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DESIGN AND FABRICATION OF GREEN POLYESTER-BASED ADHESIVES REINFORCED BY BIO-BASED NANOPARTICLES

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Abstract—This study aims to enhance the mechanical and adhesive properties of polyester (PE) resins by incorporating bio-derived chitin nanowhiskers (CNWs) into the polymer matrix. These green nanocomposites could serve as a safer alternative to commonly used epoxy adhesives. The effects of CNW loadings on the nanocomposites' chemical structures, tensile strength, adhesive performance, and thermal stability were investigated. Experimental results reveal that the tensile strength and adhesive strength increased by 23% and 10%, respectively, when 0.50 wt.% and 0.25 wt.% of CNWs were added; however, a further increase in CNW loading beyond these levels suppressed the mechanical properties, likely due to increased CNW agglomeration and ethanol-induced PE degradation. In short, this work highlights CNW-reinforced polyester resins as a promising, sustainable solution for green adhesive applications, balancing performance and environmental impact.

Polyester resins; chitin nanowhisker; green adhesive; nanocomposites; bio-derived; nanoparticles; waste; recycling; tensile strength; thermal stability; degradation

I. INTRODUCTION

Adhesives that can strongly bond substrates together play a critical role in a wide spectrum of industrial and commercial applications. The global adhesives market was valued at USD $59.79 billion and is projected to reach USD $87.04 billion by 2028 [1]. Some common types of adhesives include epoxy, polyurethane, and polyimide adhesives. Among them, epoxy represents the most widely used adhesive, accounted for a global market value of USD $12.84 billion in 2022 [2]. Advantages of epoxy adhesives include excellent mechanical properties, strong adhesion to fibers in composites, good chemical resistance, high hydrophobicity, and low shrinkage during curing [3-4]. While cured epoxy is generally safe and widely regarded as an industry-standard adhesive, exposure to uncured epoxy resins or epoxy dust can pose significant health risks, including skin irritation, allergies, and asthma. Epoxy dust, often encountered in manufacturing processes, can accumulate in respiratory system, highlighting the need for proper handling and safety measures. Moreover, epoxy resins, which rely on bisphenol A derivatives like diglycidyl ether of bisphenol A (DGEBA), are linked to various health concerns.

When moderate mechanical performance is needed, polyester adhesives and resins are widely used due to their high chemical resistance and curing efficiency. Polyester (PE) resins, while not dependent on bisphenol A, often incorporate styrene as a comonomer to modify their properties based on the resin composition. Styrene, however, poses health risks. Toxicological assessments by the United Kingdom government and an assessment report by the Government of Canada (Environment Canada and Health Canada) have documented adverse effects of styrene exposure, such as respiratory issues (irritation, wheezing, coughing, pulmonary edema), skin conditions (itching, dermatitis), and severe neurological and cardiac effects (arrhythmias, coma, dizziness, ataxia) [5-6]. Furthermore, according to the United States Centers for Disease Control and Prevention and the National Institute for Occupational Safety and Health, exposure to styrene-containing resins (e.g., polyester resins) or epoxies has been linked to increased risks of fertility problems, miscarriage, stillbirth, and birth defects [7]. Historical cases provide further evidence of these risks. In 1974 and 1976, two pregnant women exposed to styrene, polyester resins, and organic peroxides gave birth to children with severe defects. One child was stillborn due to anencephaly, while the other was born with congenital hydrocephalus, an anomaly of the right ear, and bilateral malformations of the thoracic vertebral column and ribs [8]. These health hazards associated with styrene and resins underscore the need for safer, more sustainable adhesive alternatives. Bio-based and biocompatible materials, such as chitin nanoparticles, offer a promising way to reinforce PE-based adhesives. Incorporating nanoparticles into resin formulations can improve mechanical properties and thermal stability while reducing reliance on styrene and mitigating its associated health risks. These advancements pave the way for safer, more sustainable materials across various applications.

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In this context, PE-based adhesives that are manufactured without toxic additives, such as styrene, provide an alternative with the potential to mitigate health risks through nanoparticle incorporation in polyester matrices. This would expand bonding applications to water purification [9], cosmetic additives [10], waste removal [11], and biomedical applications [12]. For low-stress or low-performing requirements, PE resins are used as they are often cheaper despite their reduced mechanical properties. Cured OE resins were found to have a tensile strength of 33 MPa [13] and a compressive strength of 109 MPa [14]. Compared to epoxy, the tensile and flexural strengths of PE resins are approximately 80% and 30%, respectively [15]. Innovative approaches are essential to enhance the mechanical properties of PE resins, enabling them to compete with epoxy-based adhesives.

