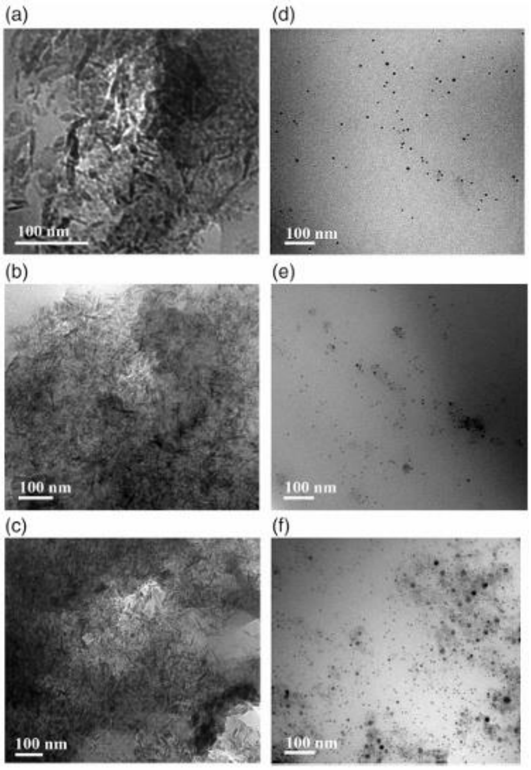


*Figure 3.TEM images of nanocomposites demonstrating the distribution of alumina nanorods at (a) 0.5%, (b) 1%, (c) 1.5%, along with spherical particles at (d) 0.5%, (e) 1%, and (f) 1.5%[97].*

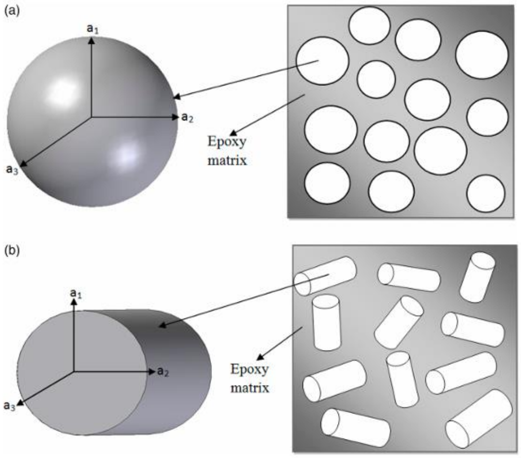


*Figure 4. Modelling a composite that includes (a) spherical alumina nanoparticles and (b) alumina nanorods [97]*

Numerous instances have been documented by researchers indicating that the size and shape of nanoparticles influence the overall properties of epoxy nanocomposites. Several significant studies are listed in Table 4.

*Table 4. Influence of the dimensions and morphology of nanoparticles on the characteristics of the composite*

