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Characterization, impact and wear properties of treated and as-received graphene nanosheets reinforcement in epoxy resin composites

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Abstract

Purpose – This study aims to explore the enhancement of mechanical properties in epoxy resin composites through the incorporation of graphene nanoparticles, focusing on their impact and wear resistance. It investigates the role of graphene, both treated and untreated, as a reinforcing agent in composites, highlighting the significance of nanoparticle dispersion and surfactant treatment in optimizing mechanical performance.

Design/methodology/approach – Employing a novel dispersion technique using a drawing brush, this research contrasts with traditional methods by examining the effects of graphene nanoparticle concentrations treated with surfactants – Polyvinylpyrrolidone (PVP) and Sulphonated Naphthalene Formaldehyde (SNF) – on the mechanical properties of epoxy resin composites. The methodology includes conducting a series of impact and wear tests to assess the influence of graphene reinforcement on the composites' performance.

Findings – The findings reveal a marked enhancement in the composites impact resistance and energy absorption capabilities, which escalate with an increase in graphene content. Additionally, the study demonstrates a significant improvement in wear resistance, attributed to the superior mechanical properties, robust interface adhesion and effective dispersion of graphene. The use of surfactants for graphene treatment is identified as a crucial factor in these advancements, offering profound insights into the development of advanced composite materials for diverse industrial uses.

Originality/value – This study introduces a unique dispersion technique for graphene in epoxy composites, setting it apart from conventional methods. By focusing on the critical role of surfactant treatment in enhancing the mechanical properties of graphene-reinforced composites, it provides a novel insight into the optimization of impact and wear resistance.

Keywords Graphene nanoparticles, Epoxy resin composites, Impact test, Wear resistance, Homogeneous dispersion, Surfactant treatment

Paper type Research paper

1. Introduction

1.1 Nanocomposites

Polymer nanocomposites offer a promising solution to the challenges faced in the applications of micro and macro composites. The primary advantages of nanocomposites

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over micro- and macro-composites include enhanced properties, sustainability by reducing material usage, increased strength-to-weight ratio, compatibility with conventional manufacturing methods such as extrusion, injection molding, compression molding, etc. improved barrier properties, increased thermal stability and degradation resistance and multi-functionality.

Among the alternative polymer matrices, epoxy resin composites are known for their superior mechanical strength, making them indispensable in a variety of engineering and industrial applications (Li *et al.*, 2023). Because of their excellent mechanical properties and adaptability, epoxy resin composites have become paramount materials in advanced engineering applications (Jiang *et al.*, 2023). Despite their excellent properties, the search for improving their mechanical properties, particularly in terms of impact and wear resistance, is an ongoing challenge in materials science. Several studies investigated and reported the tribological response of the matrix of different materials to the addition of micro, macro and nanoreinforcements of different sizes. Significant improvements have been seen in the flexural and tensile performance of epoxy composites reinforced with Kulkual fiber and TiB2 particles (Selman *et al.*, 2023). The incorporation of nanomaterials, especially graphene, has been a clear improvement in this area. Graphene's remarkable mechanical, thermal and electrical properties have made it an ideal candidate for reinforcing epoxy resin composites, leading to substantial improvements in its overall performance (Lin *et al.*, 2023; Baishya *et al.*, 2023). Graphene distinguishes itself from other nanoparticles due to its high mechanical, thermal and electrical properties (Geim and Novoselov, 2009; Algarni *et al.*, 2022; Ali *et al.*, 2023). Adding graphene to epoxy resins significantly improved their mechanical properties, such as stiffness, strength and toughness (Stankovich *et al.*, 2006; Kim *et al.*, 2010). This effect is due to the graphene's high surface area, inherent strength and ability to effectively transfer the load to the epoxy matrix (Mostovoy *et al.*, 2022; Wang *et al.*, 2023). Moreover, recent studies have demonstrated that graphene-reinforced composites exhibit improved corrosion resistance, which is crucial for their longevity in industrial applications (Tang *et al.*, 2022).

