Damage Recovery of Epoxy Coating Reinforced Carbon Nanoparticle/Polypropylene Fibers Hybrid Filler

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RESEARCH ARTICLE

Damage Recovery of Epoxy Coating Reinforced Carbon Nanoparticle/Polypropylene Fibers Hybrid Filler

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ABSTRACT

The study investigates the influence of incorporating carbon nanoparticles (CNP) and polypropylene fibers into epoxy composites on their mechanical properties, focusing on the elastic modulus and hardness. The results show that an increase in the weight percentage of polypropylene fibers leads to a corresponding rise in the elastic modulus of the epoxy composites. The presence of nanocarbon black (CNP) in various concentrations did not have a clear effect on the elastic modulus. On the other hand, the elastic modulus increased linearly with increasing the percentage of hybrid filler (CNP/PP). Regarding to hardness test that was achieved using the shore D tester, lubrication properties improved with increasing weight percentages of CNP, while the hardness decreased a lot with increasing the weight percentage of polypropylene fibers. the results of the recovery test revealed that the CNP plays a major role in a preference for healing along the width axis of the groove, while the addition of polypropylene fiber makes the epoxy matrix favor healing along the depth axis, When using hybrid filler (CNP/PP) the recovery behavior was almost equal in both the transverse and depth axes. The above results shed light on the recovery behavior of hybrid composites and the possibility of their self-healing while at the same time improving their mechanical properties and thus increasing the requirements they perform in various engineering applications.

Keywords: Carbon nanoparticles, Epoxy coating, Hybrid filler, Mechanical properties, Polypropylene fibers

Introduction

The development of self-healing materials holds significant promise for transformative applications across various industries, offering the potential to create products and components characterized by enhanced durability and extended lifespans. These materials possess the remarkable ability to autonomously or externally repair themselves when damaged, thereby restoring their functionality and ensuring prolonged effectiveness. Within the realm of engineering research, concerted efforts have been primarily directed toward two key areas: the development of novel materials with superior durability and the formulation of nondestructive evaluation techniques for material inspection. Nevertheless, it is imperative to acknowledge that, ultimately, all engineered materials will experience some form of failure.[1] The challenge in repairing structural damage lies in the fact that most current methods necessitate external intervention to bring fractured surfaces into contact before the commencement of the healing process.[2] Self-healing materials are distinguished by their intrinsic capacity to initiate autonomous or externally-triggered repair processes, effectively restoring functionality when subjected to damage. The adoption of self-healing materials presents a pioneering approach to crafting safer and more durable

products and components with extended lifespans. [1] To ensure sustained effectiveness, it is essential to implement an active protection system capable of self-healing defects in coatings. This objective can be achieved through the incorporation of intelligent release systems within the polymer matrix, enabling the development of active coatings endowed with self-healing capabilities. [3]

To date, three primary approaches have been employed to demonstrate self-healing: capsule-based systems, vascular systems, and intrinsic polymers. Three established strategies have been deployed to integrate self-healing into polymer systems: triggering polymerization of healing agents upon damage occurrence, reconstructing the molecular network via reversible chemistry, and incorporating fusible thermoplastics into a thermoset host. Self-healing is imperative for rectifying coated areas damaged due to aging or unforeseen aggressive events. Concerning self-healing coatings for corrosion protection, two main strategies have been pursued: repairing defects in the coating matrix by introducing polymerizable agents and inhibiting corrosion in specific areas through the use of corrosion inhibitors. [4,5]

In a study conducted by Xiaofan Luo *et al.*, [6] an epoxy/PCL blend, commonly employed in construction materials, exhibited a distinct differential expansive "bleeding" behavior. This unique characteristic endows it with exceptional thermal-mending capabilities, rendering it suitable as a rigid and reversible adhesive. A similar phenomenon has been observed in various other systems where a thermoset material is combined with an amorphous or semi-crystalline thermoplastic polymer.

