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Research Article

Influence of Maleinized Polybutadiene on Adhesive Strength and Toughness of Epoxy Resins

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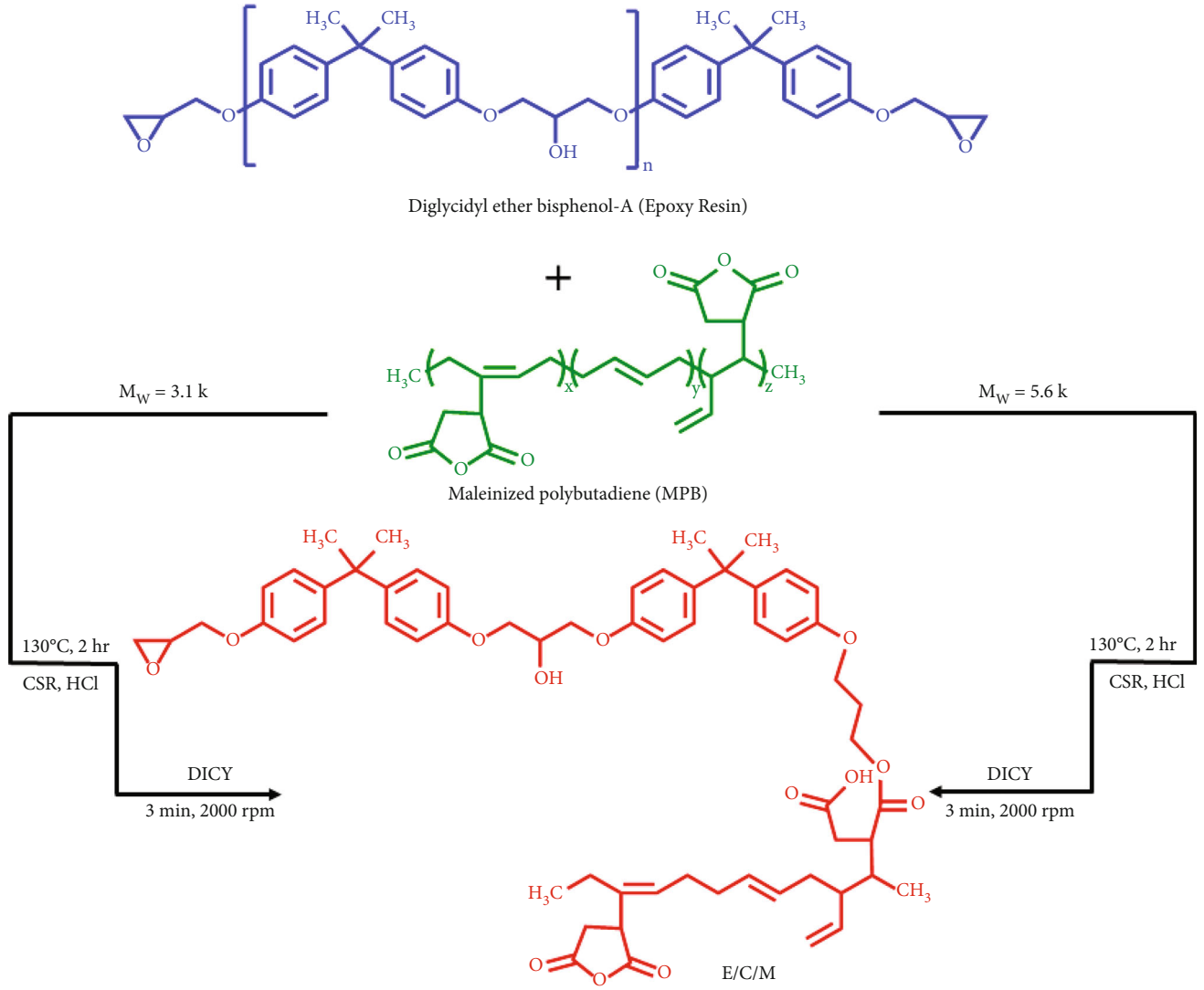
This study explored the effect of maleinized polybutadiene (MPB) on the mechanical properties of epoxy resins. Diglycidyl ether of bisphenol-A, an epoxy resin, was modified by incorporating MPB having different molecular weights in order to improve the fracture toughness and peel strength. MPB was mixed with epoxy resin at several concentrations (5, 10, and 15 phr), with the epoxy resin as the major phase and MPB as the minor phase. A comparative study was performed to investigate the influence of MPB on epoxy resins based on their molecular weight difference. Lap shear test results showed that the shear strength of the MPB-modified epoxy resins was superior to that of the neat epoxy resin. At 10 wt% MPB loading, the modified epoxy resin exhibited an 87% enhancement in T-peel strength relative to that of the neat epoxy resin. Moreover, the fracture energy of the modified epoxy system increased proportionally with the amount of MPB in the epoxy matrix. These results indicate that MPB incorporation is a simple and effective method for designing multifunctional epoxy resins, thus facilitating their industrial application in various spheres.

