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Research Article

Damage-Tolerant Woven Glass Fiber Composites Developed Using Polyvinyl Butyral (PVB) Unsaturated Polyester (UP) Blends

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The thermoset matrix is brittle and shows low damage characteristics, and their impact and damage performance can be improved significantly by blending with the thermoplastic matrix. In this way, the properties of both the matrices can be gathered in one composite. This study is focused on the development and optimization of novel blends of unsaturated polyester (UP) resin with polyvinyl butyral (PVB), a thermoplastic polymer, to improve the mechanical properties, especially delamination and impact behavior of associated glass fiber composites. The five blends of UP and PVB were prepared in different concentrations by the solution mixing method. Composite samples of woven glass fabric were fabricated using prepared blends and pure resins as matrices on compression molding. Tensile, flexural, T-peel tests, and the instrumented Charpy impact tests were conducted on the developed samples. A significant improvement in the impact energy absorption (102%) and delamination resistance (110%) was observed for a blend ratio of 40 : 60 and 50 : 50 of PVB : UP, respectively, as compared to pure UP composite samples.

1. Introduction

Thermosetting polymer matrix composites are increasingly being used in combat, civil, aerospace, and structural applications. Their high strength, excellent dimensional stability, and good thermal resistance make them ideal for a variety of applications [1]. Thermosetting resins belong to a class of chemicals that can be crosslinked by the application of heat and curing agents. A three-dimensional network structure forms after crosslinking [2, 3]. However, because of excessive crosslinking sites, thermosetting polymers are inherently less resistant to tolerate high mechanical loadings, therefore proving to be a material lacking in toughness and high impact absorption ability. Consequently, improving and customizing the thermosetting matrix composite for damage resistance under high-impact events has become a challenge [4–8]. High-performance thermoplastic resins present a promising alternative to thermosetting resins as the former type of polymers offers much better toughness, high impact strength, low density, and lower production cost [9]. The modification of thermosetting resin with thermoplastic toughening phase can impart ductility in the resin, resistance to crack propagation, and further help in controlling the viscosity of the thermoplastic melt during processing. Researchers are now working on the various possibilities of toughening the thermosetting polymer with thermoplastics and liquid rubbers so that these modified blends can be used as a matrix for composite fabrication.

Yamanaka et al. worked on the physical blending of liquid nitrile rubber and epoxy resin by varying processing conditions to optimize the process. This modified resin when cured showed a cocontinuous structure with higher peel resistance and excellent damping efficiency [10]. Inoue prepared blends of epoxy and polyethersulfone (PES) by solution processing and studied their spinodal decomposition due to curing reaction. This polymer can be used as a

matrix for fiber-reinforced composites (FRCs) [11]. Polyvinyl butyral (PVB) appeared to be a good thermoplastic material used as a toughening agent in composite recommended for a variety of different applications [12–14]. A blend of PVB and phenolic resin was prepared and characterized for thermomechanical properties. A 25% increase in impact toughness at 10 phr of PVB loading was observed as compared to unmodified phenolic. However, by increasing PVB content, a significant decrease in thermal stability was also observed [15]. Researchers reported much work on the blending of poly (vinyl butyral) with poly (methyl-methacrylate) [16], poly (3-hydroxybutyrate) [17], and poly (vinyl butyral)/poly (vinyl alcohol)/nylon 6 ternary crystalline systems [18]. Sayman and Sayman worked on the comparison of the impact properties of thermoplastic and thermosets resin-based composites. It was concluded that the type of resin used as a matrix plays an important role in the impact strength of composite [19]. Dogan also worked on the comparison of the mechanical properties of E-glass fiber reinforced composites using thermoplastic and thermoset resins. It was deduced from experiments that the thermoplastic-based composite has better resistance to low-velocity impact than thermoset-based composite [20].

Unsaturated polyester resin (UP) is the most widely used thermosetting polymer among all polymeric materials owing to its low cost, ease of processing, and higher crosslink density. UP resin holds unsaturation that is converted to a rigid, three-dimensional crosslink structure by the reaction of styrene (a diluent and curing agent) with UP chains [21]. Methyl ethyl ketone peroxide (MEKP) is a typical catalyst that triggers the crosslinking reaction in conjunction with cobalt-octoate or cobalt-amine activator that accelerates reaction at ambient temperature [22–24]. There is a need to improve the energy absorption and crack resistance of UP by adding some toughening agents. These mechanical properties of UP can be improved by blending it with other polymers or by reacting them with different additives or modifiers, which generally form a second dispersed phase after the resin is cured. Many thermoplastic polymers including polypropylene [25], polystyrene and polycarbonate [26], and poly (vinyl acetate) [27] were blended with UP. PVB is an engineering polymer composed of vinyl butyral and vinyl alcohol units. The vinyl alcohol segment is polar and thus represents the hydrophilic part, whereas the hydrophobic part is represented by the vinyl butyral segment. There is an opportunity that this random copolymer having hydrophilic and hydrophobic segments can be admixed with homopolymers to produce miscible blends mainly due to intrachain repulsion between polar and nonpolar segments [28, 29]. Thus, PVB was found to develop stable and miscible blends with a variety of polymers. PVB may yield better mechanical properties when blended with UP and such physical blend, as well as its use in composite fabrication, has not been reported previously.

