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Self-Healing Polymeric Composites Reinforced with Green Synthesized Nanoparticles

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Abstract

The development of sustainable self-healing polymeric materials is critical for enhancing durability and service life in advanced engineering applications. In this study, zinc oxide (ZnO), magnesium oxide (MgO), and ferric oxide (Fe_2O_3) nanoparticles were green-synthesized using Allium cepa extract and incorporated into epoxy-based polymeric composites to improve mechanical, thermal, and self-healing properties. This bio-based synthesis is eco-friendly and safe alternative to harsh chemical techniques, utilizing natural antioxidants to stabilize the metal ions without toxic byproducts generation. The synthesized nanoparticles were characterized using FT-IR, XRD, SEM, and TGA characterization techniques, confirming successful formation, nanoscale crystallinity, microstructure and effective dispersion within the polymer matrix. SEM of the MgO nanoparticles revealed an irregular, flake-like morphology with significant agglomeration. In contrast, the ZnO nanoparticles exhibited a spherical morphology, while the Fe_2O_3 nanoparticles displayed a rough, granular structure. Thermal analysis revealed a significant improvement in thermal stability, with MgO-reinforced composites exhibiting the highest resistance to thermal degradation. Mechanical testing demonstrated enhanced tensile strength for all nanocomposites, reaching a maximum of 50 MPa for MgO-filled composites compared to 30 MPa for the neat polymer. Self-healing efficiency studies showed superior recovery behavior, with MgO composites achieving 85% healing efficiency under ambient conditions, while ZnO-based composites exhibited exceptional performance under UV exposure due to their photocatalytic activity, exceeding 100% healing efficiency. The results highlight the synergistic effect of green-synthesized metal oxide nanoparticles in improving composite performance. This work demonstrates an eco-friendly and effective approach for developing high-performance self-healing polymeric materials suitable for aerospace, automotive, biomedical, and protective applications.

Keywords: Self-healing polymeric composites, green synthesis, metal oxide nanoparticles, allium cepa extract, UV-assisted self-healing

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INTRODUCTION

Polymeric materials are widely used in aerospace, automotive, biomedical, and protective applications because of their low density, corrosion resistance, and ease of processing. However, conventional polymers are vulnerable to microcrack formation, fatigue, and environmental degradation, which significantly reduce their service life and reliability. Self-healing polymeric composites have therefore emerged as an effective strategy to autonomously repair damage, restore mechanical integrity, and reduce maintenance costs. The incorporation of nanofillers into polymer matrices is a proven approach to improve mechanical strength, thermal stability, and healing efficiency [1–4]. Common polymer matrices used in self-healing composites include epoxy resins, polyurethane, polyethylene or polypropylene etc. Metal oxide nanoparticles such as magnesium oxide (MgO), zinc oxide (ZnO), and ferric oxide (Fe_2O_3) are particularly attractive due to their high surface area and strong interfacial interactions. MgO enhances mechanical reinforcement and thermal resistance, ZnO provides ultraviolet absorption and photocatalytic activity beneficial for UV-assisted healing, and Fe_2O_3 improves stiffness, thermal stability, and abrasion resistance [5–6].

Traditional nanoparticle synthesis methods often involve toxic reagents and high energy consumption, raising environmental concerns. Green synthesis offers a sustainable alternative by using biological resources as reducing and stabilizing agents [7]. Plant-mediated synthesis using Allium cepa extract is especially effective due to its rich content of flavonoids and polyphenols. In this study, green-synthesized ZnO, MgO, and Fe_2O_3 nanoparticles were evaluated in composites [8–9].

The development of self-healing polymeric materials has attracted significant research attention due to their ability to autonomously repair damage and extend material lifespan. The summary of literature review is as follows:

One of the earliest and most influential studies was reported by White et al., [10] who demonstrated autonomic healing in polymer composites using microencapsulated healing agents, successfully restoring mechanical properties after crack formation. This work established the fundamental concept of self-healing polymers and inspired extensive research into smart composite materials. Wool and O'Connor [11] further contributed to this field by proposing theoretical models for crack healing based on polymer chain diffusion and reversible bonding mechanisms, providing a scientific basis for intrinsic healing systems.

