

Research Article

Adhesive and Impact-Peel Strength Improvement of Epoxy Resins Modified with Mono and Diamine Functionalized Elastomers

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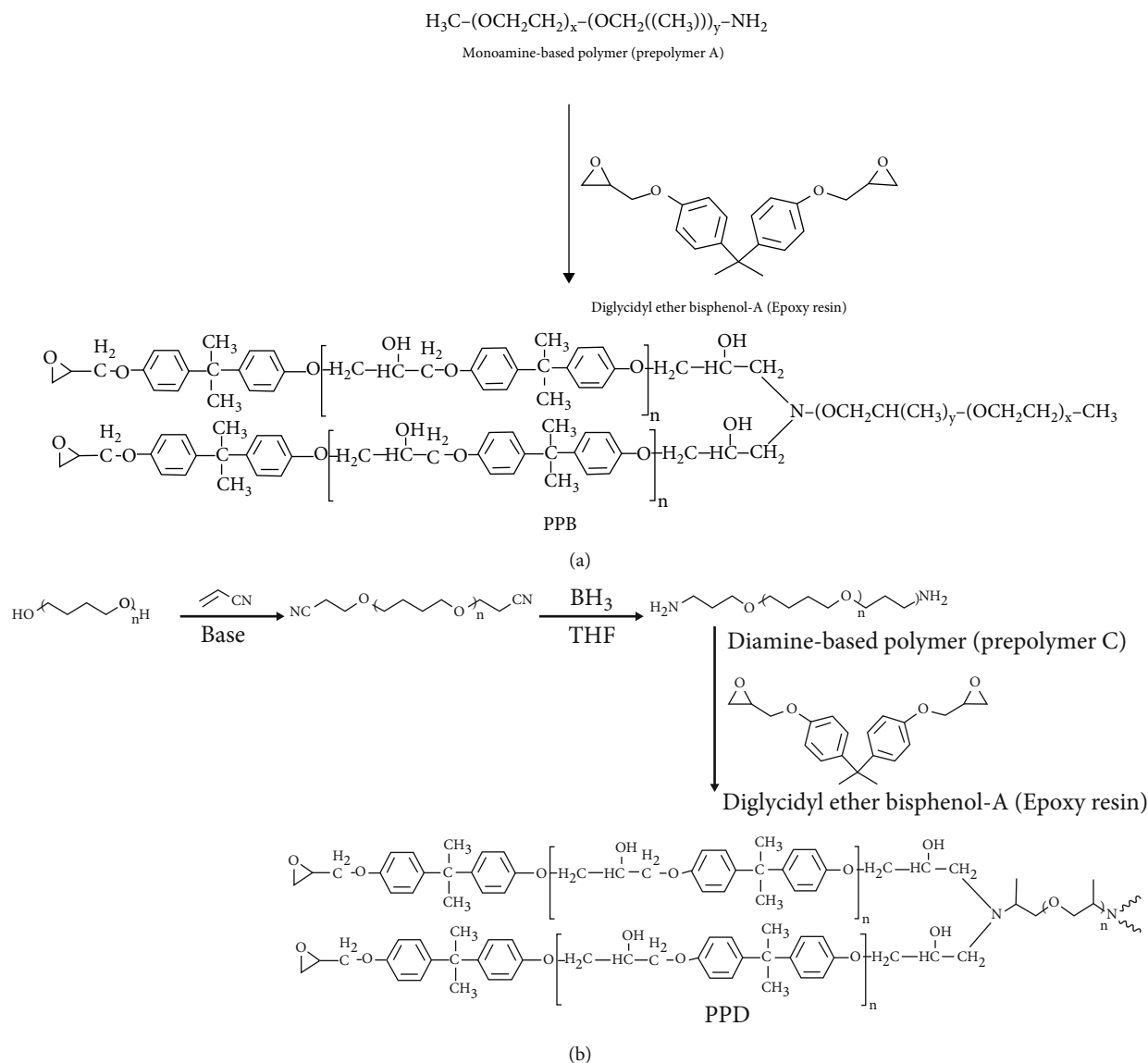
Epoxy resins are widely applied in the automotive and electronic industries. However, pure epoxy resins are brittle and thus possess poor mechanical properties. Herein, we report a facile method for improving the impact-peel and adhesive strengths of epoxy resins via the incorporation of two different types of polyether amines (monoamine-based and diamine-based prepolymers). A comparative study was performed to investigate the potential advantages of incorporating a prepolymer into an epoxy resin matrix. It was discovered that the incorporation of a diamine prepolymer significantly improved the impact-peel strength of the epoxy resin system at low (-40°C) and room (23°C) temperatures. For 15 wt% adhesive loading, the diamine prepolymer-based epoxy system demonstrated a 130% (low temperature) and 32% (room temperature) higher impact-peel strength than the monoamine prepolymer-based epoxy system. Moreover, the 15 wt% diamine prepolymer-based epoxy system exhibited a significantly improved shear strength ($\sim 36\text{ MPa}$) and T-peel strength (260 N/25 mm) owing to the effectively reduced crack propagation and cohesive interactions between the epoxy molecules. Our results suggest that the modification of epoxy resins with an appropriate amount of mono and diamine-functionalized elastomers provides a novel route for the development of highly efficient adhesive materials for various applications.