The aim of this research was to prepare a blend of UP and PVB by optimizing the blend composition and to apply this blend in glass fiber reinforced composites for high-impact applications. The prepared composite samples by varying the concentration of PVB in UP were mechanically tested to check the mechanical stability of the blend to sustain various types of loadings. Moreover, the adhesion of prepared blends for glass fabric was also checked to study the interface strength of the prepared blends.

2. Materials and Methods

The unsaturated polyester resin (UP) was purchased from Nimir Resins Limited, Lahore, Pakistan. The molecular weight and acid number of UP resin were 2500 and 25, respectively. Methyl ethyl ketone peroxide (MEKP) with 99 percent purity and cobalt octoate with 99.8 percent purity was used as initiators and accelerators, respectively. Both MEKP and cobalt-octoate were supplied by the Tianjin Nouryon Chemicals Co., Ltd. Beichen district, China. Polyvinyl butyral (PVB) was provided by the Tanyun Chemical Research Institute, Liaoning, China, with 72–82 wt.% butyral content. Methanol was obtained from Sigma Aldrich, Taufkirchen, Germany, with a 99.8% purity. The woven S-glass fabric (plain weave) with areal density (GSM) equal to 500 was used.

2.1. Preparation of PVB-UP Blend. Table 1 shows the composition details of PVB and UP for preparing different blends. For preparing a 30% w/v solution of PVB in methanol, PVB was added in methanol gradually. The stirring (200 rpm) was done at room temperature for 12 hours (overnight) to make sure of even dispersion. A viscous and transparent solution of PVB was prepared. The formed solution was then mixed with a measured amount of UP at room temperature and 200 rpm for 15 minutes. Six samples were prepared by varying the compositions of PVB solution and UP.

2.2. Fabrication of Glass Fiber Reinforced Composites. Prepared blends were then mixed with 1% by weight MEKP and 0.1% by weight cobalt-octoate and applied to each layer of glass fabric using silicon-spatula. These layers were stacked and cured in a press-mold of compression molding machine at 180°C and 13.7 MPa (2000 psi). The molds were clamped for initially 5 minutes, degassed by opening molds, and then again clamped for 10 minutes. Figures 1 and 2 show the process flow diagram and the experimental setup for the fabrication of glass fiber reinforced PVB-UP composites, respectively. The fiber volume fraction of all the samples was $40 \pm 2\%$.

Table 2 shows the test type and their corresponding ASTM standards. The tensile behavior of composites was analyzed using ASTM D 3039 and a universal testing machine (UTM model: Zwick-Z100) was used for this purpose [30]. The samples used for this test had dimensions of 25 mm and 200 mm ($W*L$). The test was operated under the strain rate of 5 mm/min. Instrumented Charpy Pendulum impact tester (Zwick/Roell HIT5, 5P) was used to investigate the impact properties of samples. The samples were cut to dimensions 10 mm × 100 mm [31]. The bending test of the composite was performed by the ASTM D7264 on UTM model: Zwick-Z100, and the samples used for this

Table 1: Details of samples of the blend of PVB and UP.

Sr. #	Polyvinyl butyral (PVB), %	Unsaturated polyester resin (UP), %
UP-P	0	100
U-PVB-1	20	80
U-PVB-2	40	60
U-PVB-3	50	50
U-PVB-4	60	40
U-PVB-5	80	20
PVB-P	100	0