Subsequent research highlighted the role of nanofillers in enhancing self-healing efficiency. Thakur and Kessler [12] reported that polymer nanocomposites exhibit superior healing behavior compared to neat polymers due to improved interfacial bonding and stress transfer [13]. Sajid [14] emphasized that the unique physicochemical properties of nanoparticles, such as high surface-area-to-volume ratio and nanoscale effects, significantly improve mechanical reinforcement and multifunctionality in polymer matrices. The synthesis method of nanoparticles was also found to be crucial; Baig et al. [15] and Ijaz et al. [16] concluded that green synthesis routes are environmentally benign and result in better nanoparticle dispersion and stability.

Plant-mediated green synthesis has emerged as an effective and sustainable approach. Álvarez-Chimal and Arenas-Alatorre [17] demonstrated that plant extracts produce stable metal oxide nanoparticles with controlled size and morphology. Metal oxide nanoparticles such as MgO, ZnO, and Fe_2O_3 have shown remarkable improvements in composite performance. Sierra-Fernandez et al. [18] reported enhanced thermal stability and interfacial adhesion in MgO- and ZnO-filled composites, while Kasinathan et al. [19] highlighted the UV-responsive and photocatalytic behavior of ZnO/MgO nanocomposites. Fe_2O_3 nanoparticles were reported by Santoshi et al. [20] to improve stiffness and thermal resistance. Recent studies by Mollajavadi et al. [21] confirmed that nanomaterial-reinforced self-healing polymers exhibit superior mechanical strength, thermal stability, and long-term durability, highlighting their potential for advanced and sustainable engineering applications.

Existing studies clearly demonstrate that self-healing polymer composites benefit significantly from nanofiller incorporation and green synthesis approaches; however, most investigations focus on single-property enhancement rather than multifunctional performance. Although metal oxide nanoparticles such as ZnO, MgO, and Fe_2O_3 have shown promise individually, comparative and synergistic studies within the same polymer system remain limited. Furthermore, the mechanisms governing nanoparticle-assisted intrinsic and UV-triggered self-healing are not yet fully understood, particularly at the filler–matrix interface. Importantly, there is a lack of systematic studies integrating green-synthesized

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nanoparticles with long-term durability, scalability, and real-service condition performance, highlighting a critical gap addressed in the present work.

MATERIALS AND METHODS

Epoxy resin and the corresponding curing agent were used as the polymer matrix for the preparation of self-healing composites. Zinc oxide (ZnO), magnesium oxide (MgO), and ferric oxide (Fe_2O_3) nanoparticles were synthesized via a green route using Allium cepa (onion) extract as a natural reducing and stabilizing agent [22]. Fresh onion extract was prepared by aqueous extraction, followed by controlled reaction with respective metal salt precursors under constant stirring and mild heating. The obtained nanoparticles were washed, dried, and calcined to improve crystallinity. Structural and morphological characteristics of the synthesized nanoparticles were analyzed using Fourier transform infrared spectroscopy (FT-IR), X-ray diffraction (XRD), scanning electron microscopy (SEM), and thermogravimetric analysis (TGA).

For composite fabrication, the green-synthesized nanoparticles were dispersed in the epoxy resin at optimized weight fractions using mechanical stirring followed by ultrasonication to ensure uniform distribution [23]. The curing agent was then added, and the mixture was cast into standardized molds and cured at room temperature followed by post-curing at elevated temperature. Mechanical properties were evaluated using tensile testing according to ASTM standards, while thermal stability was assessed through TGA. Self-healing behavior was investigated by introducing controlled microcracks and evaluating recovery efficiency under ambient and UV-assisted conditions. Fracture surface morphology after healing was examined using SEM to correlate microstructural features with healing performance.