| Mineral nanofilledEpoxy system | Findings with respect to shapes and size of nanoparticles | Ref. |
|---|---|---|
| Epoxy/nano-silica | The study shows that spherical silica nanoparticles of 8-50 nm size, with a narrow monomodal distribution averaging 16 nm in liquid resin, form core-shell structures where the interphase layer enhances stiffness, strength, and toughening in the cured nanocomposite | [98] |
| Epoxy/nano-silica | The nano-silica particles, with an average size of 20 nm and a similar inter-particle distance (around 20 nm for 4 wt.% modified epoxy), make the traditional argument of stress concentration promoting localized yielding doubtful, but crack deflection and crack pinning are considered important toughening mechanisms in this system | [99] |
| Epoxy/nano-silica | This study primarily focused on nanoscale silica particles with a size range of 10-20 nm. These nanoparticles exhibited high reactivity towards curing epoxy resins, whereas microscale silica particles (~ 5 μm) did not exhibit the same reactivity. | [100] |
| Epoxy/Talc Nanoparticles (TNPs)/ Urea-formaldehide microcapsules (UFMCs) | The flat structure of the talc nanoparticles after $NaNO_3$ loading influenced the coating properties, enabling effective inhibitor loading while maintaining barriers, pH-sensitive release, and compatibility with epoxy. | [101] |
| Epoxy/Nano-sized $CaCO_3$/commercial (micron-sized) $CaCO_3$ | Nano-sized $CaCO_3$ particles (39 nm) significantly outperform commercial (micron-sized) $CaCO_3$ in improving the mechanical and flame retarding properties of epoxy composites, with increases of up to 40% in Young's modulus, 158% in stress at break, 2867% in impact strength, and 50% in flame retardancy, due to their higher surface area and better exfoliation in the epoxy matrix | [102] |
| Epoxy/Silica/Mica/$CaCO_3$ - both mineral and precipitated forms | Comparing mineral $CaCO_3$ (0.73 μm) to precipitated $CaCO_3$ (40-80 nm), the study found that nano-$CaCO_3$ exhibited 65.7% higher tensile strength, slightly higher tensile modulus, and marginally better dielectric properties due to its increased surface area and enhanced interaction with the epoxy matrix | [103] |
| Epoxy/nano-$CaCO_3$ | Integrating $CaCO_3$ particles with an average size of 40 nm into epoxy resin significantly improved its mechanical characteristics, resulting in a 38.8% increase in tensile strength (from 55.057 to 76.421 MPa), a 20.0% enhancement in tensile modulus ( from 1374.3 to 1648.7 MPa), and a 65.1% increase in impact toughness ( from 10.2196 kJ/m2 to 16.8755 kJ/m$^2$). | [104] |
| Epoxy/nano-$CaCO_3$ | The in situ and inclusion polymerization methods achieved superior dispersion of nano-$CaCO_3$ particles (50-60 nm) compared to solution-blending (150-200 nm conglomerations), resulting in improved nanocomposite properties. | [105] |
| Epoxy/nano-$CaCO_3$ | Rod-like $CaCO_3$ nanoparticles generally exhibited better performance than cube-like particles did. For example, rod-like $CaCO_3$/epoxy composites (EP-rod-1.5) exhibited higher thermal stability with an IPDT of 533.6°C compared to 512.5°C for cube-like $CaCO_3$/epoxy composites (EP-cube-1.5). | [106] |
| Epoxy/Nano-clay | The sizes of the clay particles ranged from 1.3 to 10 μm. These materials are termed 'nanocomposites' because clay particles can exfoliate into nanoscale layers within the epoxy matrix. The organoclays exhibited increased interlayer spacing and partial exfoliation in the nanocomposites. | [107] |
| Epoxy/Nano- clay | The particle size and shape affect the fracture behaviour, with larger intercalated clay nanoplatelets (~450 nm) providing higher crack resistance than smaller, exfoliated ones (70-380 nm), whereas the platelet morphology influences the fracture toughness and surface roughness. | [108] |

### *4.2. Influence of Surface Modification of Mineral nanoparticles on properties of Epoxy nanocomposites*

The interaction at the interface between mineral nanoparticles and the epoxy matrix, along with the dispersibility of these particles, is crucial for influencing the performance of nanocomposites. A schematic representation of the nanoscale transition zone between the nanoparticles and polymer matrix, along with the nanoparticle interface, is shown in Fig. 5[109]. The modification of nanoparticle surfaces is crucial for enhancing the performance of nanocomposites by improving the interfacial compatibility between nanoparticles and the matrix. Unmodified nanoparticles often result in poor dispersion and weak interfacial bonding owing to their high surface energy and propensity to agglomerate, which can adversely affect the mechanical, thermal, and electrical properties of composites. Surface treatments, such as grafting functional groups, polymer coatings, or silanization, can be employed to tailor the surface chemistry of nanoparticles, thereby promoting better dispersion and stronger interactions with the host matrix [110].

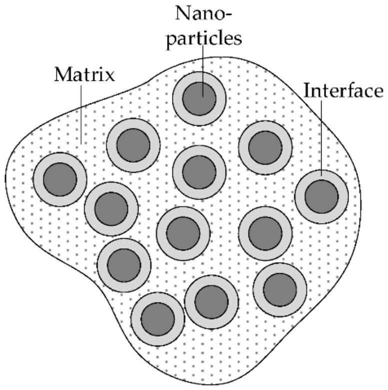


*Figure 5. Schematic diagram of the nanoparticle interface[109]*

Numerous methods exist for altering the surface characteristics of nanoparticles. The incorporation of coupling agents, such as silane, zirconate, and titanate, has been extensively investigated to modify the surface of nanoparticles, thereby enhancing the properties of epoxy composites. This modification enhances the dispersion within the epoxy matrix and improves the interfacial adhesion, resulting in superior mechanical properties, such as increased tensile strength and elongation at break [111]. An alternative approach is polymer grafting, which involves the attachment of polymer chains to the nanoparticle surface. This technique enhances the interfacial interactions between the filler and matrix, leading to improved curing, increased impact strength, and reduced wear rates of the composites [112]. The sol-gel process is an effective technique for preparing and modifying nanoparticles [113].