1.2 Biocompatibility

Nano-MoS_2 particles improved the wear characteristics of the Al-TiB_2-Gr composite significantly (Chenrayan *et al.*, 2022). The hardness and elastic modulus of high-density polyethylene thermoplastic polymer (HDPE) were improved by multi-walled carbon nanotubes (MWCNT)/nano-alumina composites. After the material composition was optimized, it was discovered that the hybrid composite with the structure (2.4% Al_2O_3 and 0.6% MWCNT) had superior mechanical capabilities than ratios with 3 and 5% MWCNTs. The addition of MWCNT to HDPE boosted the percentage of crystallization to 6%, according to thermal gravimetric analysis (TGA). With 5% MWCNT, the moisture absorption dropped to 90%. Colorimetric assay macrophage cytotoxic activity (MTT) results for a normal human epithelial cell line (1-BJ1) over Al2O3/MWCNT-HDPE showed less than 20% cytotoxic action, indicating that it can be used medicinally. Al2O3/HDPE/MWCNT nanocomposites have become a popular choice for artificial joint materials (Dabees *et al.*, 2020).

1.3 Other applications

The wear rate drops with increasing graphene concentration, according to a study on the tribological performance of TiB2-graphene Al 7075 hybrid composites manufactured by squeeze casting at both room temperature and high temperature (Chenrayan *et al.*, 2023). Adhesion drives the wear mechanism at ambient temperatures, while abrasion controls the wear process at high temperatures. The wear mechanism changes from moderate to severe as the temperature rises. The creation of a stable, coherent and adherent transfer layer was the reason why three-weight percent MWCNTs and 1.2-weight percent Al_2O_3/HDPE nanocomposites outperformed other compositions and demonstrated the best tribological performance and lowest friction coefficient. When comparing nanocomposites to pure HDPE, MWCNTs with Al_2O_3 reinforcement changed the wettability (from a contact angle of ~93° ± 2°–~116.6° ± 3°) and surface energy (from ~25 to ~17.6 mN/m). The hybrid calcium grease, including multi-walled carbon nanotubes and graphene nanosheets, demonstrated exceptional lubricating capabilities (Mohamed *et al.*, 2020). Biocompatible and degradable nanoparticles are extensively used for effective drug delivery (Yadav *et al.*, 2022). In addition to the enhancement of the structural properties of the polymers, nanomaterials are also applied as nanocatalysts, prepared by eco-friendly processing (Roy *et al.*, 2021). Up to two weight percent of MWCNTs, the conductivity was found to be unaffected by investigations into electrical characteristics. The conductivity of three-weight percent MWCNTs/HDPE nanocomposites increased sharply to 4.75 × 106 Hz, after which it declined and showed the same pattern as that of one and two-weight percent pure HDPE nanocomposites (Dabees *et al.*, 2021). The manufacture of nano-hydroxyapatite (nHAp) using eggshells from two distinct Indian breeds, Aseel and Kadaknath. Polyurethane 45-PPI was used as a porous template in the foam replica procedure to create the alloplastic implants. The sample's maximum compression strength was 5.49 ± 0.12 MPa, within the range of human trabecular bone's compression strength. The findings of the thermal and degradability investigations verified that these are extremely stable and offer the resorption required for the production of new bone tissue. Additionally, the cell survival toward MG-63 human osteoblast-like cells and the antibacterial activity against tested human microbiota are adequate, suggesting a possible route for the development of nHAp implants for bone tissue engineering (Santosh Kumar *et al.*, 2023). Eggshell particle density rises with increasing eggshell content, and the modulus and strength of eggshell/epoxy composites are found to be sensitive to strain rate fluctuations based on quasi-static compression experiments. All of the eggshell/epoxy composites, however, show greater moduli when compared to neat epoxy specimens, regardless of the strain rates. The rise in modulus of composites is between 17 and 57%, 3 and 107% and 30 and 114% for 1.43, 0.1 and 0.001 strain rates, respectively (Gossaye *et al.*, 2022). Conducting polymers, like polyaniline, polypyrrole and polythiophene reinforced with coated nanoparticles, have high conductance and dielectric properties that make them suitable for electromagnetic interference (EMI) shielding applications. Composites based on graphene and carbon nanotubes offer superior EMI shielding efficacy. Additionally, there are many aspects that impact the effectiveness of EMI shielding materials (Shahapurkar *et al.*, 2022).

1.4 Challenges

Despite these advances, challenges exist in achieving homogeneous graphene dispersion within the epoxy matrix and preventing agglomeration. These variables have a major impact on the mechanical properties and performance of composites. Recent research has investigated various techniques, including surface modification and the use of surfactants, to improve the dispersion and interaction of graphene within the resin matrix (Alshahrani *et al.*, 2022). The current study contributes to this evolving field by investigating optimal methods for mixing graphene with resin, examining the effects of graphene content on mechanical properties and exploring the role of surfactants in the dispersion and performance of graphene-reinforced epoxy composites.