Conventional polymeric materials widely used in engineering applications are highly susceptible to microcrack formation, mechanical fatigue, and environmental degradation, leading to premature failure and increased maintenance costs. Although self-healing polymers have emerged as a promising solution, many existing systems rely on complex chemistries, toxic synthesis routes, or exhibit limited healing efficiency and poor long-term stability. Moreover, the role of eco-friendly, green-synthesized metal oxide nanoparticles in enhancing both intrinsic and stimulus-assisted self-healing mechanisms remains insufficiently explored. In particular, comparative understanding of ZnO, MgO, and Fe_2O_3 nanoparticles within a single polymer matrix, along with their influence on mechanical strength, thermal stability, and healing efficiency, is lacking. Additionally, limited attention has been given to developing sustainable self-healing composites that combine environmental safety with multifunctional performance. Therefore, there is a clear need to develop and systematically investigate green-synthesized metal oxide nanoparticle–reinforced polymer composites to achieve improved durability, thermal resistance, and efficient self-healing suitable for advanced engineering applications. The composition & proportion for each metal oxide nanoparticle–reinforced polymer composites were shown in Table 1.

Self-healing polymeric composite sheets were fabricated by first functionalizing ZnO, MgO, or Fe_2O_3 nanoparticles (5 g) through dispersion in acetone or ethanol (25 mL) using ultrasonication for 30 minutes, followed by drying at 60–80°C to remove residual solvents. Epoxy resin (30 g) was preheated to 40–60°C and mixed with 7.5 g of hardener for 5–10 minutes to obtain a homogeneous matrix. The treated nanoparticles were then gradually incorporated into the epoxy system and dispersed using mechanical stirring or ultrasonication for 30–60 minutes to ensure uniform distribution. The resulting mixture was cast into molds of dimensions 50 mm × 50 mm × 2 mm and subjected to vacuum degassing for 5 minutes to eliminate entrapped air (Figure 1) [24]. Finally, the composites were cured either at room temperature for 24 hours or via thermal curing at 80°C for 2 hours followed by 120°C for 1 hour to improve mechanical strength and durability [25–27].

Figure 1. Pure resin, Fe_2O_3, Zno and Mgo polymeric composite sheets.

Table 1. Composition & proportion for nanoparticle–reinforced polymer composites.

Component	ZnO composite (g)	MgO composite (g)	Fe_2O_3 composite (g)
Epoxy Resin (Bisphenol-A epoxy)	30g	30g	30g
Harderner (Amine-based or Anhydride-based)	7.5g	7.5g	7.5g
Zinc oxide (ZnO)	5g	-	-
Magnesium oxide (MgO)	-	5g	-
Ferric oxide (Fe_2O_3)	-	-	5g
Plasticizer (PEG,PVA)	1.5g	1.5g	1.5g
Crosslinking agents (Diethylenetriamine, dicyandiamide)	1g	1g	1g

RESULT AND DISCUSSIONS

The green-synthesized ZnO, MgO, and Fe_2O_3 nanoparticles were successfully incorporated into the epoxy matrix, and their influence on the structural, thermal, mechanical, and self-healing behavior of the composites was systematically evaluated. FT-IR spectra confirmed the successful green synthesis of MgO, ZnO, and Fe_2O_3 nanoparticles and their effective interaction with bio-organic compounds from Allium cepa (Figure 2). All samples showed broad O–H stretching bands (≈3400 cm^{-1}), indicating surface hydroxyl groups and moisture adsorption, which are beneficial for interfacial bonding within the polymer matrix. Characteristic metal–oxygen vibrations were clearly observed at 667–418 cm^{-1} for Mg–O, ≈490 cm^{-1} for Zn–O, and 588–425 cm^{-1} for Fe–O, confirming the formation of respective oxide nanoparticles. The presence of minor C–O and C=O peaks suggest phytochemical capping, which improves nanoparticle stability and dispersion within the polymer network.

SEM micrographs revealed nanoscale morphology with varying degrees of agglomeration. ZnO nanoparticles (20–50 nm) showed near-spherical morphology with minimal clustering, promoting uniform stress transfer (Figure 3). MgO nanoparticles (30–60 nm) exhibited flake-like structures with rough surfaces, enhancing interfacial adhesion. Fe_2O_3 nanoparticles (40–80 nm) showed higher agglomeration, which likely reduced their reinforcement efficiency.