1. Introduction

Epoxy resins are high-temperature thermoset polymers with versatile characteristics, such as superior adhesion, low residual stress, high tensile strength and modulus, low shrinkage during curing, high chemical resistance, and excellent mechanical and dielectric properties [1–4]. These resins are used in satellite systems, high-strength adhesives, molding materials, biomedical systems, electrical laminates, and coatings [5–8]. However, some of their drawbacks are crack sensitivity, poor toughness, low fracture energy, brittleness, and low impact resistance [9–11]. Hence, in recent years, there have been concerted efforts to modify epoxy thermosets for improving their toughness and impact resistance. Researchers have developed efficient methods for improving the toughness of epoxy resins by adding core–shell polymers, inorganic nanoparticles, carbon black, and rubber particles [12–14].

Modification of epoxy resins by incorporating rubber into their matrix is an effective strategy to improve their fracture toughness and shear strength. Becu et al. studied the effects of the addition of core–shell rubber (CSR) particles into the epoxy system and found significant improvement in fracture toughness and impact strength of epoxy polymers by the addition of CSR particles up to 24 vol% [15]. In another study, Chen et al. conducted research on a diglycidyl ether of bisphenol-F epoxy system modified by the addition *n*-butyl glycidyl ether and obtained enhanced shear strength with the modification [16]. Kumar and Kothandaraman reported the dispersion of maleated depolymerized natural rubber in epoxy resins for improving their shear strength, fracture toughness, and impact resistance [17]. Maleinized polybutadiene (MPB) is the best modifier for enhancing the mechanical properties of epoxy resins owing to its good compatibility, excellent hydrophobicity, and exceptional reinforcing properties [18, 19]. MPB has a variety of applications in the electronics, automotive, and coating industries because of its excellent chemical and physical properties [20, 21]. Besides MPB, CSR nanoparticles play a significant functional role in improving the



Scheme 1: Synthetic routes of MPB-modified epoxy resin (epoxy/CSR/MPB).

toughness of brittle epoxy resins without sacrificing the glass transition temperature (T_g) of epoxy materials. CSR consists of a rubbery core and a thin layer of a glassy shell [22].

Although several strategies have been reported to improve the mechanical properties of epoxy polymers, there are only a few studies on their modification using MPB. The main goal of the present work was to investigate the influence of MPB as a modifier on the mechanical properties of cured diglycidyl ether of bisphenol-A (DGEBA) and an epoxy/CSR/MPB (E/C/M) blend. MPB additives with two molecular weights (3.1 and 5.6 K) and different concentrations were used to compare the performance after epoxy modification. The effect of MPB on the mechanical properties of the modified epoxy resins was analyzed by lap shear and T-peel tests. Experimental studies suggested that changes in the molecular weight and concentration of the MPB modifier alter the mechanical performance of the epoxy resin. The shear strength, fracture energy, and adhesion strength of the epoxy resin were enhanced after the incorporation of an appropriate amount of MPB. The MPB–epoxy curing process also improved the interfacial interaction between the MPB–epoxy filler and the epoxy resin matrix, enhancing the mechanical properties of the modified system. Moreover, fraction energy measurements indicated strong dispersion of MPB in the epoxy matrix, which strongly influences each network. The epoxy resin modified with MPB (3.1 K, 10 phr) showed a 56% improvement in shear strength compared to the neat epoxy resin.