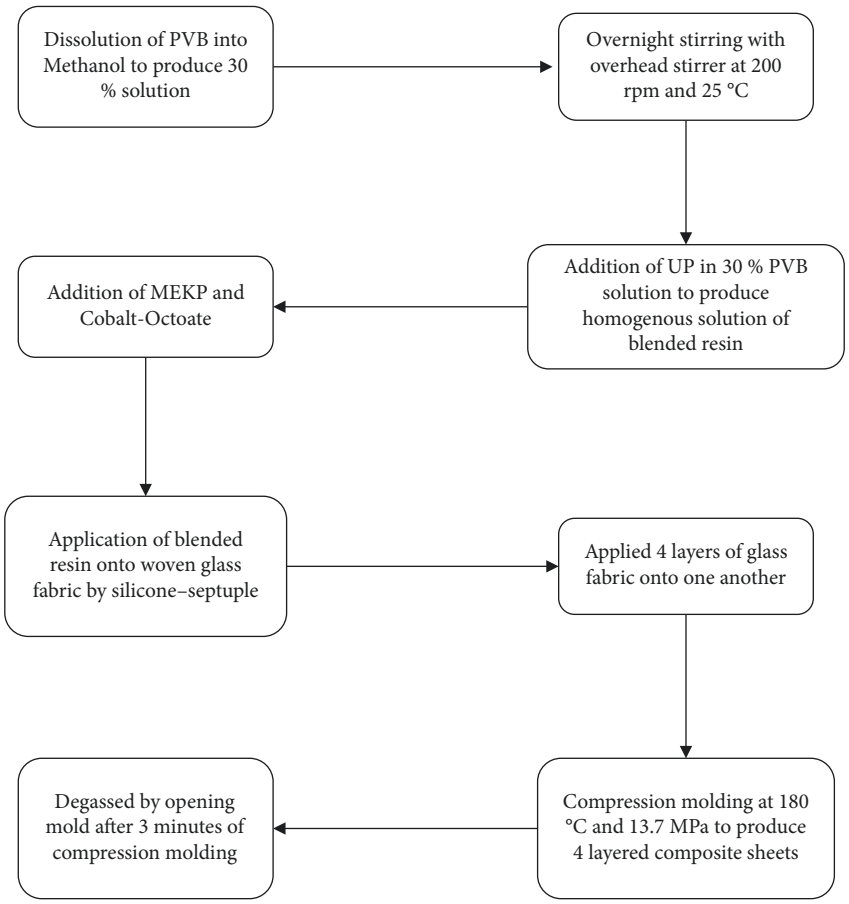

Figure 1: Process flow chart for blend and composite fabrication.

test had a dimension of 64 mm × 2 mm × 13 mm ($L \times T \times W$). The test was operated under the strain rate of 1 mm/min [32]. Similarly, the T-Peel test of produced samples was performed according to test standard ASTM D1876 on the UTM machine. The samples used for this test had dimensions of 200 mm∗25 mm ($L*W$), and the slit length was 10 cm. The test was operated under the strain rate of 1 mm/min [33].

3. Results and Discussion

Mechanical properties of the composite material such as tensile strength, flexural strength, impact resistance, and delaminating resistance mainly depend upon three parameters: (1) fiber strength and modulus, (2) chemical stability of matrix, and (3) fiber-matrix adhesion. Moreover, fiber volume fraction, alignment, and orientation also play an important role in the mechanical properties of composite materials.

3.1. Tensile Behavior. Figure 3 shows the stress-strain curves of each sample with different UP and PVB concentrations. The curve for pure PVB shows a very high modulus and strength of 235 MPa since it is an engineering polymer. The curve for a pure UP reinforced sample shows a relatively poor tensile strength of 90 MPa. The addition of PVB to UP improved its mechanical properties. In sample U-PVB-1, by adding 20 percent PVB, the tensile strength was increased to 113 MPa. The increase in tensile strength was