The incorporation of surface-modified nanoparticles with core-shell elastomers or reactive liquid rubbers constitutes another method for enhancing the toughness of epoxy composites without compromising other mechanical properties [114]. Grafting nanoparticles with specific functional groups facilitates the tailoring of their affinity for epoxy resins, thereby optimizing their dispersion and molecular-level interactions. This process ensures the uniform distribution of nanoparticles throughout the matrix, thereby enhancing its mechanical strength and thermal stability [115]. These surface modification techniques are pivotal in tailoring the properties of epoxy composites to meet specific application requirements, thereby broadening their potential use in various industries.The study of B. Feichtenschlageret. al.,[116]highlighted how surface modifications affect particle-matrix and particle-particle interactions. For zirconium dioxide ($ZrO_2$), diethylene glycol monomethyl ethers (DEG) surface modification led to the best dispersion, whereas epoxy end groups caused the largest aggregate formation. This is attributed to the balance between particle-matrix and particle-particle interactions.

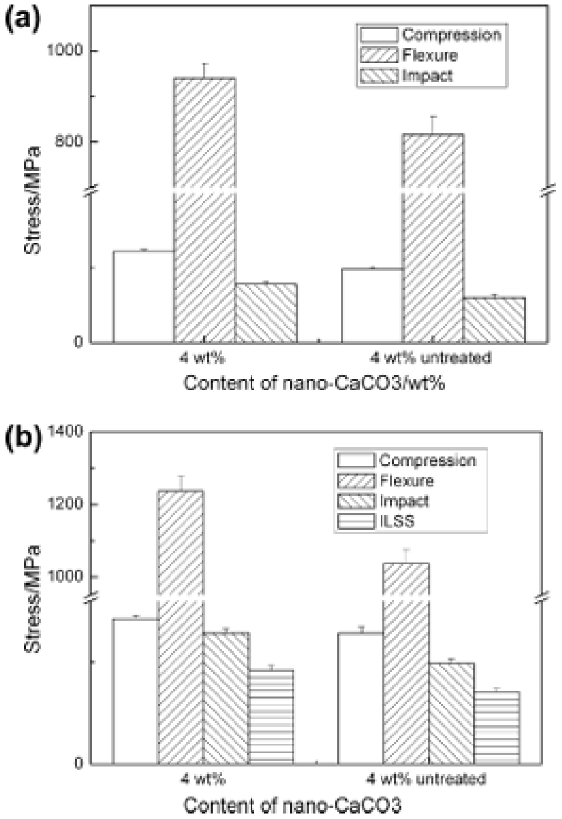


*Figure 6. Mechanical characteristics of epoxy cast and its fiber composites containing both untreated and treated nanoparticles ((a) epoxy cast; (b) epoxy/fiber composites) [117]*

H. He et. al.[117] proposed that modifying the surface of nano-$CaCO_3$ particles with a silane coupling agent (KH550) has a significant impact on the properties of composites. This study indicated that this surface modification enhanced the dispersion of nano-$CaCO_3$ particles in the epoxy matrix. The coupling agent creates a transitional layer between the fillers and polymer matrix, enhancing interfacial properties. This improved interface facilitated a more effective stress transfer between the nanoparticles and epoxy matrix. Nanoparticles that have undergone treatment exhibit a superior strengthening effect compared to those that are untreated, resulting in enhanced mechanical properties in both epoxy and epoxy/carbon-fiber composites.

As shown in Fig. 6 the treated 4 wt.% $CaCO_3$ nanoparticles show a better strengthening effect compared to untreated nanoparticles. Surface modification reduces defects at the filler-polymer interface, which is crucial at higher filler concentrations. Additionally, surface modification contributes to the increased thermal stability of the nanocomposites.

N.H.M. Zulfliet. al. [118] reported that enhanced interfacial adhesion and good dispersion of precipitated nano calcium carbonate (PNCC) are key factors in improving epoxy/GF/PNCC hybrid composites. Enhanced adhesion strengthens the bond between the epoxy and glass fibers, whereas good PNCC dispersion ensures uniform particle distribution. These factors synergistically improved the mechanical and thermal properties of the composites. Well-dispersed PNCC interacts with both epoxy and glass fibers, creating a cohesive structure that enhances flexural properties, fracture toughness, impact strength, and thermal stability

Additional studies are summarized in Table 5, which illustrates the method of surface modification for mineral nanofillers incorporated into epoxy nanocomposites. The impact on the nano-composite is also presented in column 3 of Table 5.