XRD patterns demonstrated the crystalline nature of all nanoparticles (Figure 4). MgO showed sharp peaks at $2\theta \approx 37°$, 43°, 63°, and 78°, confirming a cubic crystalline structure with nanoscale crystallite size. ZnO exhibited distinct reflections corresponding to a hexagonal wurtzite structure, with no impurity peaks, indicating high phase purity. Fe_2O_3 showed comparatively broader peaks, suggesting smaller crystallite size and partial amorphous character. This structural difference explains the relatively lower mechanical and healing performance of Fe_2O_3-based composites compared to MgO and ZnO systems.

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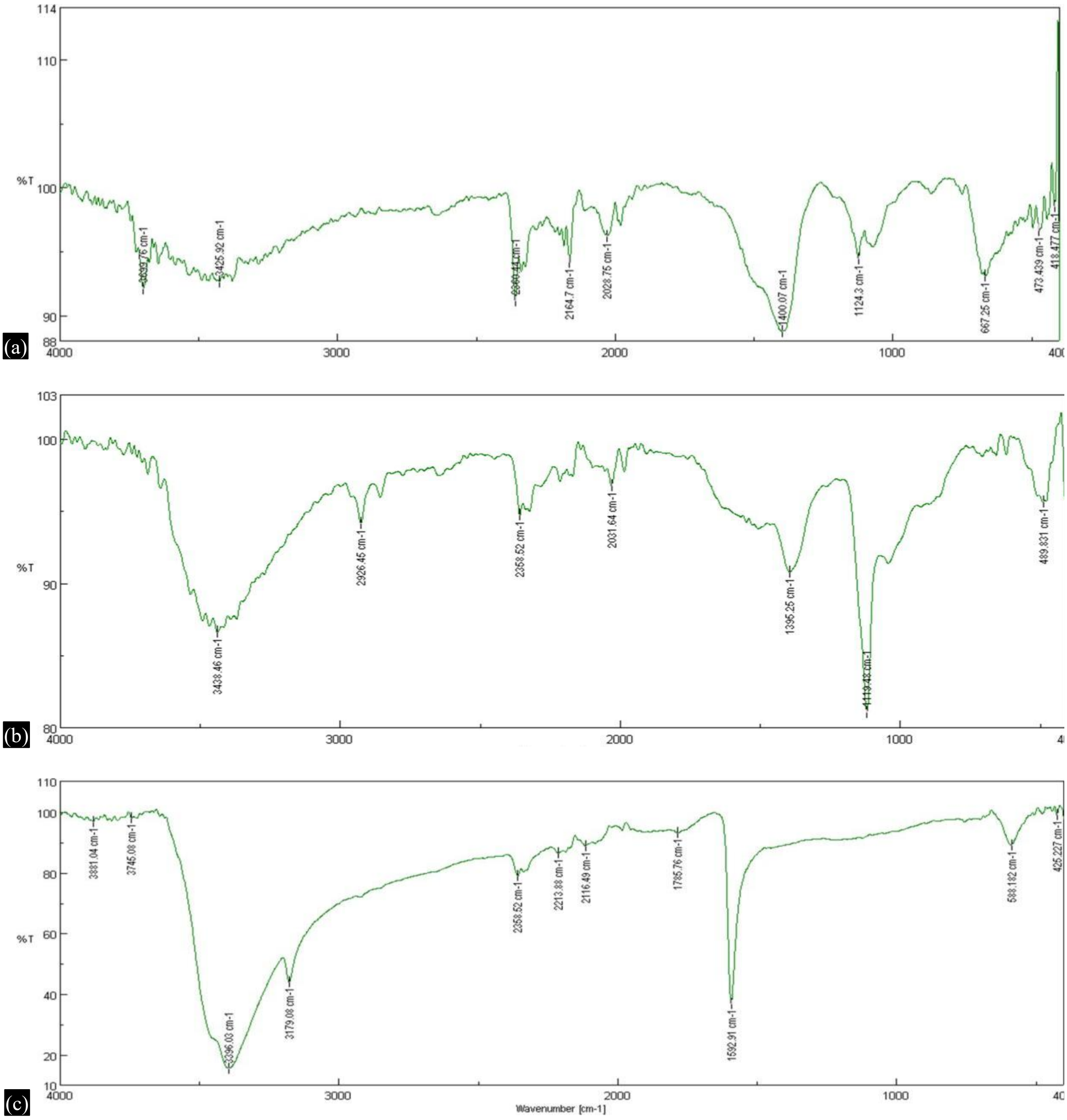