Chen *et al.* [7] have successfully developed a transparent organic polymeric material that exhibits the ability to self-heal under mild conditions. This material possesses a solid structure with mechanical properties comparable to commercially available epoxy resins at room temperature and below. Upon exposure to temperatures exceeding 120°C, a fraction of the inter-monomer linkages within the material dissociates, as confirmed by solid-state nuclear magnetic resonance spectroscopy. However, upon cooling, these linkages reconnect, resulting in a fully reversible process. This unique property enables the material to repair fractured sections multiple times without the need for additional components such as catalysts, monomers, or special surface treatments.

In a study by Cho *et al.*, [8] a dual-component system comprising silanol-terminated polydimethylsiloxane (PDMS) and the crosslinker polydiethoxysiloxane (PDES) was employed within a vinyl ester matrix, forming distinct phases. Additionally, a microencapsulated organotin catalyst was incorporated. Subject-
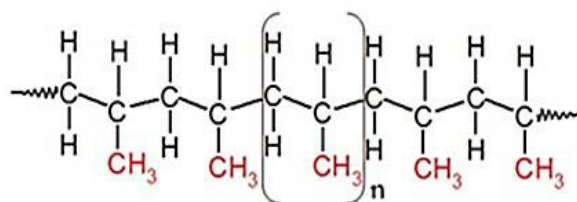

Fig. 1. Polypropylene structure adapted from. [11]

ing the material to a temperature of 50°C for 24 hours resulted in a healing efficiency of approximately 24%, as determined by the recovery of fracture toughness.

The term "hybrid composites" refers to systems that involve the incorporation or addition of one type of reinforcing or filler material into a mixture of dissimilar matrices or the presence of two or more reinforcing or filling materials within a single matrix. [9] In this specific investigation, a hybrid filler comprising thermoplastic fibers and carbon black nanoparticles was introduced to reinforce an epoxy system, to enhance its crack healing capabilities. The epoxy system, serving as the host material, is a thermoset material. [10]

The overarching goal of this study was to assess the impact of incorporating carbon nanoparticles (CNP) and polypropylene fibers into epoxy composites on their mechanical properties, specifically addressing both mechanical and structural aspects.

Materials and methods

Materials

The N220 grade of carbon black (CB), provided by Iran Carbon Co., was utilized in this investigation. The CB nanoparticles had dimensions ranging from 26 to 30 nm. Polypropylene fiber is a thermoplastic fiber with a linear structure composed of the monomer C_nH_{2n}, as depicted in Fig. 1. It is a mechanically durable material that is white in color and exhibits resistance to various chemical solvents, bases, and acids. The polypropylene fibers employed in this study were in a chopped form, with lengths of 5–10 mm and diameters of 1–3 μm.

Carbon black nanoparticle surface modification

To improve the dispersibility of nanocarbon particals in the epoxy matrix, the carbon was treated with a sodium Dodecyl sulfate (SDS) as a surfactant. Initially, one gram of (CNP)carbon nanoparticles was dispersed in 100 ml of distilled water using ultrasonic at 25°C for 2 hours. After, adding the SDS

with sonication for 30 min, the resultant mixture was finally filtered and dried at 100°C using a vacuum oven.

Synthesis of nano-composites

To prepare the nanocomposite material (NPC/Epoxy), different weight percentages of carbon nanoparticles (0.5%, 1%, and 2%) were introduced into the epoxy matrix using ultra-sonication and mechanical stirring for one hour. Later, the mixture is subjected to vacuum treatment to remove air bubbles. After that, the hardener was introduced and mixed quietly, the resultant mixture was subsequently placed in an oven, where it was cured at 100°C for four hours.

In like manner, epoxy resin mixed with different weight percentages of polypropylene fibers (0.5%, 1%, and 2%) using mechanical stirring. After, these blends were treated by vacuum at 70°C for 30 minutes, and then the hardener where introduced, followed by gentle mixing. Finally, the samples were subjected to curing in an oven at 100°C for four hours.