1. Introduction

Epoxy resins have received considerable attention from the scientific and industrial communities owing to their excellent mechanical strength, superior electrical resistance, and versatile processability [1–3]. Epoxy resins have found several important applications in structural adhesives, high-performance composites, high-tension electrical insulators, and the aerospace industry [4–7]. Unfortunately, epoxy resins typically possess the following drawbacks: long curing time, low impact resistance, poor toughness, and inherent brittleness of the cured resin [8–12]. In recent years, numerous studies have focused on the improvement of the adhesive strength, fracture toughness, and impact resistance of epoxy resins by introducing inorganic nanoparticles (carbon nanotubes (CNTs) and nanofibers), organic nanoparticles (core-shell elastomeric particles and polyhedral-oligomeric-

silsesquioxane), nanosized resinous particles (thermoplastics), and synergistic blends of nanosized particles [13–17].

Improving the toughness of epoxy resins by the addition of elastomeric particles is an effective approach; however, it reduces the thermal stability and increases the viscosity of the final product, which restricts the use of modified epoxy resins in certain practical applications [18]. The incorporation of CNTs at low levels enhances the mechanical and thermal properties of epoxy resins [19]. However, their dispersion and interfacial interaction with epoxy resins are very poor [20]. In this direction, several novel modification methods have been developed in the adhesive industry to improve the physical, thermal, and mechanical properties of epoxy resins. In recent studies, polyether amines have gained increasing attention in the epoxy research field as excellent curing agents offering various advantages [21], such as a good temperature stability, flexibility, toughness, and reactivity for epoxy resins [22, 23].



SCHEME 1: Synthetic routes of (a) PPB and (b) PPD.

The aim of the present study was to evaluate and compare the effects of incorporation of polyether amines, such as mono-prepolymers (polymers containing one amino group) and di-prepolymers (polymers containing two amino groups), as elastomeric additives for diglycidyl ether bisphenol-A (YD-128) at different weight percentages (wt%). The mechanical properties of the modified epoxy systems were investigated in detail using various characterization techniques. Mono- and diamine-based functional elastomers are known to exhibit exact variations in their backbone structures because the number of substituted amines is different. Experimental studies revealed that the addition of a diamine-based prepolymer increased the impact-peel strength of the epoxy resin. The diamine prepolymer-incorporated epoxy system exhibited a 130% improvement in the impact-peel strength at low temperatures compared with the mono-prepolymer-incorporated epoxy system. Moreover, the diamine prepolymer-based

epoxy system demonstrated an excellent T-peel strength of 260 N/25 M, which is 67% higher than that exhibited by the neat epoxy (130 N/25 M).

2. Experimental

2.1. Materials. Mono-prepolymer (monoamine-based polymer) (A1), trifunctional primary amine (TPA) T-403 (Huntsman), and di-prepolymer (diamine-based prepolymer; self-prepared) (A2) were used as essential additives; diglycidyl ether bisphenol-A (A3) (YD-128, Kukdo Chemical) was used as an epoxy resin; dicyandiamide (DICY) was used as a curing agent; 1-cyanoethyl-2-(4-methylimidazole) (2E4MZ-CNS) was used as a catalyst; [2,2-bis[(2-propenyloxy)methyl]-1-butanolato-O, O', O'']zirconium was used as an adhesion promoter; polytetramethylene ether glycol (PTMEG 1000), 15-crown-5, sodium hydride, acrylonitrile, hydrochloric acid, dichloromethane (DCM), dry tetrahydrofuran (THF), borane-tetrahydrofuran

complex, methanol, sodium hydroxide, and sodium sulfate were obtained from Sigma-Aldrich.

2.2. Modification of Epoxy Using a Monoamine-Based Prepolymer. Step 1. Preparation of prepolymer B (PPB) using an epoxy resin and a monoamine-based prepolymer (Prepolymer A) (A1).

In this reaction step, the epoxy resin was mixed with prepolymer A and TPA at an equivalent ratio of 1:0.3:0.05. The addition of a small amount of TPA enhanced the flexibility and stability of the reaction. Initially, the epoxy resin was heated at 130°C for 30 min to dissolve the polyether amines. Subsequently, it was mixed with prepolymer A and TPA. The obtained products are denoted as PPB. The PPB synthesis route is illustrated in Scheme 1(a).

Step 2. Preparation of cured epoxy using PPB.