*Table 5. Impact of surface modification on mineral nanofillers integrated into epoxy nanocomposites.*

| Mineral nanofilledEpoxy system | Method of nanofillers surface modification | Remark with respect to filler surface modification | Ref. |
|---|---|---|---|
| Epoxy/Pristine clay/Carbon fiber | Clay was surface-modified using the 'Slurry Compounding' technique: clay dispersed in water, exchanged with acetone, treated with silanization of clay by 3-(aminopropyl)trimethoxysilane (APTMS), mixed with epoxy, and acetone removed | Silane modification of clay significantly improved carbon fiber-reinforced polymer (CFRP) properties.With 0.6 vol.% silanized clay/epoxy CFRP showing 46% higher storage modulus, 24% higher flexural modulus, 16% higher tensile modulus, and 14% higher fracture toughness compared to neat epoxy CFRP | [119] |
| Epoxy/Glass fiber/Montmorillonite nano-clay particles | The montmorillonite clay surface was modified through an onium-ion substitution reaction, in which surface sodium ions were replaced with quaternary onium ions to convert it from hydrophilic to organophilic in nature. | Surface modification of montmorillonite clay enabling better dispersion in epoxy and resulting in a 44% increase in interlaminar shear strength with 1% clay loading | [120] |
| Epoxy/nano-Talc/nano-MMT | Surface modification was achieved using amino-silane coupling agents (A1100 and A1170) added during the dispersion of nanoparticles in the epoxy resin. | At 0.5% concentration, A1100 decreased the water vapor permeability by 70% and increased the tensile strength by 45%. Adhesion also improved. | [121] |
| Epoxy/nano-$CaCO_3$ | Nano-sized calcium carbonate ($nCaCO_3$) was stearic acid treated with an acidic modification | Acidic modification of the $nCaCO_3$ creates a hydrophilic surface structure that improves adhesion between the coating and the metal substrate | [122] |
| Epoxy/nano-silica | Surface modification involves reacting the hydroxyl (OH) groups present on the surface of the silica particles with organosilanes | Surface treatment creates an amino-rich interphase, shifting the resin/hardener ratio to promote epoxy homopolymerization, resulting in higher crosslinking and the formation of a rubber-like shell owing to excess hardener. | [123] |
| Epoxy/nano-epoxy grafted silica(EGS) | The surface modification is achieved through a two-step process involving TDI-grafting | The improved compatibility, better dispersion, tighter interfaces, and ability to generate deep traps in EGS nanocomposites are direct results of this surface modification. | [124] |
| Epoxy/nano-silica | Surface modification of nano-silica involves OH group activation in ethanol at 80°C, APTES bonding through stirring, followed by centrifugation, washing and multi-step drying to prepare functionalized powder | Improved dispersion in epoxy nanocomposites increased the tensile elastic modulus by 77%, yield stress by 124%, and failure strain by 32% at 5 wt.%, with Izod impact resistance up 90% at 1 wt.%, while reducing curing activation energy from 55.79 kJ/mol to 52.993 kJ/mol compared to pure epoxy's 53.179 kJ/mol. | [125] |

| | | | |
|---|---|---|---|
| Epoxy/nano-silica | Mesoporous SBA-15 was modified by preheating at 200°C for 24 h, followed by mixing equal weights of SBA-15 and KH550 in acetone with agitation and ultrasonication, followed by washing with acetone and vacuum drying. | Surface modification with KH550 improves SBA-15/epoxy nanocomposite properties through enhanced particle dispersion , reducing pore diameter to 5.4 nanometers and surface area to 420 $m^2/g$; this creates a "bonded region" limiting epoxy chain movement, increasing glass transition temperature and breakdown strength while decreasing permittivity | [126] |
| Epoxy/nano-silica | 3-(Triethoxysilyl) propylamine (APTES) was used as a coupling agent in CCS synthesis, mixed with TEOS, epoxy resin (YD-128), and THF at 60°C for 2 h, and then with acidified water for 1 h. This process linked the silica particles to the epoxy matrix, thereby strengthening the organic-inorganic interface. | APTES, used as a coupling agent in the Coupled Composite System (CCS), significantly improved the epoxy nanocomposites by enhancing silica particle dispersion, increasing glass transition temperature by 4°C, and boosting thermal stability, with the Initial Decomposition Temperature (IDT) rising from 243.71°C to 281.60°C at 20 wt.% silica loading compared to the Uncoupled Composite System (UCS). | [127] |
| Epoxy/nano-clay $Cu^{2+}$-MMT | The surface modification of montmorillonite with $Cu^{2+}$ ions, achieved through cation exchange reaction using cupric sulfate pentahydrate, resulted in a $Cu^{2+}$-MMT containing 3.5% wt. of copper | $Cu^{2+}$-MMT/Epoxy nanocomposite imparted significant antibacterial properties to the nanocomposites, with inhibition rates exceeding 96% against E. coli and 99.99% against S. aureus for 3% wt. $Cu^{2+}$-MMT nanocomposites | [128] |