Figure 2. FTIR Spectrum Of A) Mgo B) Zno C) Fe_2O_3.

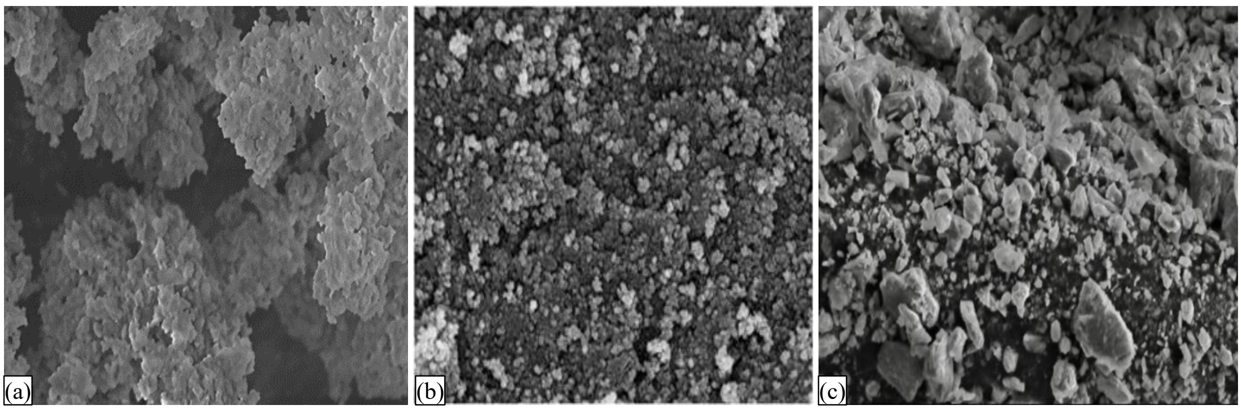

Figure 3. SEM Images of (a) Mgo; (b) Zno; (c) Fe_2O_3.