2. Experimental

2.1. Materials. Liquid epoxy resin YD 128 (Kukdo Chemical Co., Ltd., **Seoul**, South Korea) was used as the main chain of the structural adhesive DGEBA with an epoxide equivalent weight of 185 g/eq. MPB (Ricon 131MA20) with an anhydride equivalent weight of 490 was purchased from Kangshin Industrial Co., Ltd., Seoul, South Korea. Dicyandiamide (DICY) obtained from Merck (Seoul, South Korea) was used as a curing agent with its purity exceeding 99%. 2E4MZ-CNS (1-cyanoethyl-2-4-methylimidazole) was used as the catalyst

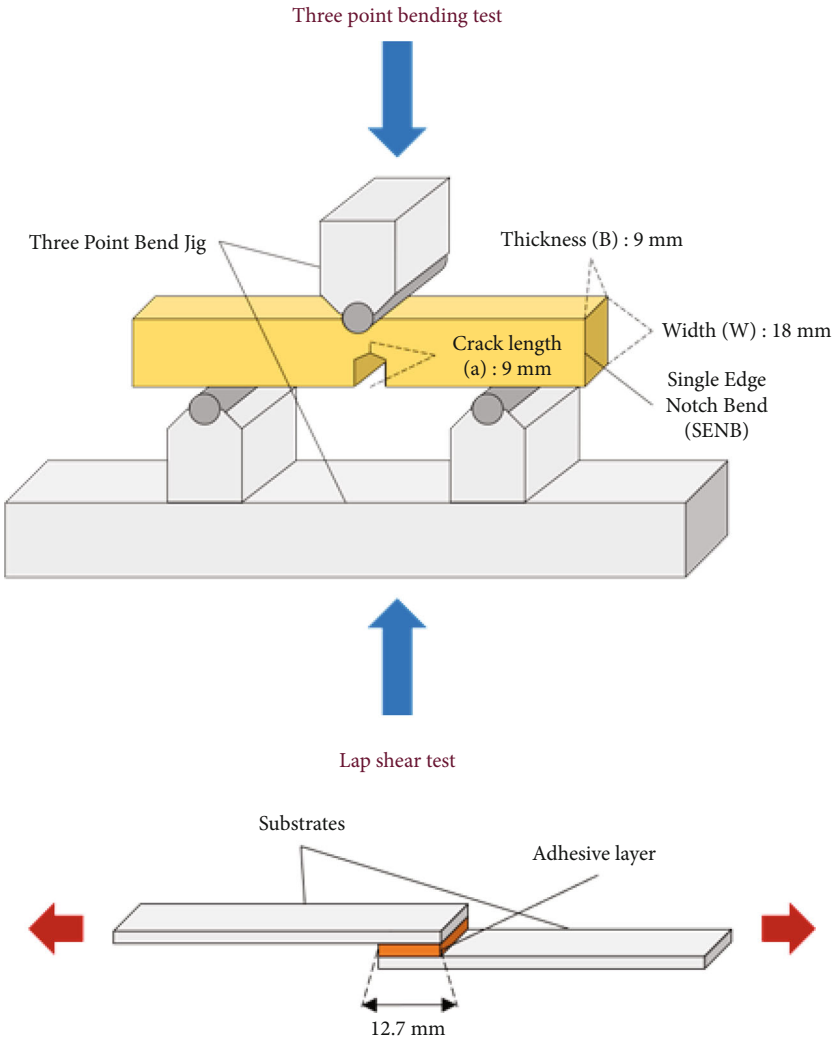


Figure 1: Specimen setup for lap shear test and fracture toughness.

for the polymer reaction. Hydrochloric acid (HCl; analytical grade) was obtained from Sigma–Aldrich, South Korea.

2.2. Preparation of Epoxy/CSR Blends. CSR particles (Paraloid EXL-2611) with an average diameter of 150 nm were based on a polybutadiene core and a poly(methyl methacrylate) shell. For the processing of epoxy/CSR blends, an appropriate amount of epoxy resin and CSR (15 phr) was preheated at 60°C and mixed in a high-speed stirring mixer at 3000 rpm for 4 minutes.