Despite potential improvements, homogeneous dispersion of graphene within the epoxy matrix and preventing agglomeration remain significant challenges. Uniform dispersion is critical because it directly affects the composite's physical behavior and overall performance (Loeffen *et al.*, 2021; Potts *et al.*, 2011; Khurshid *et al.*, 2015; Ilyas *et al.*, 2017). Furthermore, the interaction between graphene and the epoxy matrix, particularly the role of surfactants like Polyvinylpyrrolidone (PVP) and Sulphonated Naphthalene Formaldehyde (SNF), in enhancing this interaction. The current study aims to address these challenges by investigating the optimal methods for mixing graphene with resin, examining the effects of graphene content on mechanical properties and exploring the role of surfactants in the dispersion and performance of graphene-reinforced epoxy.

2. Materials and methods

The study involved a series of wear and impact tests on both control samples and those reinforced with graphene. For the epoxy samples, the process began with mixing the epoxy resin and hardener sourced from SACO Company, Riyadh, Saudi Arabia, in a 1:1 ratio. Subsequent batches were prepared by adding graphene, sourced from Shanghai Xinglu Chemical (Shanghai, China), to the resin in varying weight percentages, specifically 0.1, 0.2, 0.4, 0.6, 1 and 1.5%. Graphene was a preferred choice for nanoreinforcement due to its compatibility with the epoxy polymer matrix and improved properties such as exceptional mechanical properties and electrical and thermal conductivities, a high aspect ratio, high thermal and chemical stability, an impermeable nature offering better barrier properties and versatility offering multifunctional applications.

In the preparation of graphene-reinforced samples, two surfactants were used to treat the graphene powder: PVP, sourced from Sigma–Aldrich Chemicals and SNF, provided by Methanol Chemicals Company, Jubail, Saudi Arabia.

To assess the impact of various graphene modifications, a total of ten samples were prepared. This set included variants such as PVP-graphene, SNF-graphene and commercially available graphene. Additionally, a group of samples without any graphene served as the control.

Figure 1(a) through (d) shows the tendency of graphene particles to cluster when mixed with the epoxy resin by different methods. The graphene powder was treated by each surfactant separately using a sonication device for 10 min, followed by drying at 80 °C before being added to the epoxy resin. Once adequately mixed, the resin-hardener blend was carefully poured into predetermined molds designed for subsequent mechanical testing. To ensure the elimination of entrapped air and potential voids within the material.

To achieve a homogenized mixture of graphene within the epoxy resin, a novel approach was employed, deviating from traditional laydown techniques, which yielded a uniform composite, as shown in Figure 1(d). Instead of using standard methods, a drawing brush was utilized to spread the graphene evenly across the mold. This technique was chosen based on its potential to provide a more uniform dispersion of graphene particles throughout the resin matrix. The brush application allowed for a controlled and consistent distribution of graphene, ensuring that each area of the mold received an equal concentration of nanoparticles. This method was particularly effective in preventing the agglomeration of graphene particles, a common challenge in composite material preparation. By using the drawing brush, the graphene nanoparticles were spread in a thin, even layer, facilitating their integration into the epoxy resin during the curing process. The result was a composite material with a more uniform microstructure, potentially leading to enhanced mechanical properties.

Four types of resin substrates were prepared for each test in this study. PVP-graphene was added, the second sample was SNF-graphene, the third sample was commercial graphene and the fourth sample was a non-graphene sample. The Charpy impact test involved an average of ten samples for each type of epoxy resin examined in this study. These samples, prepared in compliance with ASTM D5942-96, the Standard Test Method for Determining Charpy Impact Strength of Polymers, were reported alongside their 95% confidence intervals.