Integrating nanoparticles into PE-based adhesive matrices has emerged as a pivotal approach to narrowing the mechanical performance gap between PE and epoxy adhesives. PE resins were chosen in various applications due to their distinctive properties and versatility. Although PE resins typically have lower mechanical strength than epoxy, they can be benefited significantly from nanoparticle incorporation. This strategy builds on extensive research demonstrating that nanoparticles can enhance the thermomechanical properties of adhesives. Studies with epoxy adhesives have shown that nanoparticles greatly enhance thermal stability and toughness. Rahman *et al.* incorporated ferric oxide, titanium oxide, and synthesized nickel ferrite nanoparticles in unsaturated PE resins, leading to improvements of 21.62% in tensile strength and 6.56% in Young's modulus [16]. Chu *et al.* incorporated ramie fabric in polyester resins and improved tensile strength from 46.8 MPa to 74.1 MPa, an increase of 27.3 MPa [17]. While studies have shown that nanoparticles significantly enhance adhesive properties — such as a 130% increase in lap-shear strength using halloysite nanotube/nanosilica core/shell nanoparticles in steel-epoxy/carbon fiber systems [18] — a pressing need remains to narrow the thermomechanical performance gap between PE resins and epoxy. Achieving this would pave the way for viable, eco-friendly alternatives. Enhanced polyester resins could serve as competitive options for high-performance applications, instead of being limited to low-performance, cosmetic, or low-stress uses.

This paper focuses on incorporating chitin nanowhiskers (CNW) into polyester-based adhesives to address health risks associated with traditional resins and explore improvements in mechanical properties. Chitin is a nontoxic, biodegradable, biocompatible, antioxidative, antimicrobial, and thermally stable polymer [20]. It is the second most abundant natural polymer after cellulose and is commonly derived from the exoskeletons of crustaceans and arthropods [21-23]. According to the Food and Agriculture Organization's 2022 *State of World Fisheries and Aquaculture* report, approximately 5.83 million tonnes of live crustaceans are captured annually, generating up to 2.04 million tonnes of shell waste [24]. Due to its abundance and beneficial properties, chitin is increasingly explored for sustainable bio-based nanocomposites. This study aims to explore the effects of incorporating CNW into PE-based adhesives, focusing on how CNW may help mitigate health risks while potentially enhancing mechanical performance and thermal stability. By examining these effects, this paper aims to provide insights into CNW's viability as a safer, more sustainable adhesive additive.

II. MATERIALS AND METHODS

A. Materials

Commercial-grade PE resin (3M Bondo® Fiberglass Resin) was used as the base material in this study. Methyl ethyl ketone peroxide (MEKP, Bondo® Liquid Hardener 912C) was used as the curing agent. Chitin nanowhiskers (CNW), in the form of a 5 wt.% ethanol suspension, were obtained from Neptune Nanotechnologies Inc. (Markham, Canada). This bio-derived nanoparticle is used as the reinforcing additive to the PE-based adhesive.

B. Fabrication of PE/CNW Nanocomposites

A slurry compounding method was employed to fabricate PE/CNW nanocomposites at varying CNW loadings, ranging from 0.25 wt.% to 1.0 wt.%. The ethanol suspension of CNW was ultrasonicated for 30 minutes at 25 kHz to suppress agglomeration and clumping of CNW. Calculated amounts of the CNW-ethanol suspension were added to the PE resin to achieve the target loading of CNW in PE. The mixture of PE, CNW, and ethanol was mechanically stirred in a beaker at 85°C to evaporate the ethanol. The mixture was periodically weighed to determine the amount of ethanol removed. Moreover, the cessation of effervescence was used to indicate complete ethanol removal. After adding 2 wt.% of 3M Bondo® Liquid Hardener, which contains MEKP curing agent, to the neat or nanocomposite resin, the mixture was mechanically stirred and degassed at -80 kPa for 10 minutes. This process removed entrapped gas bubbles and suppressed the formation of gas voids upon curing. This procedure was in accordance with the specifications recommended by the PE resin manufacturer.