For the hybrid samples, differing ratios of carbon black N220 nanoparticles to polypropylene fibers (25:75, 75:25, 50:50) were dispersed within the epoxy resin through ultra-sonication, with a consistent weight percentage of 2%. The subsequent procedural steps remained consistent with those described above.

Results and discussion

Mechanical characteristics

The three-point bending flexural test stands as a valuable technique for ascertaining the modulus of elasticity in bending. Notably, it possesses the advantage of straightforward preparation and execution. As delineated in Fig. 2a, the outcomes demonstrate that the elastic modulus of epoxy, when reinforced with polypropylene fiber, exhibits an augmentation commensurate with the increased fiber content.[12] Contrarily, the incorporation of carbon black at diverse weight percentages fails to yield a corresponding elevation in the elastic modulus of the resultant composites. This absence of improvement can be attributed to the agglomeration of carbon black, exerting an adverse influence on composite properties.[13] Conversely, Fig. 2b illustrates a noteworthy escalation in the elastic modulus when employing different proportions of the hybrid filler.

Fig. 3a, elucidates the correlation between the hardness of Nano carbon black N220 particles and the weight fraction of polypropylene fibers. In parallel, Fig. 3b depicts the association between hardness and the weight fraction of the hybrid filler, PP/CB. The findings underscore that shore D hardness experiences a slight upswing with increasing carbon black content, albeit it exhibits a marginal dip as the weight percentage of PP fibers escalates.[14,15] It is noteworthy that the judicious inclusion of carbon black as a reinforcing agent within epoxy has been observed to profoundly enhance its load-bearing capacity and mechanical attributes the inclusion of carbon black as a reinforcing agent within epoxy composites has been observed to significantly enhance their load-bearing capacity and mechanical attributes. This can be attributed to several factors. Firstly, carbon black nanoparticles have a high aspect ratio and large surface area, which allows for effective stress transfer between the epoxy matrix and the nanoparticles. This results in improved load-bearing capabilities and enhanced mechanical properties of the composites.[16]
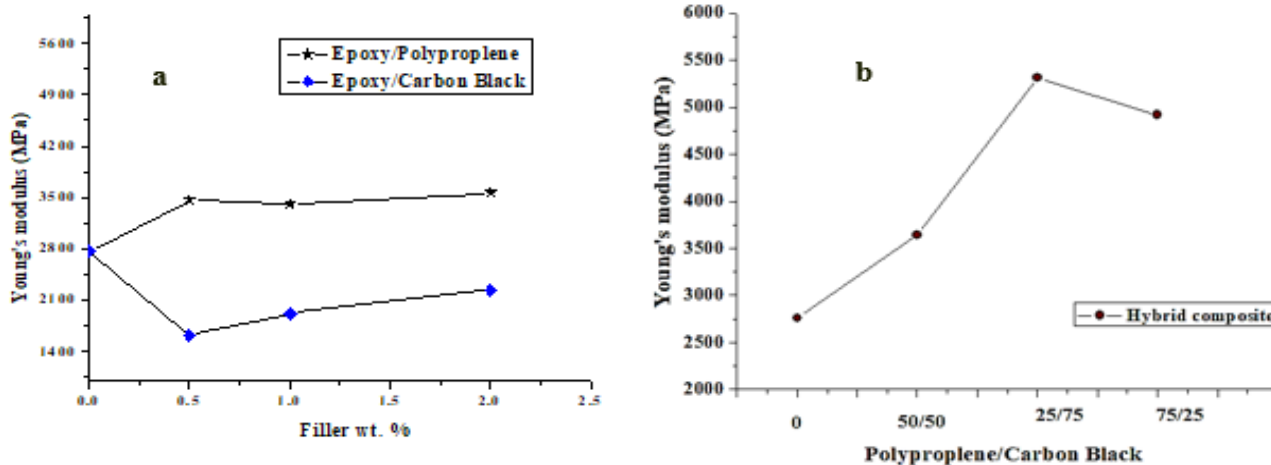