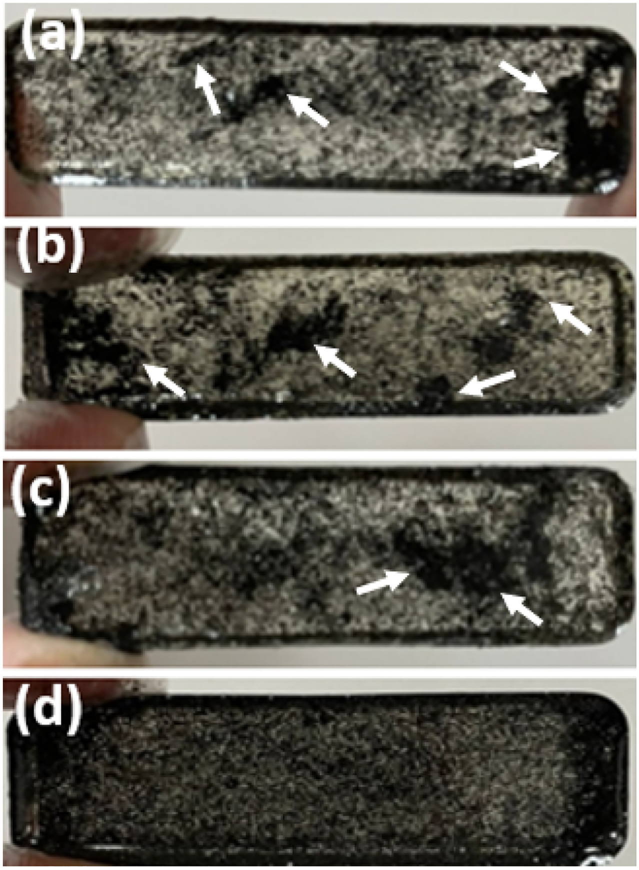


Figure 1.
Epoxy-graphene composites. (a) 1.5% wt graphene, added by traditional laydown technique. (b) 1.5% wt graphene-SNF, added by traditional laydown technique. (c) 1.5% wt graphene-PVP, added by traditional laydown technique. (d) 1.5% wt graphene-PVP, added by brush laydown

The assessment of wear resistance for resin composites containing various graphene additives as well as for neat resin composites, was executed following ASTM F732 standards. This evaluation was performed using a specifically designed tribometer, illustrated in Figure 2 as we used in our previous study (Alhazmi *et al.*, 2021). The actual picture of the tribometer during the wear test and the pin after completing the test are also shown in the figure.

For the wear resistance test, ten cylindrical samples of each material, measuring 24 mm in height and 17.6 mm in diameter, were subjected to wear testing against a 220-grit bare-wood sandpaper disc in dry sliding condition in an air-conditioned laboratory with an ambient temperature ranging between 22 and 24 °C. During testing, each specimen was subjected to a consistent load of 5N. Concurrently, the sandpaper disc, serving as the abrasive surface, was rotated at a speed of 20 rpm for five minutes. The results of these wear tests were averaged across ten specimens for each material, and the findings were presented with 95% confidence intervals. The relative velocity between the pin and the rotating disc remained constant across all tests. This constancy is attributable to the fact that it is determined by the product of the disc's rotational speed and the fixed distance of the pin from the axis of rotation. Ultimately, the average relative amount of wear, along with the weight loss and wear rate for each specimen, was computed following the methodology.

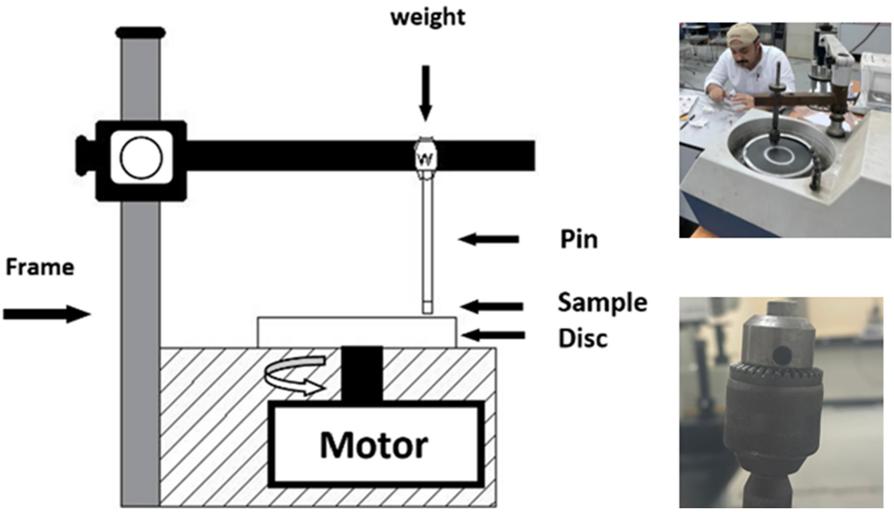


Figure 2.
Custom-built tribometer, the schematic diagram, actual picture during the wear test and the test pin after the test