C. Fourier Transform Infrared (FTIR) Analysis

FTIR spectra of the neat PE, CNW, and PE/CNW nanocomposite samples were acquired by using an FTIR spectrometer (VERTEX 70v, Bruker) in the transmission mode over a scan range of 4000 to 400 cm^{-1}. The acquired FTIR spectra were used to determine the chemical structures of the samples. Cured samples were ground into powder and subsequently sieved using a 60-grade mesh. The samples were placed onto a clean attenuated total reflectance (ATR) crystal after nominal calibration and confirmation of the interferogram signal strength. The samples were then pressed against the ATR crystal to obtain the transmission spectra.

D. Adhesive and Tensile Testing

The shear strength of the neat PE adhesive and those of their PE/CNW counterparts used to bond aluminum substrates were measured according to the procedures described in ASTM D1002. Specimens were fabricated in a single-lap shear geometry by applying the cure-activated PE or PE/CNW nanocomposite mixture on two identical pieces of cold-rolled aluminum substrates, which were manufactured in accordance with the ASTM B209 standard. The aluminum substrates had a thickness of 1.6 mm and a width of 25.4 mm. Their bonding surfaces were treated using a precision wipe for surface modification. After the adhesive fully cured, thickness measurements were conducted at six positions (i.e., the two ends and the center of the adhesive on both sides of the bonded area) using a caliper. The average thickness of the adhesive layer was measured to be 1.1 mm. The length of the overlap section was 12.7 mm, and the processed samples were kept at room conditions for 72 hours prior to the single lap shear test. The test was performed by pulling the two ends of the adhered samples in tension until failure. Seven samples were tested for each CNW loading.

For tensile testing, Type IV dog-bone samples were fabricated according to the ASTM D638 standard. Neat PE or PE/CNW nanocomposite dog-bone samples were cast in a high-strength high-temperature silicone rubber mold and cured following the manufacturer's specifications. The samples were then sanded with 400-grit sandpaper to meet specifications and conditioned under standard room conditions (i.e., 23°C) for 72 hours as per ASTM D618. Similar to the single lap shear test, seven specimens per composition were tested at 5 mm/min using a mechanical tester (ElectroPuls E3000, INSTRON).

E. *Thermal Analyses*

Differential scanning calorimetry (DSC) was performed using the DSC Q20 from TA Instruments to determine the glass transition temperature of the cured PE and PE/CNW nanocomposite samples. Thermogravimetric analyzer (TGA) was used to analyze the thermal stability of the samples. To conduct the thermal analyses, cured samples were ground into powders and sieved using an 80-grade mesh. For DSC, the powder samples were placed in a Tzero® aluminum pan that was loaded into the DSC chamber. The samples were maintained at equilibrium at 35°C before a heating rate of 10°C/min was applied to heat the samples to 400°C. For TGA, the powder samples were placed onto a platinum pan, which was tared before analysis in a thermogravimetric analyzer (TGA 55, from TA Instruments). The samples were maintained at equilibrium at 35°C before a heating rate of 20°C/min was applied under a nitrogen purge to heat the samples to 600°C.

III. RESULTS AND DISCUSSIONS

A. *FTIR Analysis of Chemical Strucutres*

Fig. 5 indicates the FTIR spectra of CNW, neat PE, and PE/CNW nanocomposites with different CNW loadings. The FTIR spectrum of CNW shows an intense band between 1000 and 1075 cm^{-1} due to the stretch of C-O associated with primary alcohols. The secondary amine groups and the hydroxyl groups contributed to the intense bands around 3324 cm^{-1} and 3700 cm^{-1}, respectively. For neat PE, an intense band was observed at 1718 cm^{-1}, attributed to the stretching vibrations of the carbonyl group. The weak band at about 1460 cm^{-1} was associated with the aromatic ring in the backbone and the crosslink structures. The strong band at 1263 cm^{-1} resulted from the twisting vibration of CH$_2$ groups. The FTIR spectra of the PE/CNW nanocomposites exhibited characteristic bands for both CNW and PE, demonstrating their successful fabrication. Most importantly, a weak band around 1579 cm^{-1} was observed in the nanocomposites, and it became more pronounced with increasing CNW loadings. This band represents characteristic of the stretching of carboxylate groups, indicating that some degree of degradation might have occurred during the slurry compounding process used to prepare the nanocomposites [25].