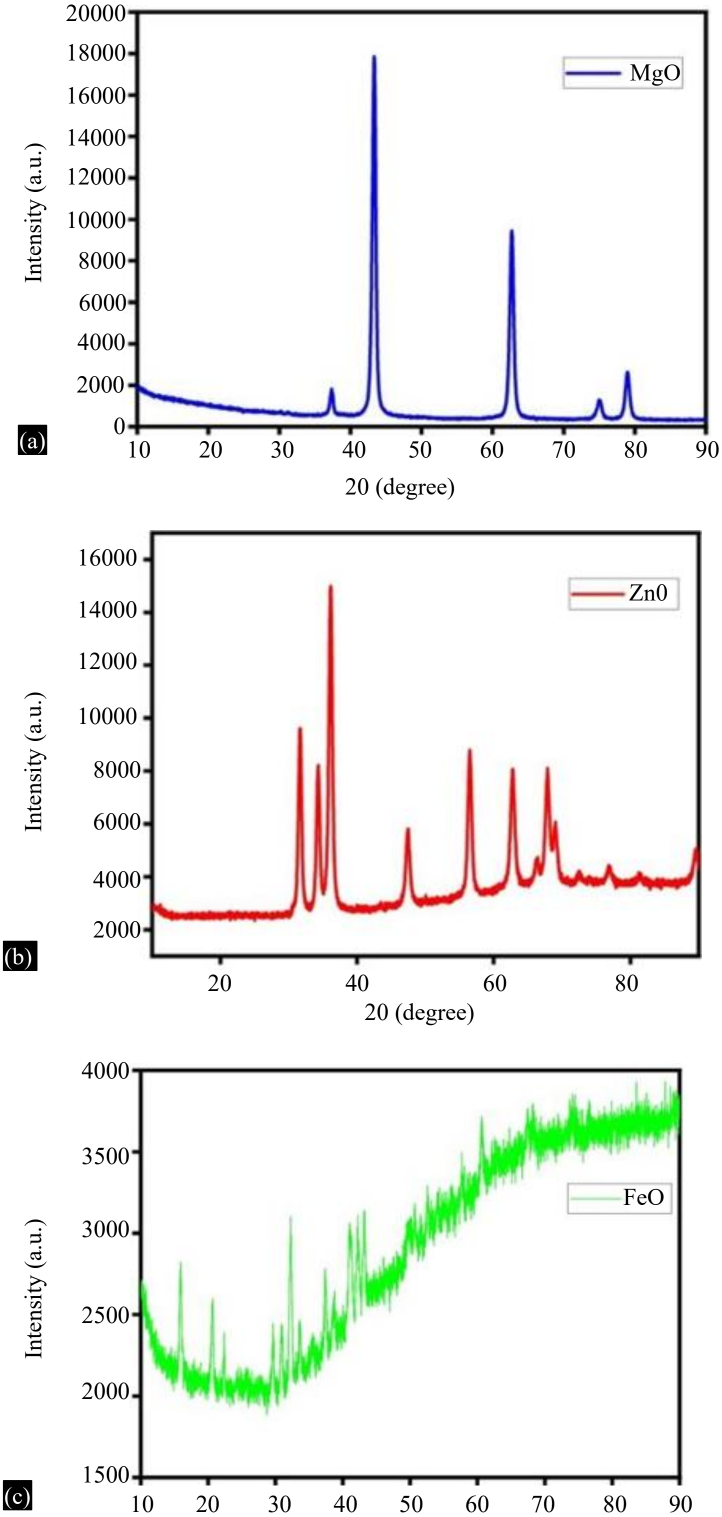

Figure 4. XRD patterns of Mgo, Zno and Fe_2O_3

Thermal analysis demonstrated a notable improvement in thermal stability for all nanoparticle-reinforced composites compared to the neat epoxy (Figure 5). Among the fillers, MgO-based composites showed the highest thermal resistance, attributed to the inherent thermal stability of MgO and its strong interfacial bonding with the polymer matrix. Mechanical testing results indicated a

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significant enhancement in tensile strength and modulus upon nanoparticle addition, with MgO-reinforced composites exhibiting the highest tensile strength, followed by ZnO and Fe_2O_3 composites (Figure 6). The improvement is primarily associated with efficient load transfer and restriction of polymer chain mobility by well-dispersed nanoparticles.

Self-healing studies revealed that the incorporation of metal oxide nanoparticles substantially enhanced healing efficiency (Figure 7). Under normal conditions, self-healing efficiency increased from 30% for the neat polymer to 60% (Fe_2O_3), 75% (ZnO), and 85% (MgO). The enhanced healing in MgO composites is associated with improved interfacial bonding and increased chain mobility at the damaged interface. Under UV irradiation, ZnO composites exhibited exceptional healing efficiency of 105%, surpassing their original strength due to ZnO's photocatalytic activity. MgO and Fe_2O_3 composites showed healing efficiencies of 89% and 66%, respectively, while the neat polymer remained largely unaffected (32%). ZnO-based composites exhibited superior UV-assisted self-healing behavior due to the photocatalytic activity of ZnO, enabling faster crack closure and higher recovery efficiency. MgO-filled composites demonstrated excellent intrinsic self-healing under ambient conditions, while Fe_2O_3 composites showed moderate but stable healing performance.