3. Results and discussion

3.1 Characterization of commercial and treated graphene

The SEM analyses, conducted at several magnifications, of the graphene flakes employed in this work are presented in Figure 3. The commercial graphene flakes are shown in Figure 3(a) with an average size of 150 nm as purchased, as apparent from the scanning electron microscope picture. Figure 3(b) shows 2D parallelogram graphene sheets treated with surfactant PVP. The area and thickness of 200 graphene flakes as measured by the ImageJ software were 30–1,700 µm^2 and a size of the order of 150 nm, respectively. These values were higher than the commercial graphene flakes that were obtained, as represented in Figure 1(a). The SEM images also confirm that the PVP surfactant treatment type does not affect the structure of the produced graphene sheets. Figure 3(b) shows 2D parallelogram graphene sheets treated with surfactant SNF, and no effect of SNF surfactant treatment is observed on the size of the graphene nanosheets; however, there is a clear change in the surface morphology. The graphene flakes treated with PVP and SNF exhibit increased surface area, which is expected to improve the interfacial interaction and hence the bonding with the epoxy polymer.

The elemental composition, as determined through energy dispersive X-ray spectroscopy (EDS), is illustrated in Figure 4(a) and (b). Figure 4(a) gives the scanning electron image of graphene nanosheets treated with PVP and Figure 4(b) shows the scanning electron image of graphene nanosheets treated with SNF. Both figures demonstrate the generated flake's

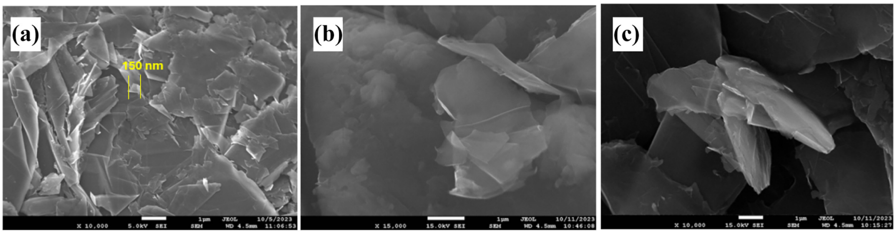


Figure 3.
Scanning electron microscope images of graphene sheets were used in this study. (a) Commercial graphene, (b) graphene + PVP and (c) graphene + SNF

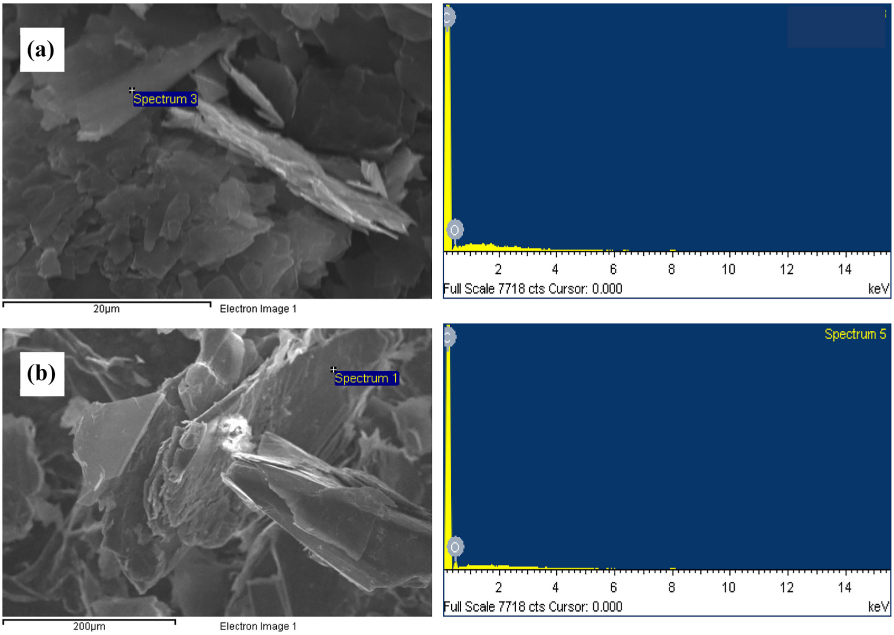


Figure 4.
SEM-EDS analyses of graphene flakes obtained by our production set-up. (a) graphene + PVP and (b) graphene + SNF

composition, with carbon constituting 98.08% and oxygen comprising 1.92%. These micrographs suggest a high-quality material formation (Shinmura *et al.*, 2012; Dresselhaus *et al.*, 2010). X-ray diffraction (XRD) analyses were carried out to further ensure that graphene was successfully synthesized and to determine its crystalline behavior. Figure 5 illustrates the XRD pattern for the commercial graphene, graphene + PVP and graphene + SNF obtained by our developed system.