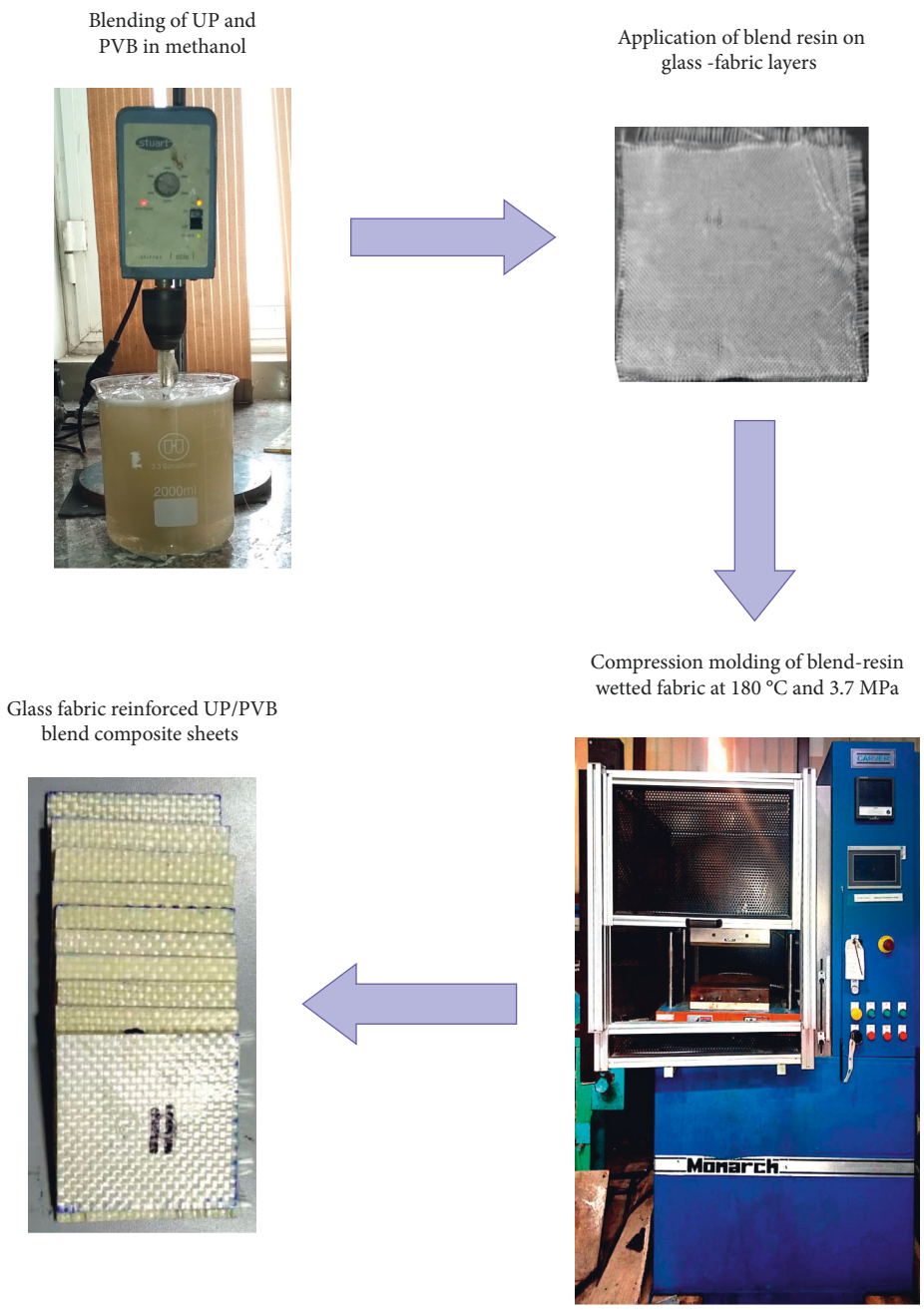

Figure 2: Experimental setup for the fabrication of PVB/UP blend composite sheets.

Table 2: Test types and standards used for tests.

Sr. no	Test name	Standard	Equipment
1	Tensile test	ASTM D 3039	UTM
2	Flexural test	ASTM D7264	UTM
3	Delamination test	ASTM D1876	UTM
4	Charpy impact test	ASTM D6110	Pendulum impact tester

25.5 percent. When the PVB was further increased to 40 percent, the tensile strength of 170 MPa was achieved. In other words, an 88.8 percent increase in strength was obtained only by the addition of 40 percent PVB in glass-UP composites. Further increase in PVB content does not yield better results. When the PVB content was increased to 50 and 60 percent, the tensile strength of 125 and 120 MPa was achieved, respectively. When the PVB content was further increased to 80 percent, the tensile strength was drastically decreased to 115 MPa. An upward trend in

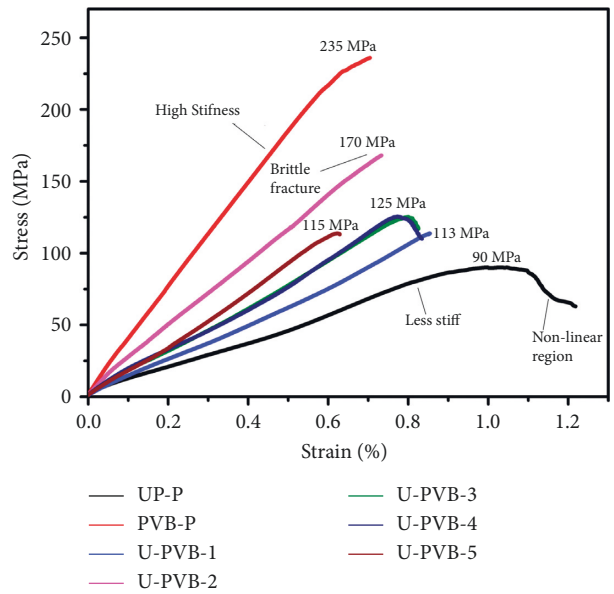

Figure 3: Stress-strain curves of test samples showing the behavior of pure and blended resin composites.

tensile strength was observed with an increase in PVB content. But this trend was only up to 40 percent PVB. After that, further increase in the PVB content causes a reduction in tensile strength. Sample U-PVB-2 having 40 percent polyvinyl butyral and 60 percent unsaturated polyester (UP) shows the highest value of tensile strength in comparison with other blends. This peak value could be due to the optimum blending ratios of PVB and UP. This optimum blending ratio is due to a good fiber-to-matrix adhesion.