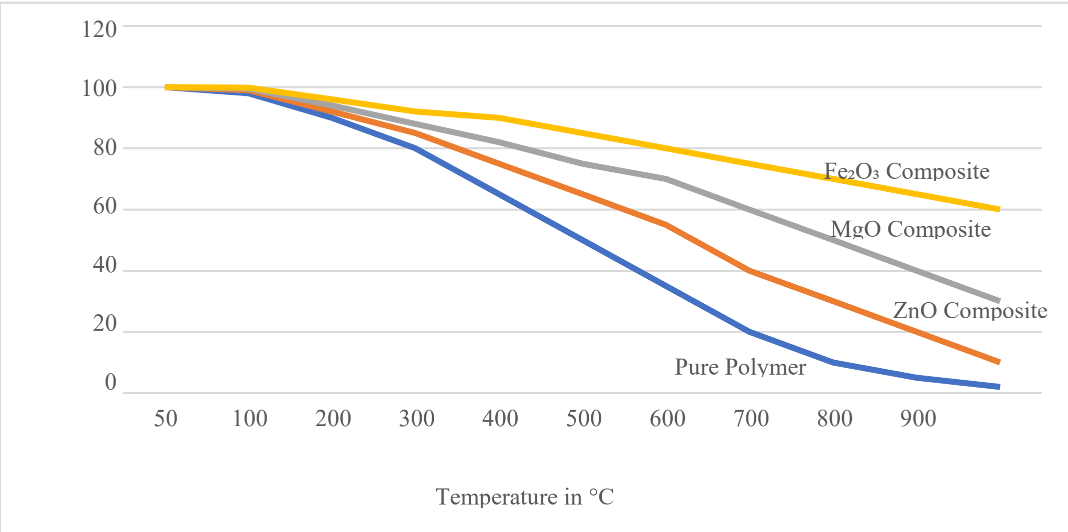

Figure 5. TGA analysis curve.

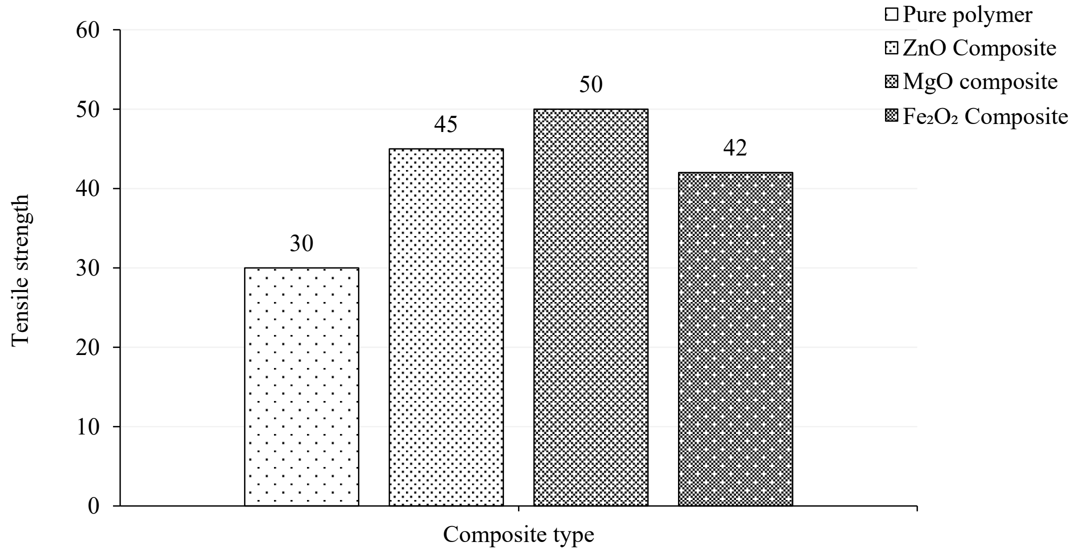

Figure 6. Tensile strength analysis.