2.3. Preparation of Epoxy/CSR/MPB (E/C/M) Blends. Two molecular weights of MPB (3.1 and 5.6 K) with various amounts (5, 10, and 15 phr) were mixed with an epoxy/CSR system (28.74 g) in the presence of a small amount of HCl (2 ml) at 130°C for 2 hours using a high-shear mixer at 3000 rpm in order to improve MPB dispersion. DICY (9.30 g) was added to the mixture and stirred for 3 minutes at 2000 rpm under atmospheric pressure until the mixture became homogeneous. The obtained epoxy/CSR/MPB (E/C/M) blend was transferred to a Teflon mold and cured at 160°C for 3 hours, followed by post-curing at 180°C for 1 hour. The prepared E/C/M blends with different molecular weights of MPB were classified into two sets according to their molecular weight and phr conditions. The first set is denoted as E/C/M (3.1 K, 5 phr), E/C/M (3.1 K, 10 phr), and E/C/M (3.1 K, 15 phr). The second set is denoted as E/C/M (5.6 K, 5 phr), E/C/M (5.6 K, 10 phr), and E/C/M (5.6 K, 15 phr). Synthetic routes of MPB-modified epoxy resin are shown in Scheme 1.

2.4. Characterization. Lap shear and T-peel tests of the samples were carried out in accordance with ASTM D-100 using an electromechanical universal testing machine (Tinius Olsen 25ST, United Kingdom). Three-point bending tests were performed to determine the fracture energy of the MPB-incorporated epoxy systems [23]. Figure 1 shows a schematic representation of the three-point bending and lap shear tests.

3. Results and Discussion

Figure 2 shows lap shear strength versus displacement curves of the neat epoxy resin and E/C/M blends having

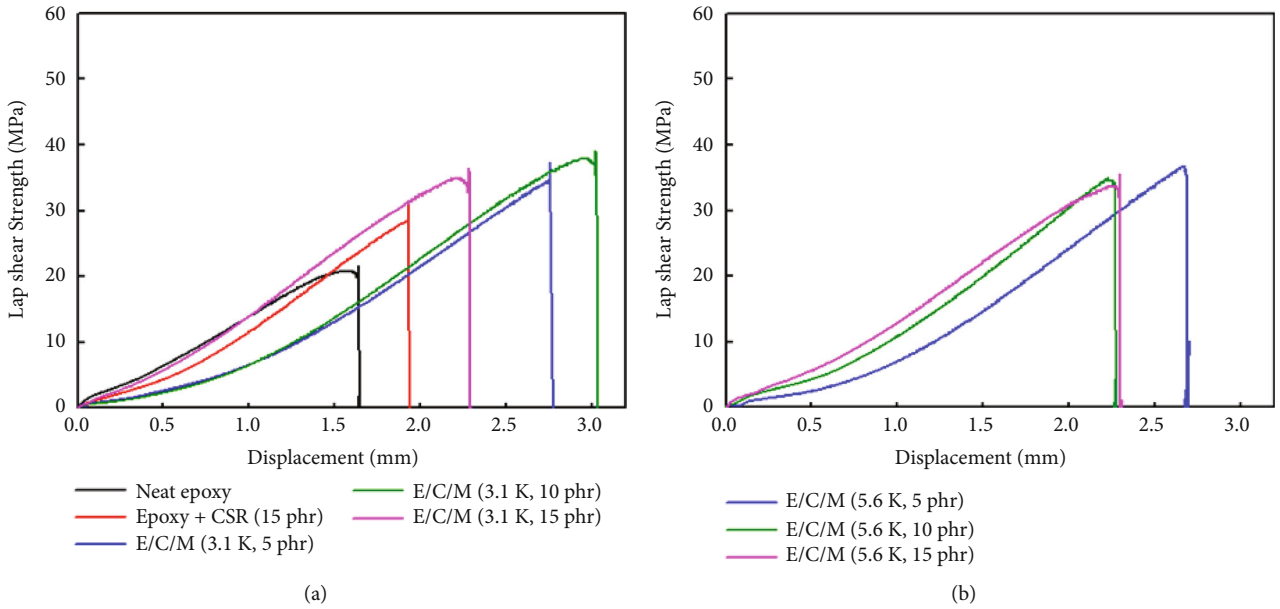


Figure 2: Lap shear strength–displacement curves obtained in the lap shear test for (a) E/C/M (3.1 K) and (b) E/C/M (5.6 K) with different phr conditions.