Although both PVB and UP show good adhesion to glass fibers and are hydrophobic in nature. However, our results show that they are only compatible with up to 40 percent concentration of PVB. This could be due to the presence of hydrophilic groups of vinyl alcohol in the backbone of PVB. These hydrophilic groups reduce the compatibility of PVB with other hydrophobic polymers and cause steric hindrance. Additionally, UP is a thermosetting polymer, having a three-dimensional structure after curing. So, there might be some chances that high concentrations of PVB act as a plasticizer and reduce its ability of proper crosslinking of UP. Figure 4 shows the images of the sample after tests for tensile testing. The delamination and fiber pull-out due to the application of tensile load is visible from the images.

3.2. Flexural Test. Figure 5 shows the flexural strength of composites prepared by varying PVB content in UP resin. The sample PVB-P that is 100 percent polyvinyl butyral reinforced composite showed the highest peak of the bending force of 174.6 MPa.

However, it had a very low percentage of deformation, which is an indication of brittleness. The sample UP-P having pure UP as matrix showed very poor bending strength of 14.33 MPa but with a very high percentage of deformation. An increase in bending force was observed when PVB was added in UP. The sample U-PVB-1 showed a bending strength of 24.52 Mpa, which is 71 percent higher than that of the pure UP composite. The sample U-PVB-2 showed a bending strength of 32.81 Mpa, which is 128 percent higher than the UP-P sample. The bending strength was further increased for samples having more PVB. For example, samples U-PVB-3, U-PVB-4, and U-PVB-5 showed bending strengths of 33.35, 34.49, and 36.38 Mpa, which are 132%, 140.6%, and 153.8% higher as compared to the UP-P sample.

A significant increase in bending strength was observed from the sample UP-P to U-PVB-2. But the onward samples from U-PVB-2 to U-PVB-5 do not follow the same trend (Figure 6). This deviation in trend could be due to a mismatch of compatibilities of UP and PVB. But no downward trend was observed throughout the bending test.

3.3. Delaminating Resistance. Figure 7 shows the schematic of the T-peel test before and after the delamination test. Figure 8 shows the actual sample after delamination showing the first and second cracks.

Both the samples have pure PVB (PVB-P) and pure UP resin (UP-P) as matrix showed poor delamination resistance as compared to their blends. Figure 9 presents different curves of standard force versus strain while delamination test of different samples with different PVB content. The curves show two peaks indicating the appearance of two cracks in the sample consistent with the previous discussion (Figures 7 and 8). Figure 10 shows the delamination trend with respect to the increase in PVB content. The sample UP-P showed delamination resistance of 172.5 kgf/m. The value of delamination resistance increases as the PVB content increases.

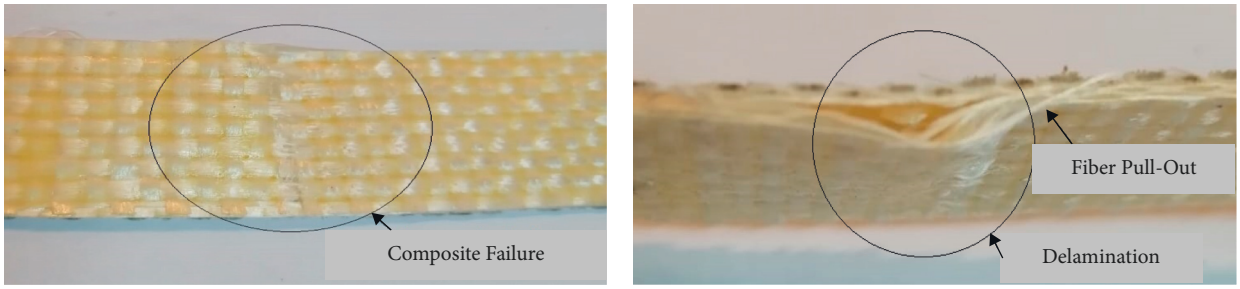

Figure 4: Samples after tensile test showing delamination and fiber pull out.

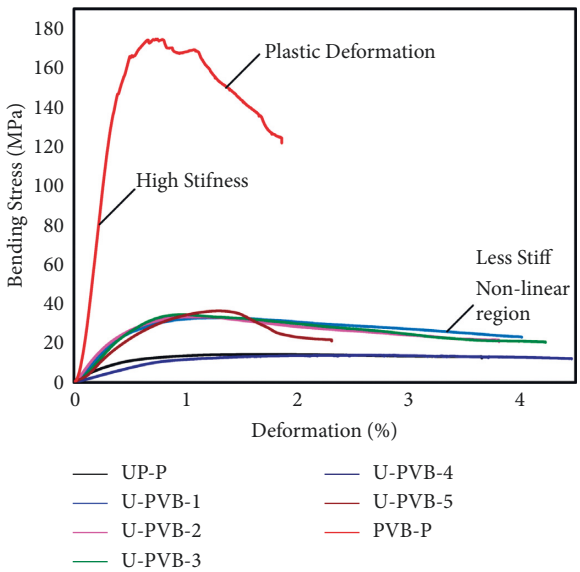

Figure 5: Force versus deformation response of developed samples of 3-point bending.