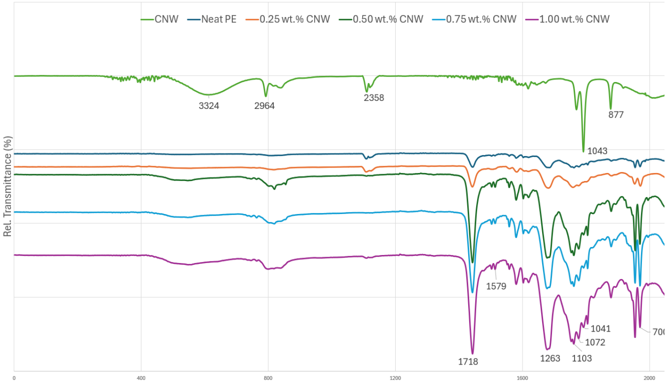

Figure 1. FTIR spectra of commercial polyester resin samples with incorporated CNWs, highlighting the characteristic functional groups.

B. *Mechanical Characterization*

Fig. 2 presents the tensile characteristics of neat polyester and their nanocomposites with different CNW loadings. The measured tensile strength of fully cured neat PE was 42.9 ± 4.7 MPa, which was comparable to the manufacturer's specified data of 40.0 ± 0.7 MPa. With the addition of CNW, the tensile strength increased to 47.6 MPa (i.e., +17.7%) and 50.6 MPa (i.e., +23.5%) when 0.25 wt.% and 0.50 wt.% of CNWs were added. This enhancement is attributed to the high aspect ratio and stiffness of CNW, which facilitate effective load transfer across the nanocomposites. The substantial improvement at lower concentrations (0.25 wt.% and 0.50 wt.%) suggests optimal interfacial bonding between the resin and the CNW when the bio-based nanoparticles are well-dispersed. However, further increases in CNW loading beyond 0.50 wt.% led to a detrimental

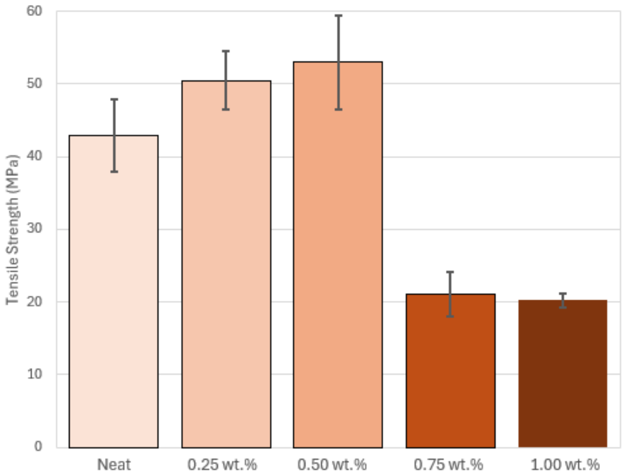

Figure 2. Tensile characteristics of commercial polyester resin samples with CNW incorporated.

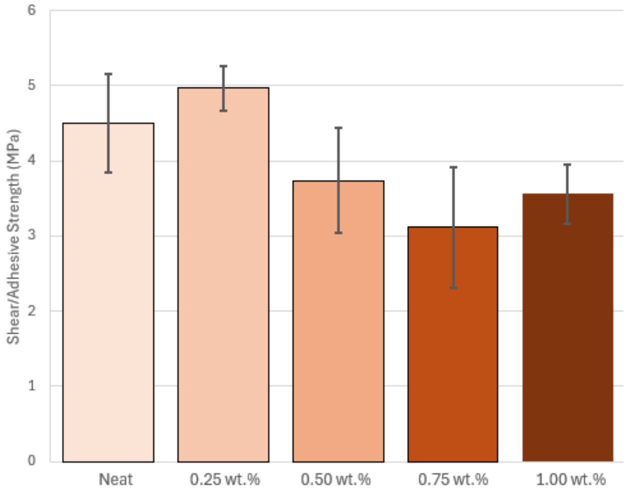

Figure 3. Shear strength of commercial polyester resin samples with CNW incorporated, determined by single-lap shear test.

effect on the tensile strength of the PE-CNW nanocomposites. It was observed that the addition of 0.75 wt.% of CNW led to a decrease of tensile strength to 18.2 MPa (-50.90%).