### *4.3. Influence of Mineral nanoparticles loading on properties of epoxy nanocomposites*

Numerous researchers have reported that the loading ratio of nanofillers significantly influences nanocomposite properties. For example, the Young's modulus, tensile stress, and yield stress of epoxy nanocomposites generally exhibit a linear increase with the addition of rigid nanoparticles such as silica [129]. Lower ZnO nanoparticle loadings increased the surface roughness of the nanocomposite. Conversely, a reduction in surface roughness can be achieved at higher nanoparticle contents due to their tendency to remain within the bulk of the coating[130].

R. Jain et. al.,[131] explored how varying amounts of nano-$CaCO_3$ filler affect the properties of epoxy composites. Their findings revealed that adding nano-$CaCO_3$ considerably boosted the mechanical, thermal, and dynamic mechanical characteristics of epoxy matrix. The study identified that the most effective filler concentration for enhancing these properties was approximately 4% (w/w) for both. At this level, the composites exhibited the greatest improvements in the flexural modulus, flexural strength, and impact strength. An increase in the filler content led to a notable increase in the glass transition temperature, suggesting better dimensional stability and a higher maximum-use temperature. Although there was a minor reduction in the initial decomposition temperature, higher filler content resulted in a greater char yield at high temperatures. Additionally, as shown in the Fig. 7, the study noted a transformation in the crystal structure of $CaCO_3$ from calcite to dolomite when it was integrated into the epoxy matrix.In summary, this study concludes that nano-$CaCO_3$ effectively strengthens the epoxy matrix, enhancing its stiffness and limiting polymer chain movement, with the best results observed at a 4 wt. % filler concentration.

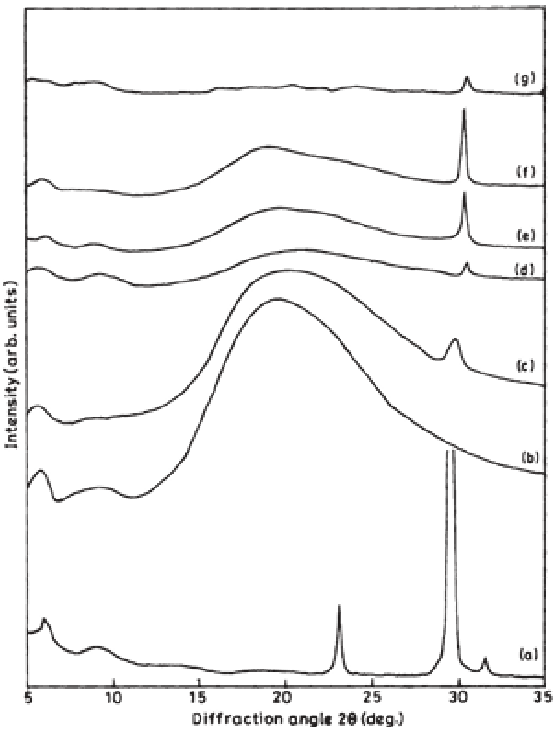


*Figure 7.X-ray diffraction patterns of $CaCO_3$, pure epoxy, and epoxy containing different quantities of calcium carbonate, where plot (a) nano-$CaCO_3$,plot (b) pure epoxy, plot (c) epoxy composite containing 2 wt% $CaCO_3$, plot (d) epoxy composite containing 4 wt% $CaCO_3$, plot (e) epoxy composite containing 6 wt% $CaCO_3$,, plot (f) epoxy composite containing 8 wt% $CaCO_3$ and plot (g) epoxy composite containing 10 wt% $CaCO_3$[131]*