The sample U-PVB-1 with 20 percent PVB content showed delamination resistance of 206.7 kgf/m, which is 40.5 percent higher than the UP-P sample. The sample U-PVB-2 having 40 percent PVB showed delamination resistance of 324.3 kgf/m, which is 77.7 percent higher than the sample with pure UP as a matrix. The highest delamination resistance of 363 was observed for sample U-PVB-3 having 50 percent PVB and 50 percent UP content. The sample U-PVB-3 had 110 percent higher delamination resistance as compared to the pure UP sample. Furthermore, an increase in PVB content does not yield more increase in delamination resistance. For instance, when PVB content was further increased to 60 percent (for sample U-PVB-4), the value of delamination resistance was decreased to 244.3 kgf/m. The PVB content was further increased to 80 percent for sample U-PVB-5, and the value of delamination resistance was further decreased to 201.6 kgf/m. The lowest delamination resistance of 135.3 kgf/m was obtained for sample PVB-P, which is a pure PVB reinforced composite.

The blends of UP and PVB showed a synergistic effect in delamination resistance. The sample U-PVB-3 showed delamination resistance about 110 percent higher as compared to the pure UP sample and 168 percent higher as compared to the pure PVB sample. This increase could be mainly due to the perfect interface between PVB and UP molecules that results in the excellent interlocking of chains and fibers and formed a blended matrix with optimum adhesion and ductility. Such optimum ratio is also responsible for the generation of tiny micelles that further enhance adhesion due to a synergistic effect.

3.4. Impact Energy. The sample UP-P having only unsaturated polyester resin as a matrix showed impact energy of 0.773 J. Since UP is a brittle material that is why it had poor impact energy. Figure 11 shows the microscopic image (taken by optical microscope) of the delaminated sample after the Charpy impact test.

When 20% of PVB was added in UP to form a sample U-PVB-1, the impact energy raised to 1.124 J, which is 45 percent higher than a pure UP sample. The impact energy was further increased to 1.562 J when PVB content was further increased to 40 percent. The sample U-PVB-2 (having 40 percent PVB content) showed about a 102% increase in impact energy as compared to the sample with pure UP.

Furthermore, an increase in PVB content does not yield more rise in impact energy. For instance, when PVB content was further increased to 50 percent in sample U-PVB-3, the impact energy decreased to 1.28 J. A sudden decrease was observed with a further increase in PVB content onward to 50. For example, the impact energy was decreased to 1.003, when PVB content was increased to 60 percent in the sample U-PVB-4. The impact energy was further decreased to 0.925 J when PVB content was further increased to 80 percent in sample U-PVB-5. The sample with pure PVB as a matrix, namely PVB-P, showed impact energy of 0.85 J. Figure 12 shows the change in impact energy with respect to the increase in PVB content. Figure 12 shows a change in impact energy with respect to an increase in PVB content.

The highest impact energy was found for the sample U-PVB-2 having 40% of PVB and 60% of UP resin blend as a matrix. This increase in impact energy is primarily due to the optimum blending ratio. At this optimum blending, excellent fiber-to-matrix interface is developed due to the generation of tiny micelles of thermoplastic in the

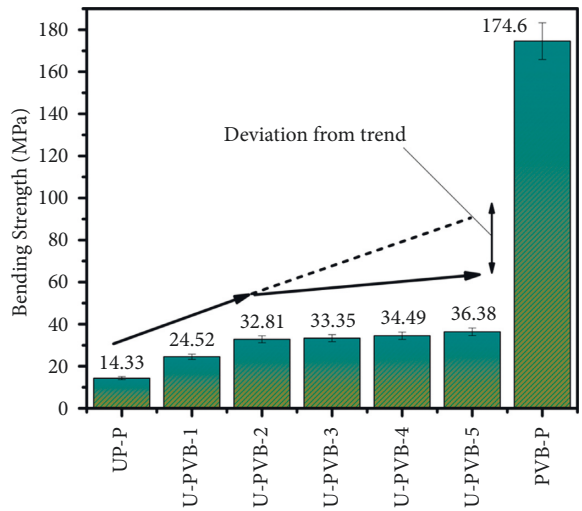

Figure 6: Flexural strength with respect to increasing PVB content.