Fig. 2. Effect of a) Nano carbon black (N220) and PP fibers, b) hybrid filler (PP/CB) on the young modulus of epoxy.
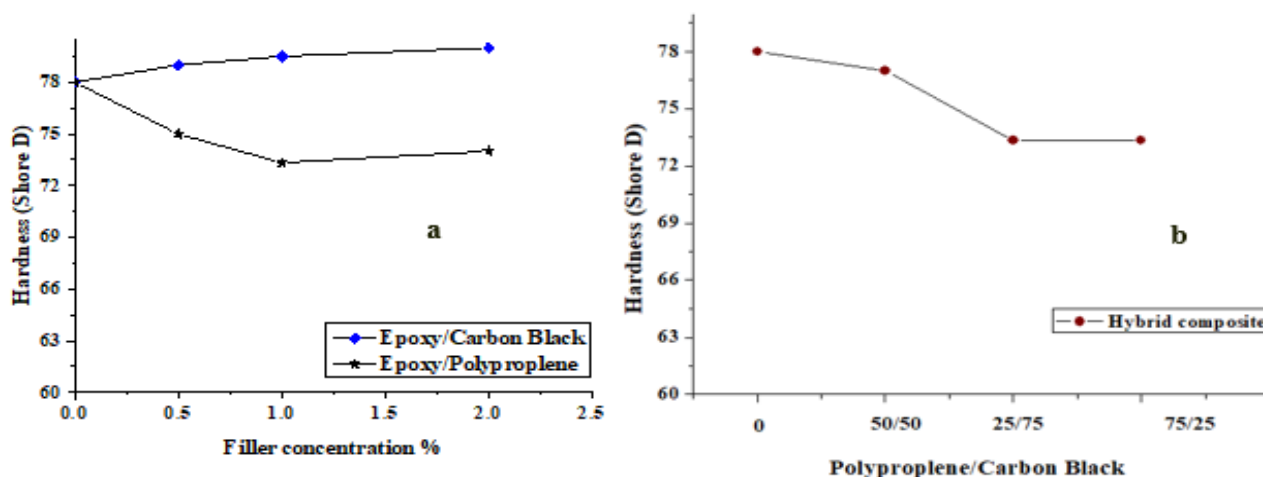
Fig. 3. Effect of a) Nano carbon black (N220) and PP fibers b) hybrid filler (PP/CB) on hardness properties of epoxy.

Secondly, the presence of carbon black nanoparticles can promote the formation of a percolation network within the epoxy matrix. This network enhances the overall strength and stiffness of the composites, leading to increased load-bearing capacity.[17] Furthermore, carbon black nanoparticles exhibit excellent dispersion and interfacial bonding with the epoxy matrix. This uniform dispersion and strong interfacial adhesion contribute to the effective transfer of stress and load across the composite, resulting in improved mechanical properties.[18] This enhancement is particularly evident when considering the maximum hardness achieved, which registers at 81, in contrast to the initial hardness of pristine epoxy, quantified at 78.

Recovery examination

The assessment of recovery behavior, about morphology, was undertaken by employing optical microscopy. Fresh scratches were deliberately introduced onto the surface of the cured samples, utilizing a precise razor blade. After the creation of these scratches, the ensuing recovery behavior was scrutinized, subject to heat treatment at a controlled temperature of 130°C, employing an optical microscope (OM). As portrayed in Fig. 4, the OM images chronicle the evolution of scratches on the surfaces of nanocomposites formulated from epoxy and carbon black. Additionally, these images document the progression of scratches on epoxy composites fortified with polypropylene fibers at varying weight percentages (0, 0.5, 1, and 2 wt. %) The weight percentages of polypropylene fibers in epoxy composites can have an impact on the progression of scratches. As the weight percentage of polypropylene fibers increases,

the scratch width demonstrates a significant decline, while the scratch depth registers an increment. This means that the scratches become narrower but deeper as the polypropylene fiber content increases.[19] The addition of polypropylene fibers can improve the toughness and impact resistance of the epoxy composites. The fibers act as reinforcement, absorbing and distributing stress, which leads to a reduction in scratch width. The increase in scratch depth is because the polypropylene fibers have a weak bond with the epoxy matrix. This leads to the creation of stress concentration points on the interphase, leading to increased plastic deformation behavior and thus a lower recovery rate and deeper scratching 19. This explanation is consistent with a study conducted by 19, which investigated the effect of weight ratios of polypropylene fibers on the mechanical properties of epoxy composites. The results showed that as the polypropylene fiber content increased, the scratch width decreased, while the scratch depth increased.