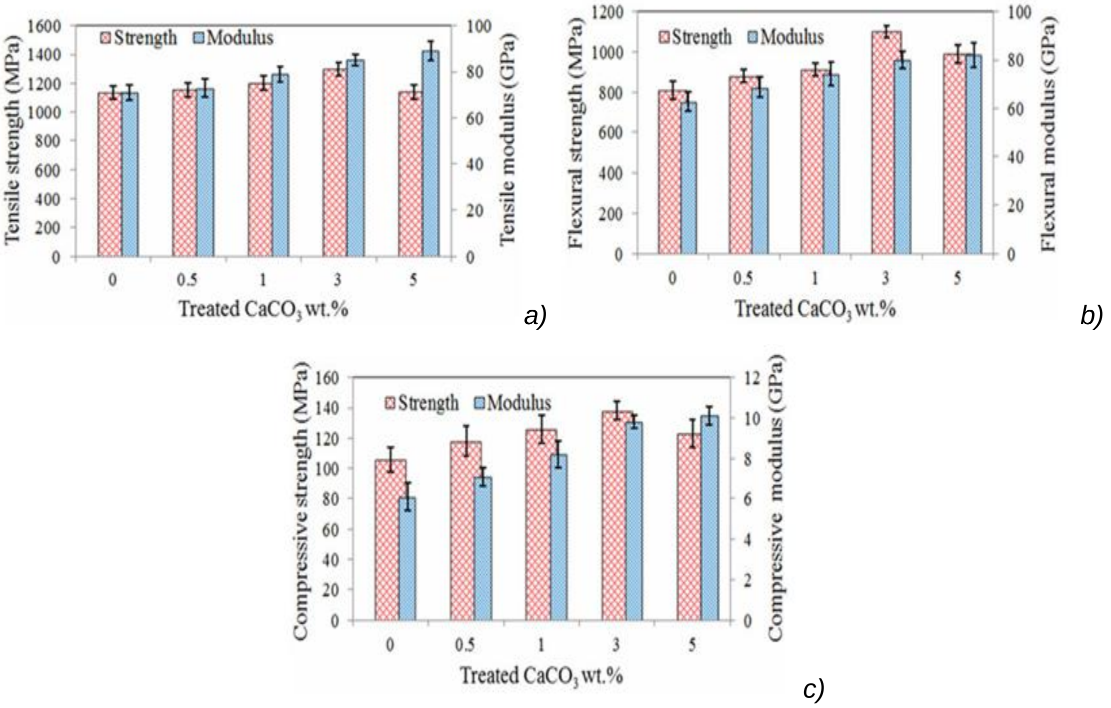


*Figure 8. (a) The change in tensile strength and modulus in relation to treated $CaCO_3$ loading, (b) The change in flexural strength and modulus in relation to treated $CaCO_3$ loading, (c) The change in compressive strength and modulus in relation to treated $CaCO_3$loading[132]*

Shahbakhsh, Tohidlou, Khosravi investigated the effects of silanized calcium carbonate ($CaCO_3$) nanoparticles on the mechanical properties of carbon fiber/epoxy composites. The authors found that adding 5 wt. % of treated $CaCO_3$ increased the tensile, flexural, and compressive moduli by 26%, 31%, and 65% respectively, compared to the neat composite. The optimal loading was 3 wt. % $CaCO_3$ led to improvements of 14%, 36%, and 30% in tensile, flexural, and compressive strengths, respectively (Fig 8). However, higher loadings (5 wt. %) decreased mechanical strengths due to nanoparticle agglomeration. The researchers also observed enhanced carbon fiber-matrix adhesion in $CaCO_3$-filled composites through SEM analysis, which contributed to the improved mechanical properties[132].

Numerous references cited by researchers in their publications indicate that the loading concentrations of mineral nanofillers have a significant impact not only on the mechanical and thermal properties of epoxy nanocomposites but also on various other properties, which are thoroughly detailed in Table 6.