The commercial graphene's XRD spectrum is shown in Figure 5(a), whereas the graphene + PVP and graphene + SNF XRD peaks are shown in Figure 5(b) and (c), respectively. The pattern for natural graphite shows the presence of a sharp peak at $2\theta = 26{,}55°$, with a corresponding interlayer spacing of 0.335 nm, followed by a peak at 54,68°, both confirming the presence of a well-organized layered structure. Figure 5(b) and (c) show a notable decrease in the intensity of the (002) peak for graphene sheets compared to commercial graphene Figure 5(a). This may suggest a reduction in the number of layers, while the unchanged peak position implies preservation of the original structure and the absence of oxygen-containing groups (Rossetti *et al.*, 2015).

3.2 Impact strength of graphene-epoxy composites

The results presented in Figure 6 demonstrate a significant reinforcing effect as graphene loading increases. A significant increase was observed at 1% graphene weight, with commercial graphene composites absorbing 1.2 Joules. PVP-graphene composites demonstrated an increase at 2.2 Joules, while SNF-graphene showed an increase at 1.74 Joules, indicating that graphene plays a larger role in energy dissipation during impact. The increasing trend of energy absorption with rising graphene content indicates that graphene's incorporation into epoxy composites significantly enhances their impact resistance. This enhancement is most likely due to graphene's exceptional mechanical properties, which include high intrinsic strength and stiffness. PVP-graphene and SNF-graphene composites,

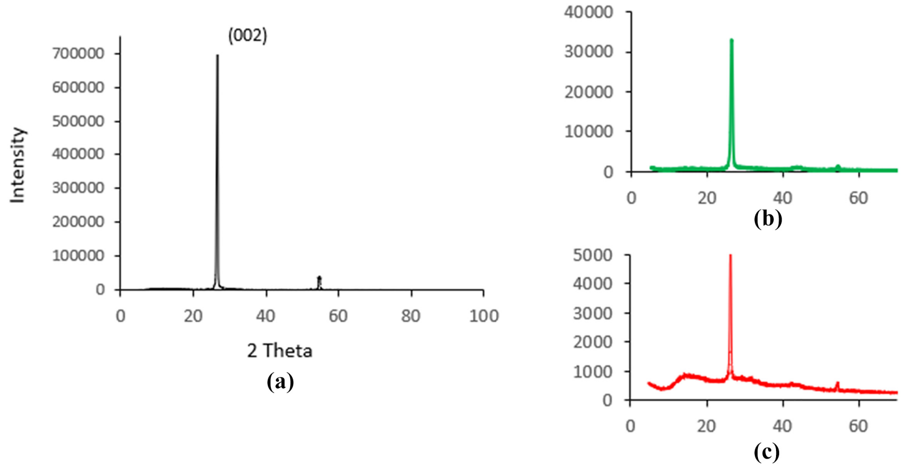


Figure 5.
XRD patterns of (a) commercial graphene, (b) graphene + PVP and (c) graphene + SNF