From the above reaction, different weight percentages (5, 10, and 15 wt%) of PPB were added to neat epoxy with a curing agent, DICY, and a catalyst, 2E4MZ-CNS (wt% represents the net weight of the monoamine-based polymer without the epoxy). [2,2-bis[(2-propenyloxy)methyl]-1-butanolato-O, O', O'']zirconium was used as an adhesion promoter to prepare cured epoxy systems. The flexible epoxy resin was mixed with a curing agent at an equivalent ratio of 1:0.85, as well as an accelerator (1.0 phr), SiO₂ nanosized particles (5.0 phr), and adhesion promoter (0.2 phr). All epoxy blends were cured at 180°C for 30 min. The final products with different wt% were denoted as Epo/PPB 5 wt%, Epo/PPB 10 wt%, and Epo/PPB 15 wt%, based on their treatment procedures.

2.3. Modification of Epoxy Using a Diamine-Based Prepolymer. Step 1. Preparation of diamine-based prepolymer (prepolymer C) (A2).

Polytetramethylene ether glycol (PTMEG 1000; 1.9 g; 2.00 mmol) and 15-crown-5 (8.8 mg; 0.04 mmol) were added to acrylonitrile (8 mL) and stirred for 15 min in an ice bath. Subsequently, sodium hydride (1.6 mg 60% dispersion in mineral oil; 0.04 mmol) was added to the mixed solution and stirred at 0°C for approximately 25 min. A drop of hydrochloric acid was then added to quench the reaction, and the solution was filtered and concentrated under a low vacuum. The left-over residue was added to 10 mL dichloromethane and centrifuged at 6500 rpm. The resulting product was drained, filtered, and concentrated under a low vacuum. A slightly yellow crystalline powder was obtained.

The product obtained from the above reaction was dissolved in dry THF (30 mL) and added to a solution containing a borane-tetrahydrofuran complex (8 mL of 1 M solution in THF, 8.0 mmol) and dry THF (50 mL) at 0°C. It was mixed well for 40 min and heated to reflux for 4 h. Thereafter, it was cooled to 0°C, and 8 mL of methanol was added to it. Subsequently, HCl (0.4 mL) was added to quench the reaction; the solution was stirred for 1 h and was completely evaporated under a low vacuum. A slightly yellow, viscous liquid was obtained. A 30 mL NaOH solution was added to the obtained product and extracted four times with diethyl ether (40 mL). The organic layers in the solution were dried with Na₂SO₄ and filtered. The remaining solvent was then

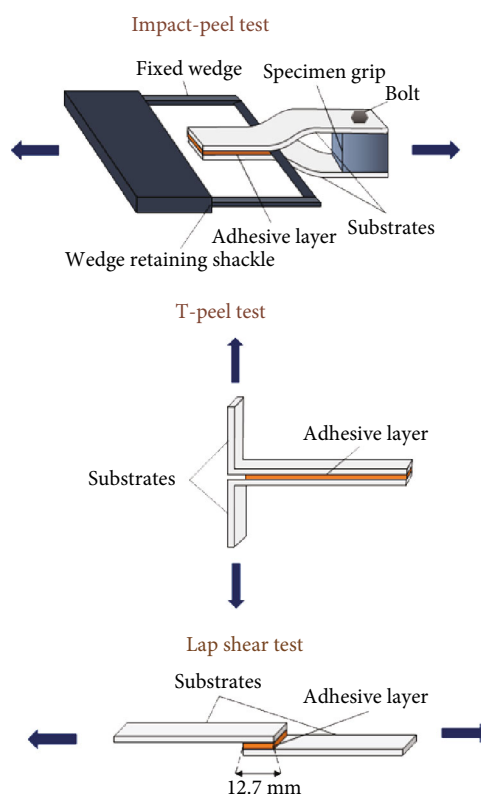


FIGURE 1: Specimen setup for impact-peel test, T-peel test, and lap shear test.

evaporated. After evaporation, a white powder (prepolymer C) (A2) was obtained as a product.

Step 2. Synthesis of prepolymer D using epoxy resin and prepolymer C.

The epoxy resin was mixed with prepolymer C and TPA at an equivalent ratio of 1:0.85:0.35. Initially, the epoxy resin was heated at 130°C for 30 min to dissolve the polyether amines. Thereafter, it was mixed with prepolymer C and TPA. The obtained product was denoted as prepolymer D (PPD). The PPD synthesis route is depicted in Scheme 1(b).

Step 3. Preparation of cured epoxy using PPD.

From the above reaction, different weight percentages (5, 10, and 15 wt%) of PPD were added to neat epoxy with a curing agent, DICY, and a catalyst, 2E4MZ-CNS (wt% represents the net weight of the diamine-based polymer without the epoxy). [2,2-bis[(2-propenyloxy)methyl]-1-butanolato-O,O',O''] was used as an adhesion promoter to prepare cured epoxy systems. The flexible epoxy resin was mixed with a curing agent at an equivalent ratio of 1:0.85, as well as an accelerator (1.0 phr), SiO₂ nanosized particles (5.0 phr), and an adhesion promoter (0.2 phr). All epoxy blends were cured at 180°C for 30 min. The final products with different wt% were denoted as Epo/PPD 5 wt%, Epo/PPD 10 wt%, and Epo/PPD 15 wt%, based on their treatment procedures.