The images showed the effect of the black carbon fiber and polypropylene hybrid filler, with different mixing ratios (50/50, 25/75, and 75/25). The mechanism of transformation of scratches on epoxy compounds reinforced with a hybrid filler. Insights on the transformation of scratches on epoxy compounds.

In the case of pure epoxy, the percent of the recovery in the scratch width shows a slight increase when exposed to temperatures up to 130°C, without any obvious change in the scratch depth. This is due to the thermal stability and mechanical properties of pure epoxy.

Epoxy is characterized by its tolerance to relatively high temperatures without collapse or changes in its physical properties, meaning that its excellent thermal stability characterizes it, we note that the epoxy
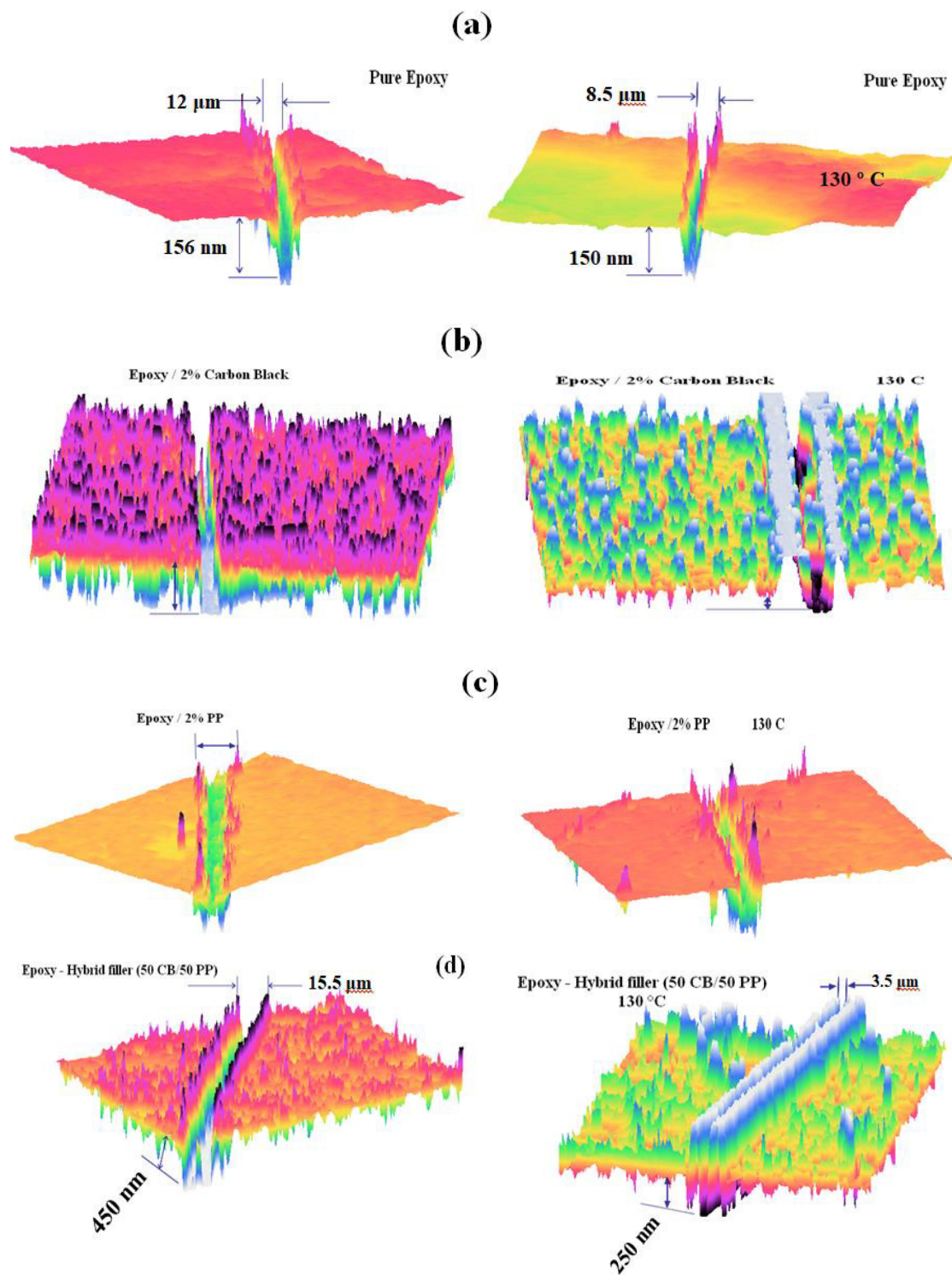
Fig. 4. It presents the results of optical microscope images that show the scratch recovery mechanism for several coatings. a) Pure epoxy, b) epoxy/carbon black nanocomposites, c) epoxy/polypropylene composites, and d) epoxy reinforced with a hybrid filler of carbon black and polypropylene at a 50/50 mixing ratio.