different molecular weights. Lap shear test was carried out to determine the adhesive bonding strength of MPB incorporated epoxy resins. The lap shear test provides information on the performance of adhesives and surface treatments [24]. The shear strength obtained for the neat epoxy resin was 22.43 MPa (Figure 2(a)), whereas that for E/C/M (3.1 K, 10 phr) was 40.60 MPa. This difference can be attributed to the increased toughness of the epoxy material because of the perfect MPB blending. Among the E/C/M (3.1 K) blends, E/C/M (3.1 K, 10 phr) was found to be the most effective with the highest shear strength (40.60 MPa), which is 56% higher than that of the neat epoxy material. This improved shear strength indicates strong interfacial bonding between the epoxy matrix and MPB with enhanced load transfer efficiency [25]. The shear strength of E/C/M (3.1 K, 15 phr) was significantly decreased upon increasing the amount of MPB from 10 to 15 phr, which was possibly due to particle agglomeration that resulted in reduced surface free energy and poor interfacial bonding between MPB and the epoxy matrix [26]. Figure 2(b) shows the lap shear strength of the E/C/M (5.6 K) blends. The lap shear strength of E/C/M (5.6 K, 5 phr) was higher than that of E/C/M (5.6 K, 10 phr) and E/C/M (5.6 K, 15 phr). For a brittle neat epoxy resin with high molecular weight and low flexibility, the incorporation of an approximate amount of MPB can dampen the external stress and enhances the ability of epoxy resin for resisting against crack propagation. Therefore, lap shear strength increases. The shear strength of E/C/M (5.6 K, 5 phr) is 37 MPa, which is 51% greater than that of the neat epoxy resin, demonstrating good adhesion between the epoxy resin and MPB.

The prepared E/C/M (3.1 K, 10 phr) and E/C/M (5.6 K, 5 phr) samples were selected and compared their lap shear strength values using a bar chart, as shown in Figure 3. All MPB-incorporated epoxy systems displayed a higher lap shear strength than neat epoxy and epoxy/CSR systems. Among them, at 10 wt% loading, E/C/M (3.1 K, 10 phr) showed the maximum enhancement in the lap shear strength due to the superior structural integrity of the epoxy resin with an appropriate amount of MPB. The inferior peel strength of E/C/M (5.6 K, 5 phr) as compared to that of E/C/M (3.1 K, 10 phr) indicated that the addition of high-molecular-weight MPB decreased the bonding strength between epoxy molecules, which in turn affected the peeling strength. Lap shear strength values of all MPB-modified epoxy samples are presented in Table S1.

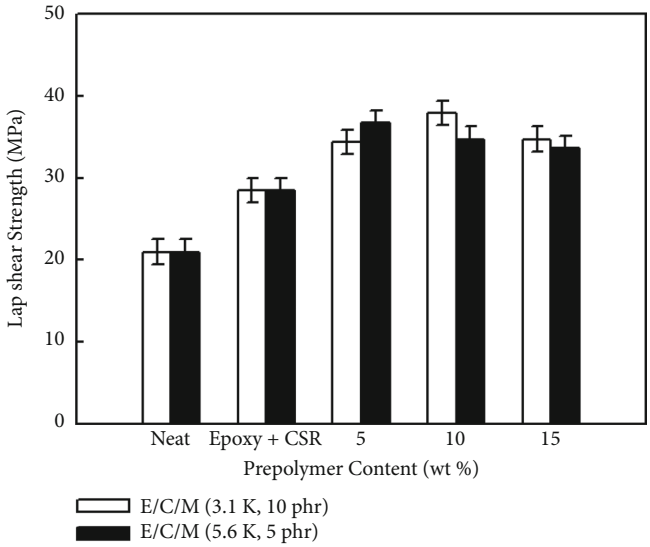


Figure 3: Comparison plot of lap shear strength between E/C/M (3.1 K) and E/C/M (5.6 K) epoxy resins using different MPB wt%.