2.4. Characterization. The lap shear and T-peel tests for each system were conducted according to ASTM D-100 using a universal testing machine (Tinius Olsen 25ST). The

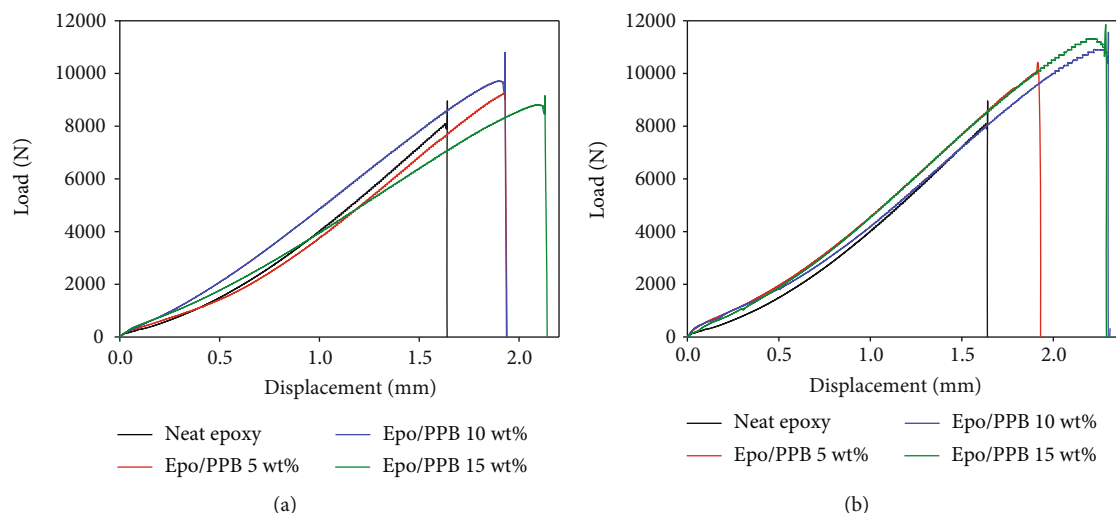


FIGURE 2: Load-displacement curves obtained in the lap shear test for (a) Epo/PPB and (b) Epo/PPD with different wt%.

impact-peel tests were performed using a high-strength steel (SPRC440) plates (specimens) and an Instron CEAST 9350 testing machine. The schematics for the impact-peel, T-peel, and lap shear tests are presented in Figure 1.

3. Results and Discussion

The load versus displacement curves of the lap shear tests for the Epo/PPB and Epo/PPD samples with different wt% are illustrated in Figure 2. The lap shear strength can be evaluated based on these curves by dividing the critical fracture load by the product of the lap joint length and width. The lap shear strength provides information regarding the ability of an adhesive to resist forces in the plane of the bonded surfaces. The shear strength of the neat epoxy was calculated to be ~ 24 MPa. After the incorporation of PPB, a gradual increase in the lap shear strength is observed (Figure 2(a)). Among the PPB-based epoxy resins, 10 wt% Epo/PPB exhibits the highest shear strength of ~ 30 MPa. This improvement can be attributed to the lower stress concentration zones in the epoxy material owing to the incorporation of a particular amount of PPB. In contrast, Epo/PPD 15 wt% exhibits a shear strength of ~ 36 MPa at room temperature, which is 19% higher than that exhibited by Epo/PPB 10 wt% (Figure 2(b)). This significant improvement in the peak load of Epo/PPD 15 wt% might be attributed to the higher interaction of PPD with the epoxy adhesive owing to the high aspect ratio [24]. Moreover, it provided a larger surface area to boost the load-transfer efficiency. A comparison plot of the lap shear test results for the PPB and PPD epoxy resins is presented in Figure 3; evidently, the incorporation of the diamine-based prepolymer in the epoxy resin results in a greater improvement in the shear strength than the addition of monoamine-based prepolymers. It can be seen that the lap shear strength of Epo/PPB decreases with the increase of PPB content. This could be due to the reduction of bond strength and low hardness between epoxy and PPB.