material is not affected even when the temperature reaches 130°C, which makes it highly resistant to deformation. The slight decrease in the width of the scratch may be due to the slight expansion that occurs in the epoxy matrix at high temperatures, with no apparent change in the depth of the scratch.

The results also showed that most of the mechanical properties of pure epoxy, such as its hardness and stiffness, remain relatively stable at temperatures up to 130 degrees Celsius. It is worth noting that at high temperatures, the specific behavior of pure epoxy depends on several factors such as the structural composition of the epoxy, the curing conditions, the nature of the scratch, or the amount of load applied. Further research and experimentation may be necessary to fully understand the thermal response of pure epoxy in the context of scratch progression.

Conversely, within the Epoxy/CB nanocomposites, the depth of the scratch undergoes a substantial decrease (measuring 29.7%), while the width experiences a minor reduction compared to pure epoxy, as elucidated in Fig. 4b. In contrast, when epoxy is fortified with polypropylene fiber, the scratch width demonstrates a significant decline (57.8%), whereas the depth registers an 85.6% increment. This observation signifies that epoxy reinforced with CB favors self-healing along the x-axis direction, while epoxy reinforced with PP exhibits a proclivity for self-healing along the z-axis direction.[20] These results underscore that the presence of CB within the epoxy matrix promotes self-healing behavior in one direction, while the incorporation of PP within the matrix fosters self-healing behavior in an orthogonal direction.[21–23]

The behavior of hybrid composites is characterized by a more balanced recovery, encompassing both the depth and width of the scratch. This equilibrium arises due to the role of carbon black in facilitating even heat distribution within the composite structure, thereby expediting the thermal expansion of polymer fibers and closure of the scratch groove's sidewalls, as depicted in Fig. 4d. Table 1 provides an overview of the recovery percentages for depth and width across different epoxy composites it appears that the concentration with the highest recovery percentage for both depth and width is at the 8th concentration level. The recovery percentage for depth at the 8th concentration level is 95.2%, which is the highest among all the concentrations. This indicates that the epoxy compound at this concentration showed the highest ability to restore its original depth after being scratched. It was also noted that the recovery percentage of width at the eighth concentration is 79%, which is also one of the highest percentages compared to other concentrations. The above results showed that the epoxy compound at this concentration can restore its original width after being scratched.

Table 1. Summarizes the recovery ratios (depth/ width) obtained for several epoxy coatings and their composites.