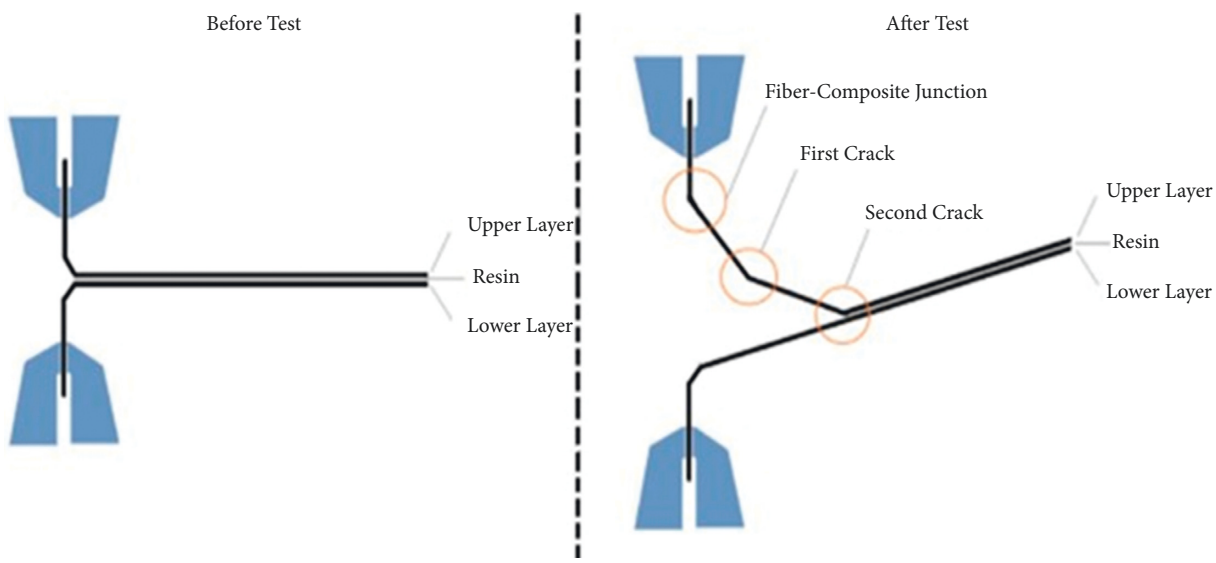

Figure 7: Schematic of samples before and after delamination test.

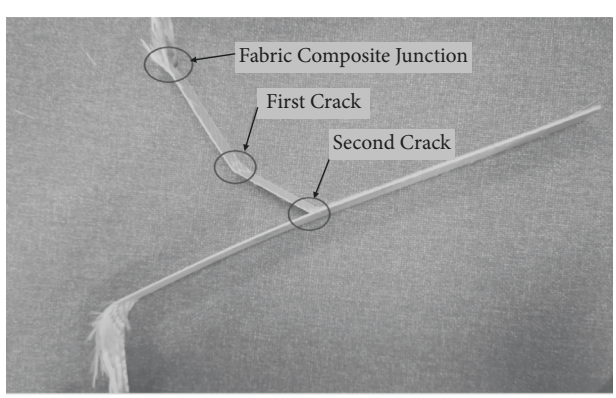

Figure 8: Actual sample after delamination test showing first crack and second crack.

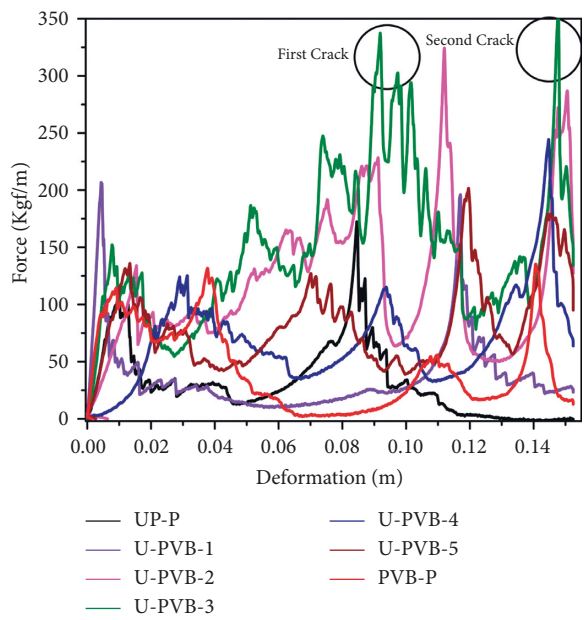

Figure 9: Different curves of standard force versus strain of delamination test of different samples with different PVB content.

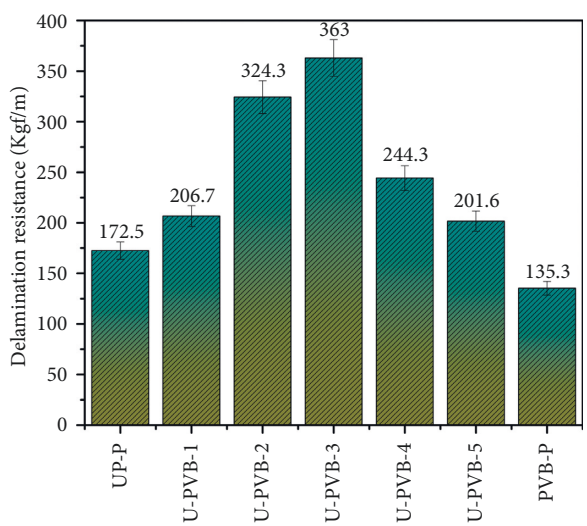

Figure 10: Change in delamination resistance with respect to increase in the PVB content.

thermoset continuum. This tiny micelle also plays a role as plasticizer due to the rolling effect, which leads to an increase in mechanical properties especially impact energy.