The adhesion strength is a significant factor in estimating the superiority of epoxy layers. T-peel strength tests were conducted to investigate the adhesion strength of the modi-

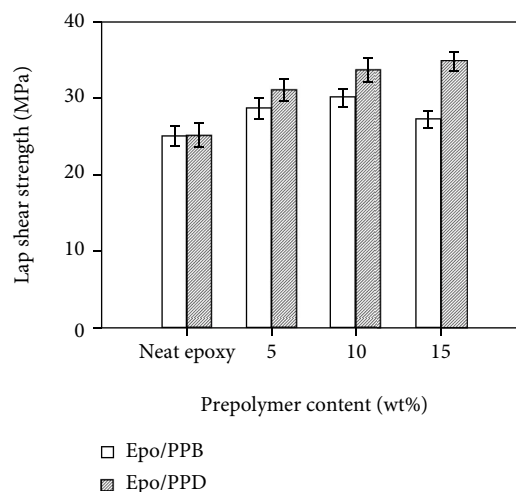


FIGURE 3: Comparison plot of lap shear strength between PPB and PPD incorporated epoxy resins using different wt%. Error bars show standard deviation in each data set.

fied epoxy resins at room temperature. This test offers insights into the cohesive strength and crack-blocking ability of epoxy resins. Moreover, it provides the environmental durability of adhesively bonded systems for optimization in processes and applications. Figure 4 displays the T-Peel strength—displacement curves for the epoxy resins with their matrices modified by PPB and PPD. As depicted in Figures 4(a) and 4(b), the neat epoxy and PPB-based epoxy resins exhibit a linear tendency after a particular displacement, with some minor fluctuations. The neat epoxy resin dissipated more impact energy owing to the presence of undesirable microcracks and high microcrack line concentrations. However, the incorporation of the monoamine-based prepolymer into the epoxy layers endowed it with the ability to resist crack propagation through the bulk resin and improved the structure by absorbing the external impact stress (Figure 4(a)). The T-peel adhesive strength of the neat epoxy is approximately 130 N/25 M. However, Epo/PPB

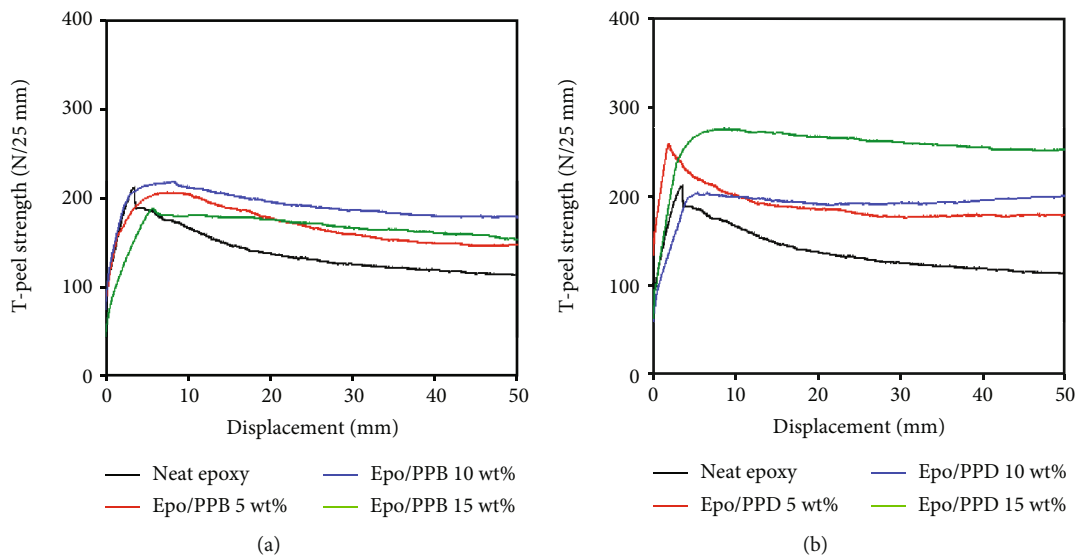


FIGURE 4: T-peel strength-displacement curves for (a) Epo/PPB and (b) Epo/PPD with different wt%.

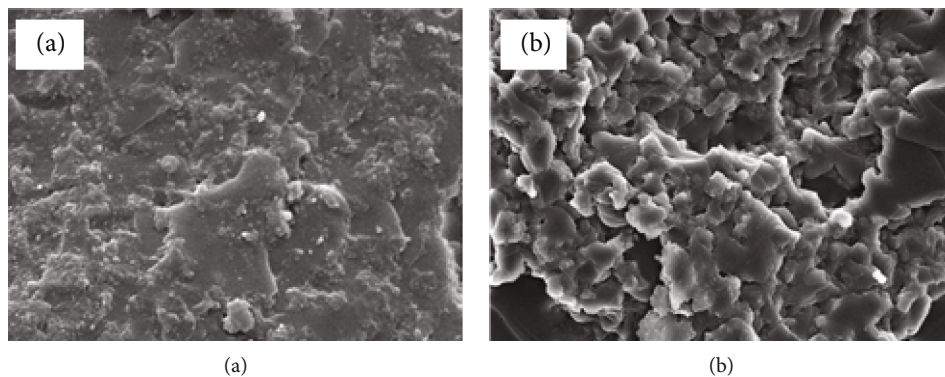


FIGURE 5: Fracture surfaces of epoxy resin; (a) less toughened (Epo/PPB 10 wt%) and (b) fully toughened (Epo/PPD 15 wt%).