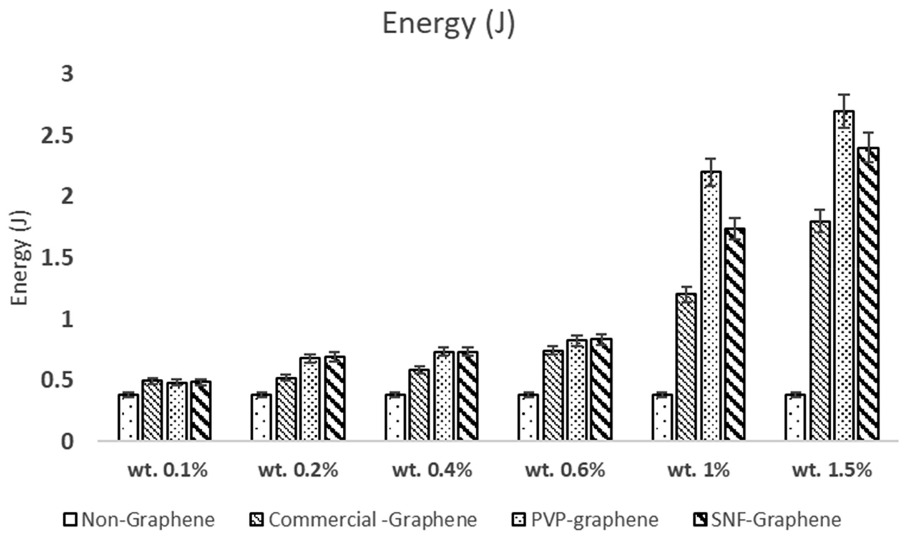


Figure 6.
Impact test results

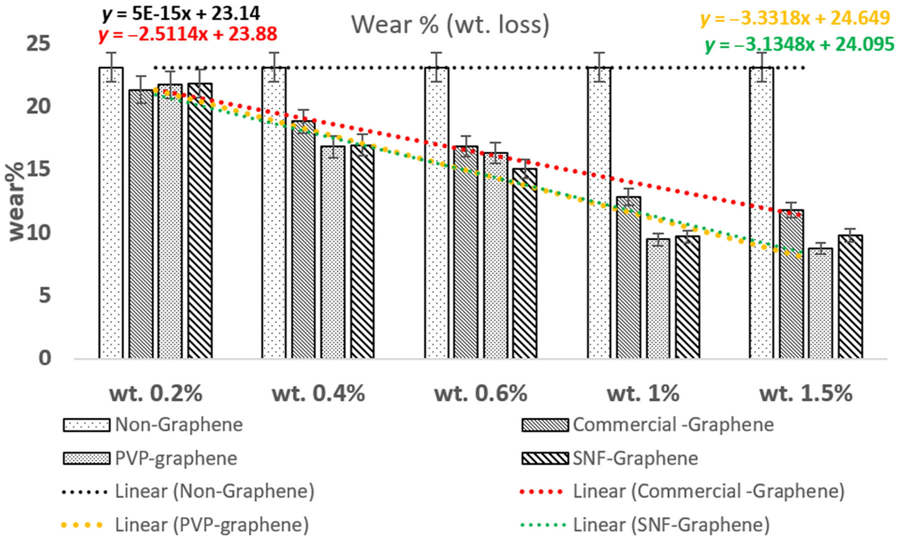
in particular, demonstrate superior performance, which may be attributed to better dispersion and interaction of the treated graphene within the epoxy matrix. These findings underscore the potential of graphene-reinforced epoxy composites for applications where high-impact resistance is paramount.

From these results, it can be inferred that the addition of graphene to epoxy resin composites leads to an increase in energy absorption during impact testing, suggesting improved impact resistance. The data show a clear trend of increasing impact energy absorption with increasing graphene content, with PVP-graphene and SNF-graphene composites typically absorbing more energy than commercial graphene composites. This indicates that the type of graphene and its compatibility with the resin matrix can have a significant effect on the impact resistance of the composite material.

The impact strength of the epoxy-graphene composites increases, more so as the composition of graphene increases, and when compared to the treated and untreated graphene, the best impact strength is exhibited by the treated graphene with PVP. The reason for improved impact strength may be attributed to the improved dispersion and alignment of treated graphene, enhanced surface adhesion with the epoxy, improved fracture toughness, homogenous microstructure and increased energy absorption due to the high aspect ratio of graphene, which also reduced peak stresses and reduced plasticity of the matrix due to the high stiffness and resistance to high strain rate loading conditions. The synergistic effect of graphene addition, treatment with surfactants, dispersion improvement and interfacial adhesion resulted in improved impact strength of graphene-epoxy composites.

3.3 Wear properties of graphene-epoxy composites

The wear results in weight loss percentages for non-graphene and graphene-reinforced epoxy samples at different compositions are given in Figure 7. At the 0.1% graphene weight percentage, all samples, regardless of the type of graphene used, show similar wear percentages, which are roughly equivalent to the wear percentage of the non-graphene sample. At 0.2% graphene addition, there is a slight reduction in wear for all graphene-reinforced samples compared to the non-graphene sample, with SNF-graphene showing the least wear resistance among the graphene. At this concentration, a significant improvement in wear resistance is observed in the graphene-reinforced samples compared to the non-graphene sample, with PVP-graphene and SNF-graphene samples showing similar wear percentages and better performance than the commercial graphene sample. At 0.6% graphene, the trend of improved wear resistance continues, particularly notable in the SNF-graphene sample, which outperforms the other samples. At 1% graphene, there is a marked decrease in wear percentage for the graphene-reinforced samples compared to the non-graphene sample. The PVP-graphene and SNF-graphene samples show exceptional wear resistance, with PVP-graphene having the lowest wear percentage. The incorporation of 1.5% graphene resulted in the lowest wear percentages observed in the study. The non-graphene sample maintained a consistent wear percentage of