*Table 6.Detailed summary of the impact of mineral filler loading on the characteristics of epoxy nanocomposites*

| Mineral nanofilled epoxy system | Amount of nanofillers added | Effect of different loading concentrations of nanoparticles on properties of nanocomposites. | Ref |
|---|---|---|---|
| Epoxy/nano-Talc/nano-MMT | 0.5, 1.0, and 1.5 phr. | Nano-talc outperformed nano-MMT in solventless epoxy coatings, with 0.5% talc coupled with primary amine-based silane reducing water vapour permeability by 14% and increasing tensile strength by 45% compared to the pristine polymer | [121] |
| Epoxy/nano spherical SiO2 (silica) | 1 wt.% to 8 wt.% | 4 wt.% SiO2 nanoparticles provided optimal mechanical properties, showing 44% higher tensile strength, 58% higher Young's modulus, and 144% increased toughness; however, higher concentrations caused degradation due to agglomeration. | [133] |
| Epoxy/nano-Silica | 1, 3, 5, and 8 wt.% | Fracture toughness and elastic modulus generally increased up to 8% weight fraction of silica nanoparticles for post-cured specimens, while for non-post-cured specimens, fracture toughness peaked at 5% before decreasing | [134] |

| | | | |
|---|---|---|---|
| Epoxy/nano-clay (N-C)/nano-calcium carbonate (N-CC) | 1, 3, and 5 wt.% | Adding 5 wt.% N-C and N-CC to epoxy increased the density (1.74%, 1.82%) and hardness (14.9%, 12.5%), decreased the tensile strength (22.2%, 33.3%), affected the tensile modulus (+23.5% for N-C), and improved the impact strength (33.33% for 3 wt.% N-C, 62% for 5 wt.% N-CC). | [135] |
| Epoxy/flyash/nano-$CaCO_3$ | 0, 1, 3, and 5 wt.% | Finding optimal performance at different concentrations: 5 wt.% maximized the tensile strength (52.41% increase), while 3 wt.% optimized the impact and flexural strengths (43.24% and 42.36% improvements, respectively), and also showed enhanced thermal stability with higher char yields at 750 °C. | [136] |
| Epoxy/nano-$CaCO_3$ | 1 wt.%, 2 wt.%, and 3 wt.% | 2 wt.% $CaCO_3$ nanoparticle loading provided optimal improvements in mechanical properties, with tensile strength of laminated nanocomposites increasing by 48.4%, flexural strength improving by 48%, and impact threshold forces showing enhancements of 16.8%, 13.4%, and 11.3% at impact velocities of 2 m/s, 2.5 m/s, and 3 m/s, respectively | [137] |
| Epoxy/nano-$CaCO_3$ | 0, 2, 4, 6 and 8 wt.% | Nano-$CaCO_3$ content accelerated the curing reaction and maintained a stable Tg up to 6 wt.%; however, it decreased from 240°C to 232°C at 8 wt.%. The crosslinking density increased linearly, while the thermal stability improved with the IDT rising from2 279°C to 309°C at 8 wt.%. The CTE in the rubbery region decreased from 408 × $10^{-5}$/°C to 264 × $10^{-5}$/°C at 8 wt.% nano-$CaCO_3$. | [138] |
| Epoxy/nano-$CaCO_3$ | 1, 2.5, and 5 wt.% | 2.5 wt.% $CaCO_3$ nanoparticles in epoxy resin enhanced properties, increasing glass transition temperature by 12°C, Young's modulus by 59%, hardness by 38%, and water contact angle from 89° to 97°, while avoiding agglomeration issues at higher loadings | [139] |
| Epoxy/nano-silica | 0.1, 0.3 and 0.5 wt.% | Nano-silica loading affects epoxy properties: permittivity and dielectric loss reached minimum at 0.3wt.% before increasing, with tan delta below unfilled epoxy up to 0.3% but higher above 1.0%; DC resistivity decreased, with unfilled epoxy at $6.5x10^{14}$ Ohm-meter; glass transition temperature changed minimally from 111.5°C to 115.2°C at 5.0wt.% | [140] |
| Epoxy/nano-silica | 0 to 15 wt.% | The study examined silica concentrations up to 15 wt.%; however, it was found that 10 wt.% was the highest concentration that could be added without agglomerate formation. Increasing silica content led to improvements in modulus, yield strength, and fracture toughness | [141] |
| Epoxy/nano-silica | 0, 1, 3, 6, 7, 10, 13, and 15 vol.% | The study found that increasing the silica nanoparticle content in epoxy composites from 0 to 15 vol. % improved mechanical properties (hardness increased from 0.289 GPa to 0.438 GPa), wear resistance (wear volume decreased by 6 times under 80 µN load), and the threshold for wear mechanism transition, showing enhanced performance. | [142] |
| Epoxy/nano-clay/GF | 1, 2, 3, 5, and 7 wt.% | The incorporation of 3 wt.% nanocomposites (NC) enhanced the tensile properties, increasing the elastic modulus, tensile strength by 11 %, and bending strength by 15 %, 11 %, and 11%, respectively. At 5 wt.% NC, the flexural modulus improved by 48%, while 5-7 wt.% NC increased the ballistic speed limit from 116 m/s to 130 m/s. | [143] |
| Epoxy/nano-clay organophilic montmorillonite (OMMT) | 1, 3, and 5 wt.% | Increasing OMMT clay concentration from 1 to 5 wt.% in the 10% BMEPC-DGEBA system improved tensile strength (13% to 22%) and flexural strength (24% to 34%), enhanced thermal stability and char yield, and increased dielectric constant (ε') and dielectric loss (ε'') in both DGEBA and TGDDM systems | [144] |
| Epoxy/nano-clay Taurine-modified MgAl layered double hydroxide (T-LDH) | 6, 9, 12, 15, and 18 wt.%. | The study shows that T-LDH loading affects epoxy nanocomposite properties: Tg increases to 15 wt% then drops; RAF reaches 40 wt.% at 18 wt%; cooperativity length decreases from 0.39 nm to 0.26 nm (>9 wt.%); α-relaxation shifts higher; and T-LDH interlayer distance varies with concentration. | [145] |