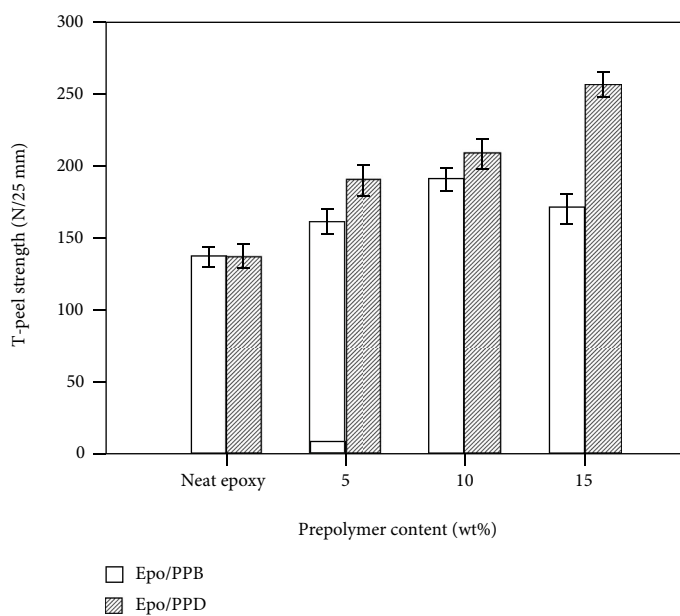


FIGURE 6: Comparison plot of T-peel strength between PPB and PPD incorporated epoxy resins using different wt%. Error bars show standard deviation in each data set.