Two factors could contribute to this reduction in mechanical properties. Firstly, some degree of degradation of the PE resin might have occurred during the slurry compounding process as indicated in the FTIR spectra of PE/CNW nanocomposites. As the CNW loadings increased, a larger amount of ethanol suspension was used to prepare the nanocomposites. A low boiling point alcohol, such as ethanol, could contribute to a mild degradation of the PE backbone [26]. Secondly, higher loadings of CNW would likely be detrimental to their uniform dispersion in the PE matrix. The agglomeration of CNW would reduce their effective surface area, impairing the load distribution performance. In summary, these structural defects weaken the nanocomposites, leading to the observed decline in tensile strength. This indicates that beyond a critical concentration, the challenges of processing the PE/CNW nanocomposites and dispersing CNWs outweigh the reinforcing potential of the nanoparticles. Achieving uniform dispersion by suppressing agglomeration is therefore crucial for leveraging the mechanical benefits of CNWs in PE-based adhesives.

The single-lap shear test revealed that the failure mode of the adhesive was cohesive for all neat and CNW-reinforced samples, indicating that failure occurred within the adhesive layer rather than at the bond interface. This indicates that the adhesive strength at the interface exceeded the shear strength of the cured resin. It also suggests that the addition of CNWs did not compromise adhesive bonding with the aluminum substrates. The shear strength of the cured adhesive increased by 0.47 MPa (+10.4%) upon adding 0.25 wt.% CNW. This improvement is attributed to the enhanced load distribution and increased stiffness of the resin matrix. At this concentration, CNW likely achieved uniform dispersion, which enabled their reinforcing effects without introducing structural defects. However, higher CNW concentrations (0.50 wt.% or more) negatively impacted the resin's shear strength, showing a reduction of 0.76 MPa (-16.89%) at 0.50 wt.% CNW. This decline in shear strength at higher CNW concentrations mirrors the behavior observed in the tensile strength tests. This is attributed to the agglomeration of CNW, which disrupts the uniformity of the adhesive layer. Such agglomerates act as localized stress concentrators, weakening athe dhesive layer and compromising its ability to withstand shear forces effectively. The clustering of CNWs reduces their effective surface area and interferes with the resin's ability to form a homogenous, cohesive matrix. Moreover, the higher ethanol content required for preparing PE/CNW nanocomposites with increased CNW loadings led to mild degradation of the PE, ultimately diminishing its overall mechanical performance. These findings highlight the importance of achieving optimal CNW dispersion and exploring alternative suspension media to maximize their reinforcing potential in PE resin systems.

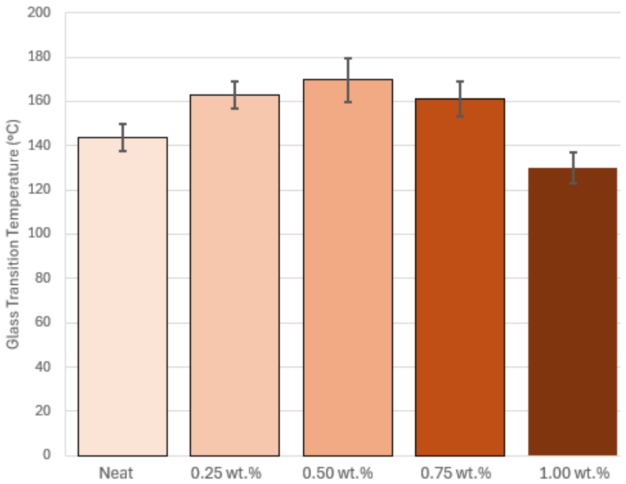

Figure 4. Glass transition temperatures of commercial polyester resin samples with CNW incorporated, determined by DSC.

C. *Thermal Analyses*

The glass transition temperatures (T_g) of neat PE and PE/CNW nanocomposites are shown in Fig. 4. It can be observed that the addition of CNW into the PE matrix resulted in an increase in T_g due to the suppressed mobility of the polymer chain in the presence of uniformly distributed nanoparticles. However, as the CNW loadings increased further to 0.75 wt.% and 1.00 wt.%, the nanocomposites' T_g decreased. This could be attributed to the formation of CNW agglomerates

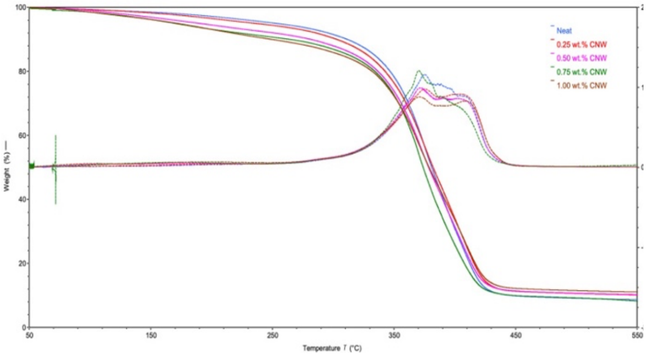

Figure 5. Thermogravic analysis of the commercial polyester resin samples with CNW incorporated using TGA.