## 5. CONCLUSIONS

The dimensions and morphology of mineral nanofillers significantly influence the properties of epoxy nanocomposites. Smaller particles enhance the properties of the material owing to the increased surface area and better distribution. The surface modification of nanofillers through coupling agents, polymer grafting, and functionalization improves their compatibility with epoxy matrices, leading to better dispersion and stronger interfacial interactions. The content of mineral nanofillers affects the composite characteristics, with an optimal loading level beyond which the properties may deteriorate owing to agglomeration. Various nanofillers, such as nano-silica, nano-clay, nano-calcium carbonate, and nano-talc, provide distinct property enhancements, enabling customization for specific applications. The improved properties make these composites suitable for aerospace, automotive, electronics, coating, and structural applications. Despite advances in understanding mineral nanofillers in epoxy composites, further research is required on surface modification techniques, the synergistic effects of multiple nanofillers, and large-scale processing methods. This review demonstrates that, through careful consideration of nanofiller morphology, surface modification, and loading, advanced materials with customized properties can be engineered for specific applications, enabling the development of high-performance, cost-effective composite materials.

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## IZVOD

### POBOLJŠANJE EPOKSIDNIH NANOKOMPOZITA MINERALNIM NANOPUNILIMA:SVEOBUHVATNI PREGLED VELIČINE, OBLIKA I KONCENTRACIJE ČESTICA

*Mineralna nanopunila su se pojavila kao obećavajuća pojačanja za epoksidne nanokompozite, nudeći poboljšana mehanička, termička i fizička svojstva potonjih. Ovaj pregled sveobuhvatno istražuje uticaj morfologije mineralnog nanopunila, modifikacije površine i opterećenja na performanse epoksidnih nanokompozita. Veličina i oblik nanočestica značajno utiču na međupovršinske interakcije i disperziju unutar epoksidne matrice, pri čemu manje čestice i veći odnosi širine i visine generalno dovode do poboljšanih svojstava. Tehnike modifikacije površine, kao što su upotreba sredstava za vezivanje, kalemljenje polimera i funkcionalizacija, ključne su za optimizaciju kompatibilnosti i vezivanja između nanopunila i epoksidne matrice. Koncentracija opterećenja mineralnih nanopunila igra ključnu ulogu u određivanju konačnih svojstava nanokompozita, pri čemu optimalna opterećenja variraju u zavisnosti od specifičnog nanopunila i željenih poboljšanja. Ključna mineralna nanopunila, uključujući nano-silicijum dioksid, nano-glinu, nano-kalcijum karbonat (CaCO3) i nano-talk, pokazala su značajna poboljšanja u mehaničkoj čvrstoći, termičkoj stabilnosti i barijernim svojstvima kada se ugrade u epoksidnu matricu. Ovaj pregled pruža vredne uvide u složenu interakciju između karakteristika nanopunila i performansi epoksidnih nanokompozita, vodeći dizajn i razvoj naprednih materijala za različite primene.*

***Ključne reči:*** *Epoksidni kompoziti, mineralna nanopunila, veličina čestica, oblik čestica, morfologija, modifikacija površine, punjenje punila, nano-silicijum dioksid, nano-glina, nano-CaCO*$_3$*, nano-talk*

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