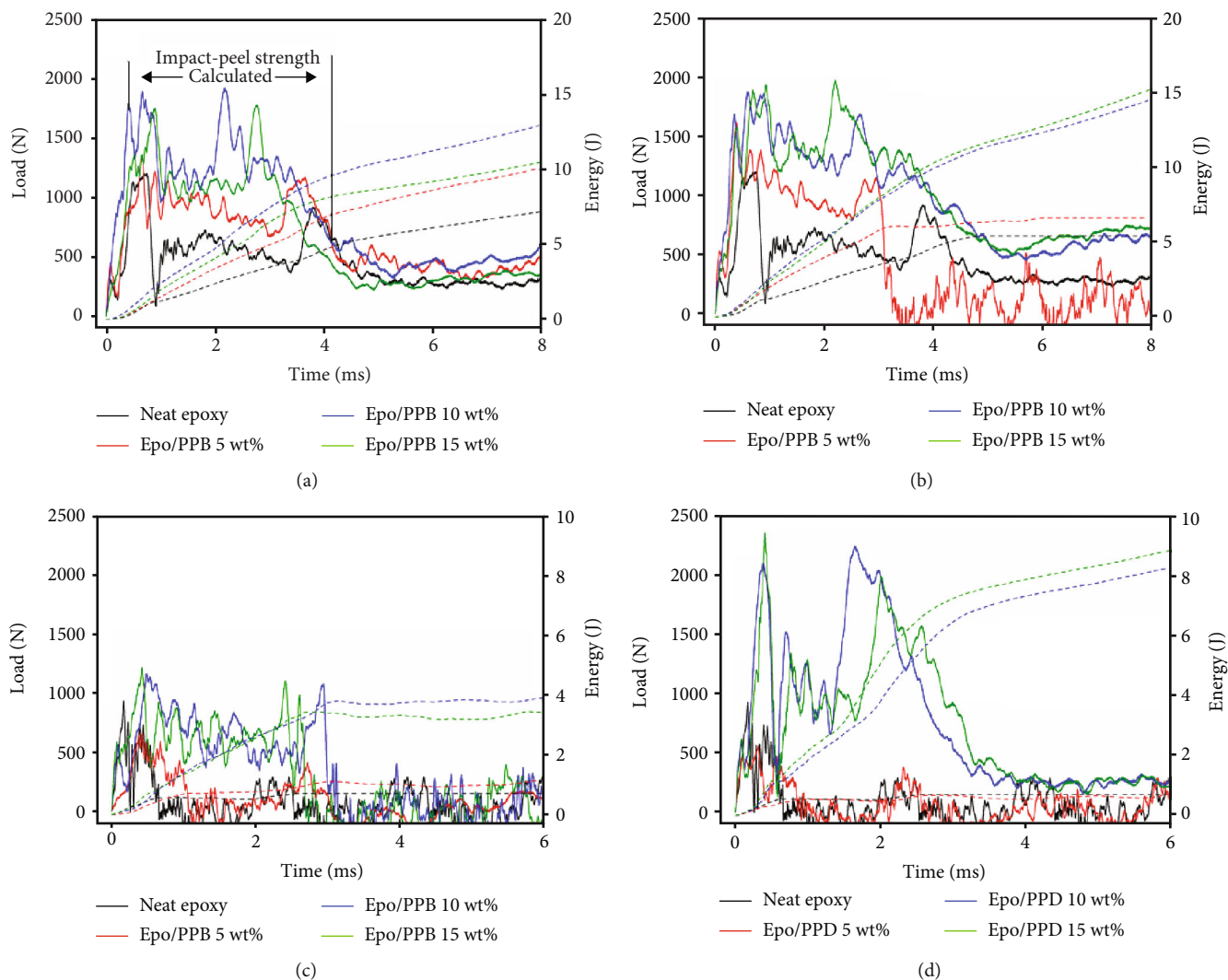


FIGURE 7: Impact load and impact energy vs. time curves of (a) Epoxy/PPB at 23°C, (b) Epoxy/PPD at 23°C, (c) Epoxy/PPB at -40°C, and (d) Epoxy/PPD at -40°C.

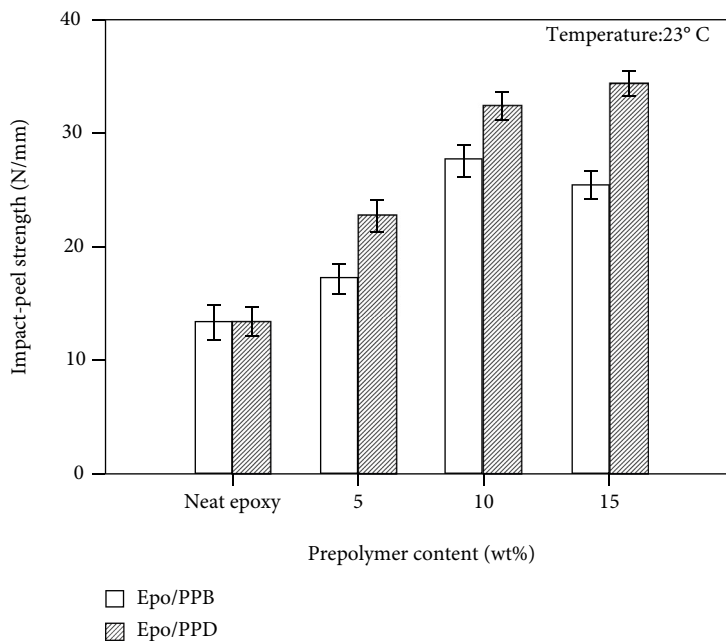
10 wt% exhibits a significant T-peel strength of 190 N/25 M, which is 38% higher than that of neat epoxy. All other modified epoxy resins also demonstrate an improvement in the T-peel strength after the incorporation of the monoamine-based prepolymer. However, PPB-based epoxy resins were found to exhibit brittleness and crack sensitivity after their synthesis. This degraded the quality of the resins for further applications. The fracture toughness test (three-point bending test) was used to determine the brittleness of the prepared materials as shown in Figure S1. Fracture energy and fracture surface indicate that the epoxy resin was effectively toughened by incorporating flexible prepolymer into epoxy resin. The fracture energy measured by three-point bending test ranged from 1039.6 to 1472.2 J/m².

Figure 4(b) demonstrates the T-peel strength—displacement curves for the epoxy resins with PPD at room temperature. The T-peel strength of Epo/PPD 15 wt% (260 N/25 M) is higher than that of the other three samples and 67% higher than that of neat epoxy (130 N/25 M). This remarkable improvement in the T-peel strength provided an excel-

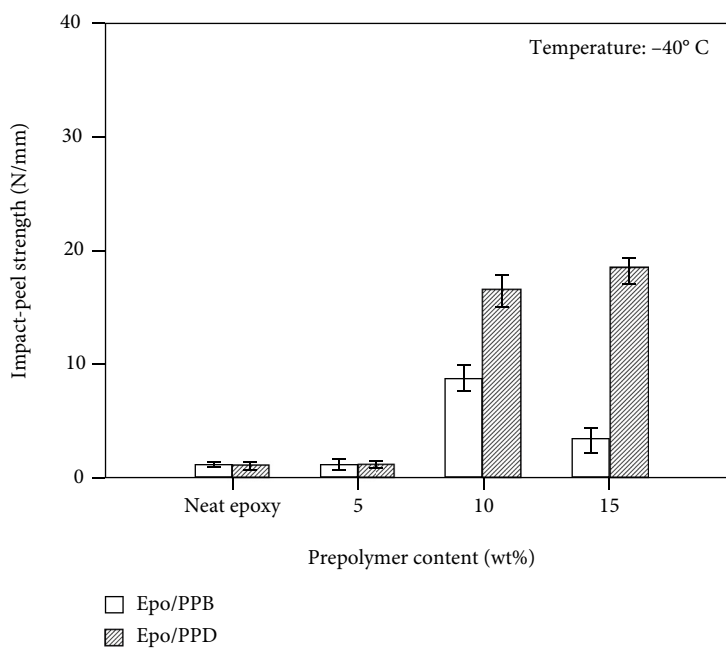
lent network structure and flexible properties for Epo/PPD 15 wt%. The introduction of PPD into the epoxy resin matrices could effectively block microcrack propagation and reduce the cohesive interaction between the polymer molecules, resulting in an enhanced blend toughness as shown in Figure 5.