Adhesion strength is the most essential factor in determining the performance of epoxy resins [27]. The T-peel

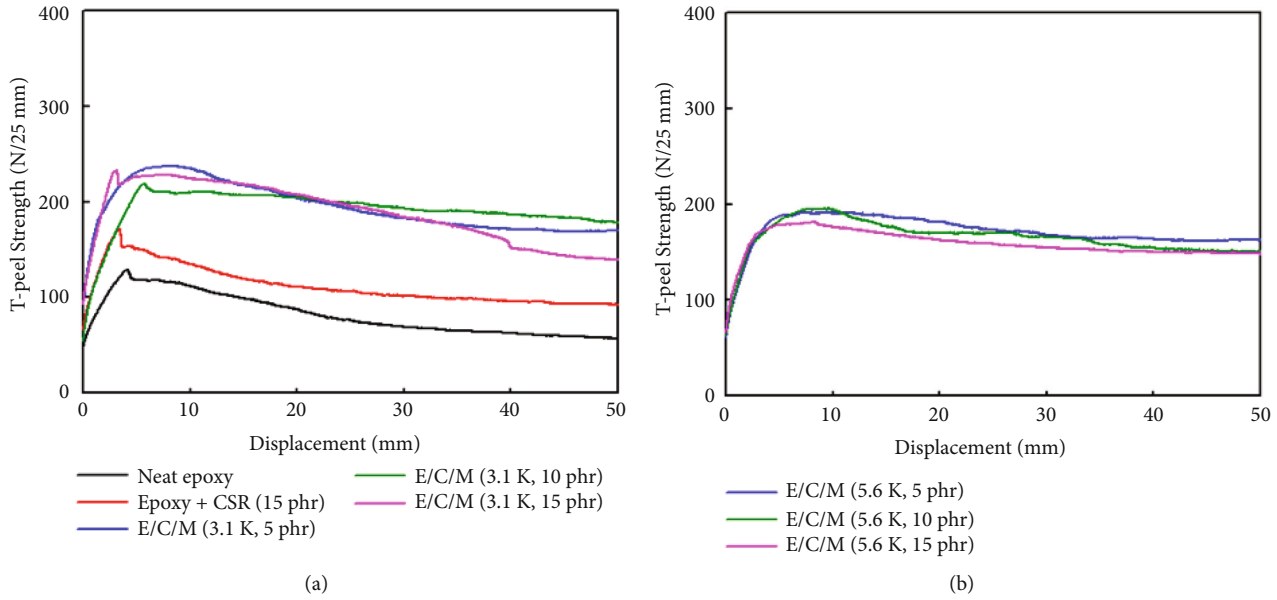


Figure 4: T-peel strength–displacement curves for (a) E/C/M (3.1 K) and (b) E/C/M (5.6 K) with different phr conditions.

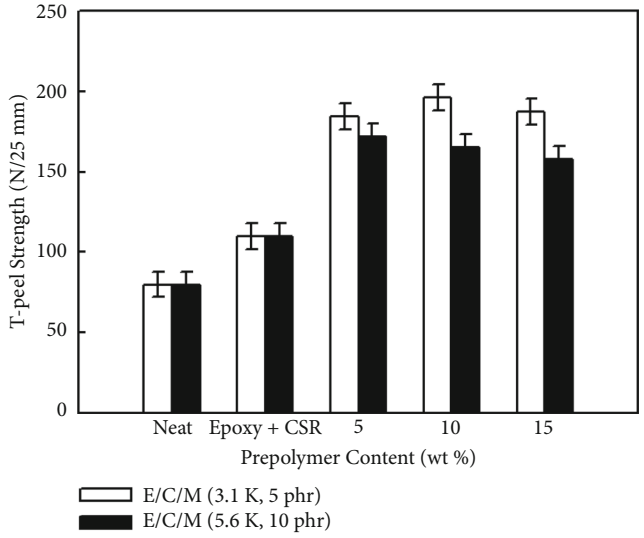


Figure 5: Comparison plot of T-peel strength between E/C/M (3.1 K) and E/C/M (5.6 K) epoxy resins using different MPB wt%.