Figure 13 shows force vs. deformation curve using the instrumented Charpy pendulum tester. The figure shows two peaks. The first peak show matrix failure and the second peak show the peak impact force. A peak force of 170 N was observed for the U-PVB-2 sample. The area under the curve of the samples U-PVB-2 and U-PVB-3 is comparable and shows maximum energy absorption by these samples.

Figure 11: Microscopic image of the delaminated sample showing crack after impact test.

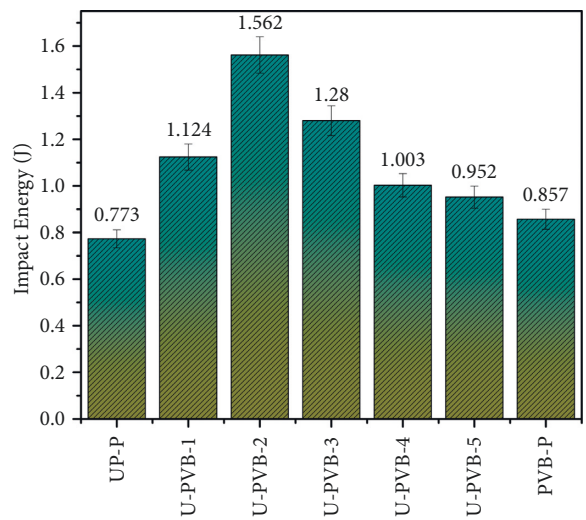

Figure 12: Change in impact energy with respect to increase in PVB content.

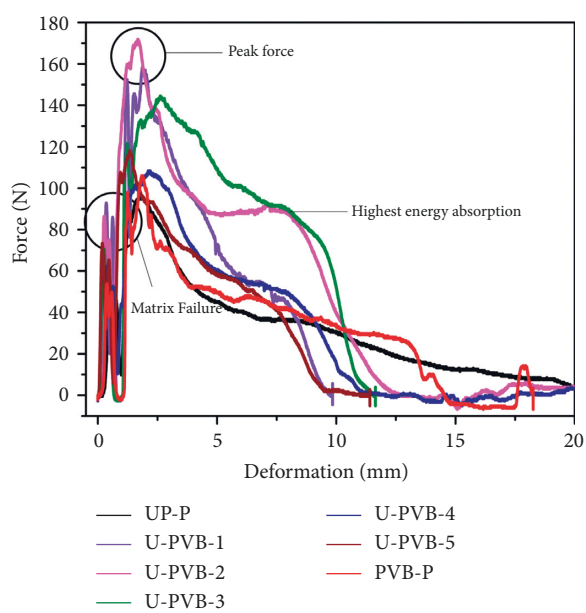

Figure 13: Force vs. deformation curve using instrumented Charpy pendulum tester.

4. Conclusion

In this article, the tensile, flexural, delamination, and the Charpy impact characteristics of glass reinforced composites were investigated. The PVB and UP were blended to find the optimized blend to further use in different applications. The results showed that the thermoplastic PVB can be effectively used by blending this with unsaturated polyester resin as a matrix for glass fibers reinforced composites. It plays the role of additive in the thermosetting polyester matrix and improves almost all mechanical properties up to an optimum concentration. The sample U-PVB-2 having 40 percent PVB was discovered best in all the mechanical tests; however, the sample U-PVB-3 having 50 percent PVB and 50 percent UP were found the best in delamination resistance. The presence of PVB in UP not only improves the flexibility but also improves fiber to matrix adhesion and shows a synergistic effect for all mechanical properties. A 40 percent PVB was found optimum for the PVB/UP blend matrix. The mechanical properties of composite improve initially by increasing the percentage of PVB, but then further increase negatively affects the mechanical properties due to the nature of both types of matrices, as increasing the percentage of PVB beyond the optimum value reduces the efficacy of both matrices. Furthermore, UP is a thermosetting polymer, having a three-dimensional structure after curing. So, there might be some chances that high concentrations of PVB act as a plasticizer and reduce the ability of proper crosslinking of the UP results in low mechanical properties. In addition, the performance of PVB/UP blend resin can be improved by the addition of a suitable compatibilizing agent.

Data Availability

Data will be made available on request.

Conflicts of Interest

The authors declare that they have no conflicts of interest.

Acknowledgments

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