Figure 6 depicts a comparison plot of the T-peel strength between the PPB- and PPD-based epoxy resins. The experimental plots indicate that the PPD-based epoxy resins exhibit a significantly higher T-peel strength than the PPB-based epoxy systems at all wt% (31% improvement in T-peel strength). Therefore, PPD-based epoxy resins with high T-peel strengths are perfect alternative materials for the production of automotive body structures and adhesives. The peel strength of Epo/PPB suddenly decreased with the increase of polymer content (15 wt%). The increase in modulus arising from epoxy polymers with PPB component resulted in a lower peel strength.

The impact load and impact energy versus time of epoxy/PPB and epoxy/PPD at 5, 10, and 15 wt% were



(a)



(b)

FIGURE 8: Comparison of impact-peel strength of Epoxy/PPB and Epoxy/PPD at (a) 23°C and (b) -40°C. Error bars show standard deviation in each data set.

measured at 23 and -40°C. The results are summarized in Figure 7. The impact-peel strengths of the two systems at different temperatures are compared in Figure 8. The impact-peel strength was determined by calculating the area under the load curve. The measured values of the peel strength and impact resistance provided additional details on the impact-peel resistance of the epoxy materials. In the PPB-based epoxy systems (Figures 7(a) and 7(c)), the Epo/PPB 10 wt% exhibits the highest peak load value among all the other samples. In contrast, Epo/PPD 15 wt% demon-

strates an excellent impact-peel strength and impact energy in the epoxy/PPD system for both temperature conditions (Figures 7(b) and 7(d)). The improved performances of Epo/PPB 10 wt% and Epo/PPD 15 wt% indicate that the cohesive interaction inside the epoxy matrix significantly decreases after the incorporation of an appropriate amount of monoamine and diamine-based prepolymers. Furthermore, the addition of these two materials enhances the energy absorption of the epoxy resin by hindering the development of new microcracks. However, Epo/PPB 10 wt% was

found to exhibit a tendency for minor crack initiation and easy propagation at certain points, which may affect the performance of the epoxy materials. As depicted in Figure 8(a), Epo/PPD 15 wt% exhibits a significantly greater impact strength at 23°C than Epo/PPB 10 wt%. Among the 15 wt% samples, the impact strength of Epo/PPD 15 wt% was greater than that of Epo/PPB 15 wt% by approximately 32% owing to good dispersion of PPD in the epoxy matrix. This implies that PPD incorporation is an effective strategy for producing highly efficient epoxy resins with a high impact-peel strength and impact energy. Figure 8(b) presents a comparison for the impact strengths of both samples at -40°C. Neat epoxy and 5 wt% samples demonstrate almost a zero impact-peel strength at -40°C. However, Epo/PPD 15 wt% exhibits a superior impact strength, which is 130% higher than that of Epo/PPB 15 wt%. The overall results of the impact test suggest that the preparation of epoxy resins with a particular amount of diamine-based prepolymers allows the attainment of high impact-peel strengths and impact resistances under normal and low-temperature conditions.

4. Conclusions

The primary objective of this study was to investigate and compare the improved adhesive properties of modified epoxy resins incorporated with monoamine-based PPB and self-prepared diamine-based PPD. The Epo/PPD 15 wt% epoxy system exhibited the highest T-peel strength among the tested modified epoxy systems (67% greater than that of neat epoxy and 31% greater than that of Epo/PPB 10 wt%). The impact-peel test revealed that the addition of 15 wt% PPD significantly reduced the cohesive interaction and hindered the crack propagation in the epoxy matrix. In addition, the impact resistance of the adhesive system was found to significantly depend on the content of the incorporated prepolymers. Epo/PPD 15 wt% demonstrated superior impact strengths at low and room temperatures, which were 130 and 32% higher, respectively, than those of Epo/PPB 15 wt%. The lap shear tests confirmed strong interactions between the diamine-based prepolymer and epoxy adhesive: the resin system exhibited an excellent shear strength of ~36 MPa. Thus, overall, it can be concluded that the highly efficient PPD-incorporated epoxy resin developed in this work is suitable for various future adhesive applications owing to its exceptional peeling behavior.

Data Availability

The data used to support the findings of this study are available from the corresponding author upon request.

Conflicts of Interest

The authors declare no competing financial interests regarding the publication of this study.

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Supplementary Materials

Figure S1: schematic diagram of fracture toughness test (three-point bending test). (*Supplementary Materials*)

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