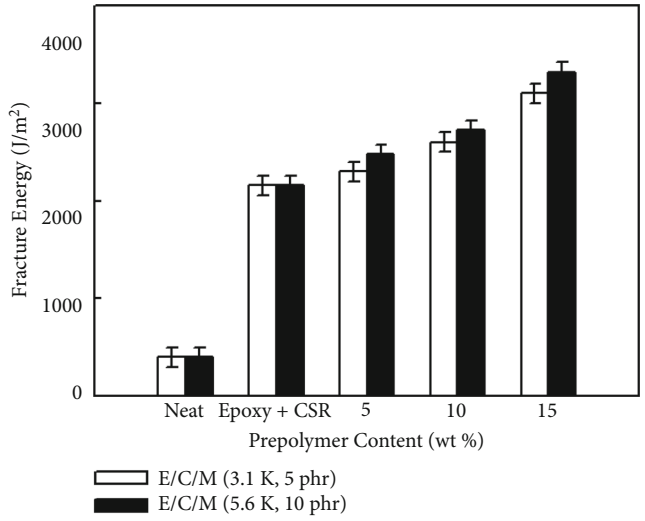
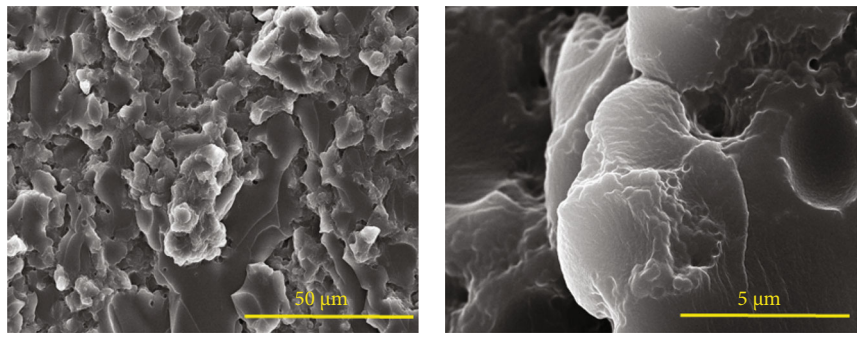


Figure 6: Comparison plot of fracture energy between E/C/M (3.1 K) and E/C/M (5.6 K) epoxy resins using different MPB wt%.

strength test was performed to analyze the adhesion strength of the MPB-modified epoxy resin and gain for a better understanding of the surface adhesion properties of two flexible substrates. This test is useful for analyzing the detailed crack-blocking capability and cohesive strength of the prepared samples [28]. Peel strength–displacement curves for the neat epoxy resin and MPB-modified epoxy resins with different molecular weights are presented in Figure 4. The T-peel strength of the neat epoxy resin is approximately 125.05 N/25 mm, which is lower than that of the blended samples. The poor peeling strength of the neat epoxy resin might be due to the presence of cracks on the inner side of the epoxy matrix [29]. These cracks cause instabilities and reduce compressive strength and toughness, resulting in the dissipation of more impact energy. The T-peel strength and displacement of the epoxy resins improved after the addition of MPB. Among the E/C/M (3.1 K) samples (Figure 4(a)), E/C/M (3.1 K, 5 phr) showed an outstanding T-peel strength of 240.81 N/25 M, which is 63% higher than that of the neat epoxy resin. This improved peel strength demonstrates that the incorporation of MPB remarkably suppresses the brittle nature of the epoxy resin and blocks the formation and propagation of microcracks in the epoxy matrix. This further improves the toughness of the epoxy system. All other E/C/M (3.1 K) samples also showed superior T-peel strength compared to that of the neat epoxy resin because of the flexible structure achieved after the addition of MPB.

The T-peel strength–displacement curves of E/C/M (5.6 K) samples are shown in Figure 4(b). The addition of



(a) (b)

Figure 7: Scanning electron micrographs of (a) 50 μm and (b) 5 μm of E/C/M (3.1K, 5phr) system.

high molecular mass MPB particles significantly enhances the fracture toughness of the epoxy resin. This decreases the brittleness and cohesive interactions between the epoxy molecules [30]. Moreover, the improved toughness enhances impact resistance, elongation, and resistance to crack propagation [31]. The T-peel strength of E/C/M (5.6 K, 5 phr) was 190.42 N/25 M, whereas that of E/C/M (5.6 K, 10 phr) showed a slight increase, that is, 195.83 N/25 M. The enhanced T-peel strength imparts excellent physical strength and resistance to vibration, impact, and shock [32]. However, the E/C/M (5.1 K) samples showed slight crack propagation and had a brittle nature after the curing process, which drastically affects the development of first-rate epoxy resin adhesives for practical applications. T-peel strength values of all MPB-modified epoxy samples are summarized in Table S2.