Sample names	Depth recovery %	Groove width recovery %
0%	3	29
0.5%CB	27.7	15.8
1%CB	23.3	18
2%CB	29.7	12.07
0.5%PP	33.25	−16.2
1%PP	45	−35
2%PP	57.8	−85.6

Conclusion

This work investigated the mechanical behavior of epoxy composites reinforced with carbon nanoparticles and polypropylene fibres.The outcomes revealed that the incorporation of polypropylene fiber resulted in an augmented elastic modulus of the epoxy, whereas the inclusion of carbon black engendered a converse effect, leading to a decrement in the elastic modulus. Moreover, a notable surge in the elastic modulus was observed when different proportions of the hybrid filler (comprising carbon nanoparticles and polypropylene fiber) were introduced into the epoxy matrix.

The evaluation of epoxy composite hardness exhibited a moderate enhancement with escalating carbon black content. Nonetheless, there was a slight reduction in hardness associated with higher weight percentages of polypropylene fiber.

Regarding recovery behavior, the epoxy-reinforced matrix containing carbon black exhibited a preference for recovery along the width axis (x-axis), while the epoxy-reinforced polypropylene matrix displayed a predilection for recovery along the depth axis (z-axis). In the instance of hybrid composites, the recovery behavior demonstrated a more balanced nature, encompassing both depth and width dimensions.

Acknowledgment

Authors' declaration

- Conflicts of Interest: None.
- We hereby confirm that all the Figures and Tables in the manuscript are ours. Furthermore, any Figures and images, that are not ours, have been included with the necessary permission for republication, which is attached to the manuscript.

- No animal studies are present in the manuscript.
- No human studies are present in the manuscript.
- Ethical Clearance: The project was approved by the local ethical committee at University of Babylon.

Authors' contribution statement

A. E. A. contributed to the conceptualization of the research, methodology development, data collection and analysis, and manuscript writing. M. A. A. M. played a key role in the investigation, data acquisition, and contributed to the drafting of the manuscript. Y. A. A. contributed to the research design, project supervision, and manuscript review and editing. M. K. I. contributed in AFM inspection and Image analysis.

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دراسة خصائص الشفاء الذاتي لطلاء الايبوكسي المدعم بحشو هجين من الياف البروبلين وجسيمات الكاربون النانوية

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الخلاصة

ركز البحث على تقييم تأثير دمج جزيئات الكربون النانوية (CNP) وألياف البولي بروبيلين في مركبات الإيبوكسي على خواصها الميكانيكية في مجال علم المواد. أظهرت التجربة أن زيادة النسبة الوزنية لألياف البولي بروبيلين أدت إلى زيادة معامل المرونة في متراكبات الايبوكسي. وعلى العكس من ذلك، فإن دمج أسود الكربون، بنسب وزن متفاوتة، لم يسفر عن زيادة مقابلة في معامل المرونة للمركبات الناتجة. على العكس من ذلك، لوحظ تحسن ملحوظ في معامل المرونه عند استخدام نسب مختلفة من الحشو الهجين، المشار إليه بـ CNP/PP. علاوة على ذلك، أشار تحليل الصلابة، والتي تم قياسها باستخدام مقياس Shore D، إلى تحسن طفيف مع تصاعد تركيز أسود الكربون، في حين لوحظ انخفاض طفيف مع زيادة نسبة وزن ألياف البولي بروبيلين. سلط البحث أيضًا الضوء على سلوكيات التعافي المتميزة في العينات المعززة بالإيبوكسي، حيث أظهرت عينات أسود الكربون تفضيلًا للشفاء على طول محور العرض، و عينات البولي بروبيلين تفضل الشفاء على طول محور العمق. في سياق المركبات الهجينة، تبين أن سلوك الاسترداد كان موزعًا بشكل أكثر توازناً، بما في ذلك أبعاد العمق والعرض.

الكلمات المفتاحية: جسميات الكاربون النانوية، طلاء الايبوكسي، الحشوات الهجينه، الخواص الميكانيكية الياف البولي بروبلين، الشفاء الذاتي.