Note(s): Linear regression equations are given on the chart

Figure 7.
Wear test results, wear % indicates the percent weight loss in wear

23.14%. However, commercial graphene reduced the wear to 11.79%, PVP-graphene to 8.78% and SNF-graphene to 9.81%. From the results, we can conclude that the addition of graphene, regardless of the type, generally improves the wear resistance of the epoxy samples as the percentage of graphene increases. The PVP-graphene and SNF-graphene tend to perform better than the commercial graphene, especially at higher weight percentages, indicating that the surfactant-treated graphene types are more effective in enhancing wear resistance.

The trend observed here aligns with the expected outcome that graphene reinforcement generally improves the wear resistance of epoxy composites. Notably, PVP-graphene shows the best performance in this set of results, closely followed by SNF-graphene. These findings suggest that the surface treatment of graphene particles has a significant impact on the wear resistance properties of the composites, with both PVP and SNF surfactants enhancing the performance of graphene within the epoxy matrix. The linear regression equations are given in Figure 7, and linear trend lines have been plotted. All compositions show a negative slope except the pure epoxy sample, which exhibits almost a straight line. The slopes of PVP-graphene and SNF-graphene are quite near as there is little difference in the wear rate. Among the two, PVP-graphene exhibits a marginally higher slope.

Neat graphene (i.e. graphene without surfactant) samples consistently exhibit the lowest properties across all test sets. However, after the introduction of surfactants, there has been a significant improvement observed across all samples in wear and impact properties. This improvement is due to the fact that both surfactants can enhance graphene dispersion. By comparing the two surfactants, it is obvious that PVP samples show higher values in their properties than those of SNF samples, indicating that PVP generates better dispersion than SNF.

The addition of graphene has improved the wear resistance of the nanocomposites due to the inherent superior properties of graphene and improved lubrication in dry sliding conditions due to the high lubricity of graphene attributed to its unique atomic structure, acting as a solid lubricant. The barrier effect of graphene may have inhibited the penetration of the wear-inducing particles and debris, reducing the contact area between the mating surface and hence reducing wear. The generation of heat during the dry sliding wear process is obvious due to friction, and it accelerates. The presence of graphene in the nanocomposites may have improved the thermal conductivity of the nanocomposites, resulting in enhanced dissipation of heat generated during the wear test and limiting the acceleration of the wear rate. Furthermore, the low surface energy of graphene and the reduced temperature between the mating surfaces may have inhibited the local adhesion of epoxy to the counterface, reducing the wear. Improved bonding of treated graphene with epoxy resulted in even more wear resistance.

In summary, the scientific reasons for improved wear resistance in graphene-reinforced epoxy composites are multifaceted, involving the enhancement of mechanical properties, strong interface adhesion, structural integrity under stress and optimal dispersion of graphene within the resin matrix. These factors collectively contribute to the composites' ability to withstand wear.

4. Conclusions

This study found considerable insights into the interaction between graphene and epoxy resin across different compositions through detailed experimentation involving impact and wear tests, with a particular focus on dispersion techniques and the resulting composite properties. The following primary outcomes evolved from the study:

(1) Considerable insights into the interaction between graphene and epoxy resin across different compositions have been revealed.

(2) The incorporation of graphene, especially when homogeneously mixed using innovative techniques like the drawing brush method, gives uniformly dispersed composites.

(3) A clear trend of increasing impact energy absorption with the rising content of graphene is observed.

(4) Graphene's multifaceted role in increasing wear resistance is revealed using the novel fabrication technique.

(5) At lower graphene weight percentages (0.1%–0.6%), there is a consistent improvement in impact energy absorption. As the weight percentage of graphene increases to 1% and beyond, the impact energy absorption capacity of the composites rises dramatically.

(6) The PVP-treated graphene may provide a more effective reinforcement than SNF graphene or commercial graphene at higher concentrations.

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