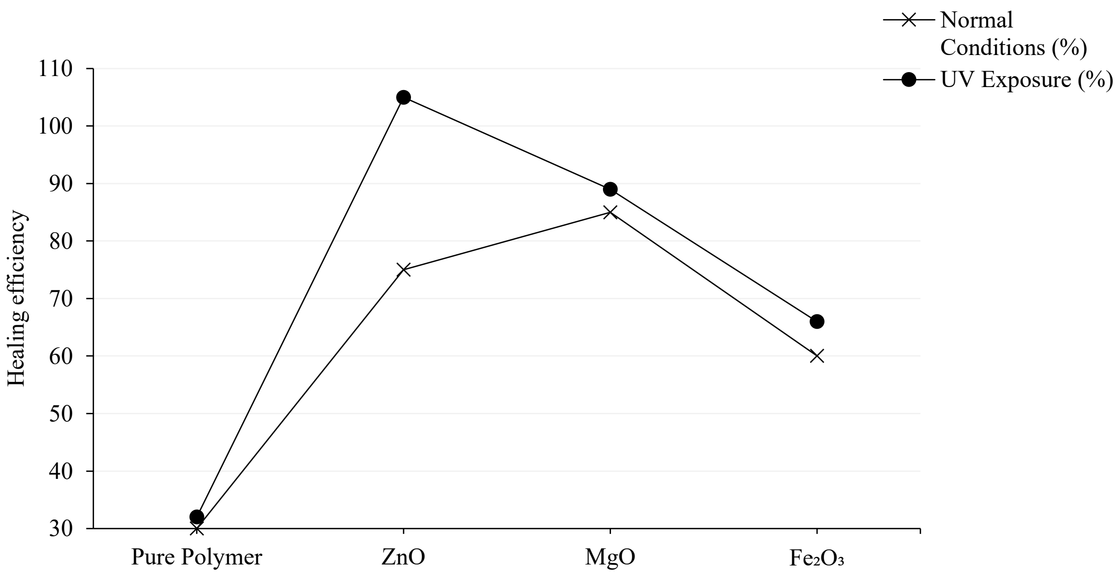

Figure 7. Comparative self-healing efficiency.

CONCLUSIONS

Green-synthesized MgO, ZnO, and Fe_2O_3 nanoparticles derived from Allium cepa were successfully incorporated into epoxy-based self-healing polymeric composites. Structural analyses confirmed high crystallinity and effective nanoparticle–polymer interactions. Thermogravimetric Analysis (TGA) showed the thermal stability of the developed polymeric composite sheets. Thermal studies revealed a significant improvement in stability, with MgO composites exhibiting the highest degradation resistance, establishing a stability trend of MgO > ZnO > Fe_2O_3 > Pure Polymer. Mechanical testing demonstrated a substantial enhancement in tensile strength, reaching 50 MPa for MgO-filled composites while ZnO Composite showed a considerable increase of 45 Mpa. Fe_2O_3 Composite exhibited improved tensile strength (42 MPa), though slightly lower than ZnO and MgO due to nanoparticle agglomeration. Self-healing efficiency under ambient conditions was 60% for Fe_2O and 75% for ZnO while it reached a maximum of 85% in MgO composites due to improved interfacial bonding and chain mobility. Under UV irradiation, ZnO composites showed exceptional healing efficiency (105%) attributed to photocatalytic activation while MgO Composite maintained strong healing efficiency (89%) and Fe_2O_3 Composite showed moderate healing improvement (66%). Fe_2O_3 composites provided moderate improvements in all properties.

Therefore, the incorporation of green-synthesized MgO nanoparticles proves to be the most effective strategy for maximizing both mechanical strengths, thermal stability and self-healing recovery in epoxy composites. Overall, the specific type of nanoparticle significantly governs the performance of the composite. This study highlights the potential of green nanotechnology for designing sustainable, high-performance self-healing materials.

Future studies may focus on optimizing nanoparticle loading to balance mechanical strength and self-healing efficiency. Hybrid reinforcement using combined MgO–ZnO systems could be explored to achieve simultaneous thermal stability and UV-responsive healing. Long-term durability, fatigue behavior, and cyclic healing performance should be evaluated for real-world applications. Advanced characterization techniques such as DMA and in situ healing analysis can provide deeper insight into healing mechanisms process. Extending this green synthesis approach to other bio-extracts and polymer matrices may broaden application potential in aerospace, biomedical, and protective coating technologies, providing a pathway for smarter infrastructure.

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