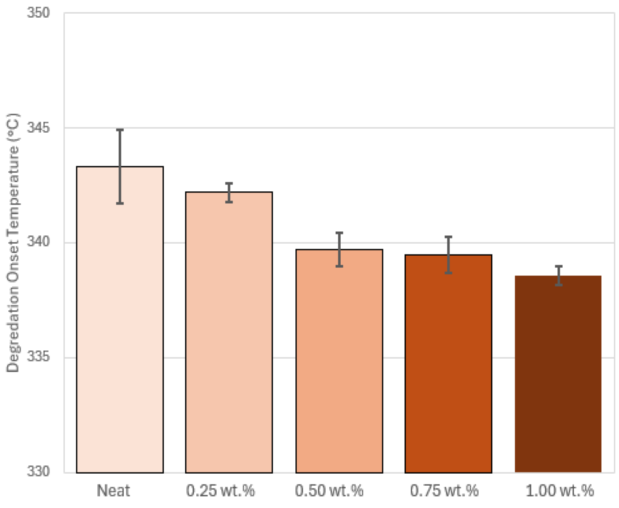

Figure 6. Degredation onset temperatures of commercial polyester resin samples with CNW incorporated, determined by TGA.

at higher CNW loadings, suppressing the ability of the nanoparticles to hinder the polymer chain mobility. More importantly, the T_g of the nanocomposite loaded with 1.00 wt.% CNW was lower than that of the neat PE sample. This can be explained by the promoted PE degradation due to the presence of a larger amount of ethanol.

Fig. 5 illustrates the weight loss over time and the corresponding rate at which it happened for neat PE and the PE/CNW nanocomposites. The onset temperatures of thermal degradation of the samples are shown in Fig. 6. A significant decrease in the onset temperature of thermal degradation was observed as CNW loadings increased, particularly at 0.50 wt.% or higher. Thus, dispersing CNW in the PE matrix appears to negatively impact the thermal stability of the resin. This is likely due to the increased use of ethanol when preparing PE/CNW nanocomposites with higher CNW loadings. Furthermore, the intrinsic properties of CNWs, which contain functional groups like hydroxyl, may lead to decomposition at lower temperatures, releasing volatile byproducts such as water molecules. These volatiles may exacerbate PE resin degradation by creating pathways for heat transfer and localized structural weaknesses. The water molecules may also interact with the resin, undergoing hydrolysis reaction that results in localized breakdown and weaken the matrix.

IV. CONCLUSION

The addition of bio-derived chitin nanowhiskers (CNW) into polyester (PE) resin demonstrates significant potential for enhancing mechanical properties, particularly tensile and adhesive strengths. Low CNW concentrations (0.25 wt.% and 0.50 wt.%) yielded notable improvements, increasing tensile strength by up to 23.5% and adhesive strength by 10.4%. These improvements were attributed to uniform CNW dispersion, enabling effective load transfer. However, higher CNW concentrations (⩾0.75 wt.%) led to agglomeration, resulting in structural defects. Moreover, at higher CNW loadings, the presence of larger amounts of ethanol during the slurry compounding process introduced mild hydrolysis of PE, suppressing both tensile and shear strengths. This was evident from the more pronounced 1579 cm^{-1} bands (i.e., characteristic of carboxylate groups) in the FTIR spectra of PE/CNW nanocomposites, particularly those with higher CNW loadings. Similarly, thermal characterization revealed that high CNW loadings decreased the resin's thermal stability. Overall, this study highlights the potential of CNW to enhance the mechanical properties of PE resins, thereby narrowing the performance gap with epoxy resins. Additionally, CNW integration could help reduce reliance on styrene by improving resin performance without requiring excessive styrene content. By addressing challenges such as agglomeration and choice of suspension medium for CNW, CNW-reinforced PE resins might offer a more sustainable, safer, and high-performance solution for advanced applications. These resins may serve as competitive alternatives to traditional epoxy systems.

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