A bar chart is used to compare the T-peel strength between E/C/M (3.1 K, 5 phr) and E/C/M (5.6 K, 10 phr), as shown in Figure 5. These samples were used for comparison because of their excellent performance at different molecular weights. E/C/M (3.1 K, 5 phr) exhibits higher T-peel strength compared to E/C/M (5.6 K, 10 phr) at all loadings. At 10 wt%, E/C/M (3.1 K, 5 phr) exhibited the maximum T-peel strength of 205.03 N/25 M, whereas the corresponding value for E/C/M (5.6 K, 10 phr) was 174.84 N/25 M. These results indicate that short-chain MPBs are better additives for improving the physical and mechanical properties of epoxy resins. Therefore, epoxy resins modified with short-chain MPBs are more flexible and convenient for producing high-performance adhesives.

Figure 6 shows the changes in fracture energy for both neat epoxy resins and epoxy/CSR polymers modified with different molecular weights of MPB under different phr conditions as a function of MPB content. Fracture energy is one of the key parameters that reveal cracking resistance and fracture toughness of MPB-based epoxy resins. Fracture energy of 350 J m^{-2} was obtained for the neat epoxy resin. The incorporation of 5 wt% MPB into the epoxy resin increased this value to 2200 J m^{-2} for E/C/M (3.1 K, 5 phr) and 2650 J m^{-2} for E/C/M (5.6 K, 10 phr). These results reveal that MPB could create a large dispersion to block crack growth in the epoxy matrix by toughening crack deflection. These changes can improve the fracture resistance of resins. Moreover, the fracture energy increases proportionally with the amount of MPB in the epoxy matrix. A more pronounced enhancement in fracture energy was observed upon the incorporation of 15 wt% MPB; the fracture energy values increased to 3200 J m^{-2} for E/C/M (3.1 K, 5 phr) and 3450 J m^{-2} for E/C/M (5.6 K, 10 phr). These values increased by 160% and 163% for E/C/M (3.1 K, 5 phr) and E/C/M (5.6 K, 10 phr), respectively, as compared with that of the neat epoxy resin. The above results implied that the fracture energy increases intensely with increasing MPB molecular weight owing to the improved flexibility of the epoxy chains. The higher fracture energy recorded for the modified epoxy resins with respect to the prepolymer content (wt%) suggests strong dispersion of MPB in the epoxy matrix, leading to effective load transfer between the MPB layers and the matrix resin.

Figure 7 shows the scanning electron microscopy images of the fracture surface of the cured E/C/M (3.1 K, 5 phr) system under different magnifications. The micrographs confirm the appearance of MPB on the surface of modified epoxy resin. The morphology of the cured E/C/M (3.1 K, 5 phr) system consists of relatively small MPB polymer particles uniformly dispersed in the epoxy matrix. The uniform distribution of the particles throughout the matrix is very important for toughening, as it allows the yielding process to operate throughout the matrix [33].

4. Conclusions

In this study, modified high-performance epoxy resins were synthesized by incorporating MPB having two different molecular weights at different concentrations using a mechanical stirring method. The mechanical properties, such as T-peel strength, lap shear strength, and fracture energy of the modified epoxy system, were evaluated and compared using different characterization techniques. The weight percentage of the MPB-modified epoxy resin was varied for comparison. E/C/M (3.1 K, 10 phr) exhibited greater lap shear strength (40.60 MPa) as compared with the other modified and neat epoxy resin (22.43 MPa). The T-peel test results indicated that the peel strength of the modified epoxy system was strongly dependent on the amount of MPB. The maximum T-peel strength for E/C/M (3.1 K, 5 phr) was 17% higher than that of E/C/M (5.6 K, 10 phr) at 10 wt% loading. Moreover, the fracture energy of

the MPB-modified epoxy resins systematically increased with increasing weight percentage of MPB in the epoxy matrix owing to an improvement in chain flexibility. The findings of the present work would be helpful in the development of ideal epoxy resins for a wide range of industrial applications.

Data Availability

Data supporting this research article are available from the corresponding author or first author on reasonable request.

Conflicts of Interest

The author(s) declare(s) that they have no conflicts of interest.

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Supplementary Materials

Table S1 Lap shear strength values of the MPB-modified epoxy samples. Table S2 T-peel strength values of the MPB-modified epoxy samples. *(Supplementary materials)*

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