

Influence of Flexographic Photopolymer-Plate Residue Incorporation on the Mechanical Properties of Glass-Fiber-Reinforced Polyester Composites

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In this study, the effects of incorporating recycled photopolymer-plate residues from a packaging flexography process into polyester-glass fiber composites were examined. Ternary composites with an unsaturated polyester matrix with elastomer particles from recycled photopolymer-plate residues were evaluated using two types of glass fibers: in the forms of a fabric with bidirectional fibers and a blanket with multidirectional fibers. The composites were prepared by hand lay-up lamination using different rubber contents (0, 2.5, 5, and 10 wt% based on the polyester resin mass fraction), and were characterized for their void content, flexural and impact strengths, and dynamic mechanical properties. Primary results indicated that the incorporation of the rubber particles increased the difficulty of lamination, while promoting greater void formation with higher filler content. The rubber particles decreased the impact resistance properties but did not reduce the flexural strength or storage modulus, indicating that despite the elastomeric composition, this residue from the photopolymer plates showed a reinforcing rather than toughening character.

Keywords: *polyester, residues, composites, glass fiber, elastomer residue.*

1. Introduction

Owing to their low weight, high specific strength, and high stiffness, fiber-reinforced polymer (FRP) composites are widely used in the aerospace, automotive, and marine industries. A thermoset resin, which is an unsaturated polyester, possesses higher application rate than other resins in the aforementioned industries because of its relatively low cost, ease of processing, and favorable compatibility with various fillers. However, unsaturated polyesters have poor mechanical properties. Particularly, they have low impact strengths, which limit their industrial applications^{1,2}. Although, has good fiber aggregation capacity, that makes its use feasible for composites matrix. One of the possible methods to enhance the mechanical properties of polyester composites is to improve the properties of the matrix by incorporating particles in the resin, as reported by Mousa et al.¹ In this study, low-density recycled rubber particles are utilized as fillers to enhance the impact properties of the composite, which is beneficial for specific applications.

Recycled elastomer particles have been extensively investigated to improve the mechanical properties of fiber-reinforced polymers composites. The toughening of unsaturated polyester by incorporating rubber particles was studied by Maspoch and Martinez³, which made it possible to understand more about the morphological properties of this material. A year later, Sjögren and Berglund⁴ studied the toughening mechanisms for rubber-modified glass-fiber-reinforced polyester composites to investigate the cracks along the material. The mechanical properties and environmental stress-cracking resistance of rubber-toughened polyester reinforced with kenaf⁵ and clay⁶ have also been studied.

Recently, Nuzaimah et al.⁷ evaluated the potential of using waste rubber gloves as fillers in unsaturated polyester-based composites. The addition of rubber glove fragments, with rubber contents ranging from 5 to 40 wt%, was found to increase the toughness of the composite by improving the impact strength, while it decreased the tensile and flexural strength, and elastic modulus. Similar results were reported by Elenien et al. in 2020⁸. The authors developed a polyester-

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rubber particle composite obtained from recycled car-tire waste. By varying the volume fraction from 0 to 60%, the observed results indicated an improvement in the impact properties and a corresponding reduction in the composite density. Additionally, Rajaei et al.² studied the microstructures and mechanical properties of the glass-fiber-reinforced unsaturated polyester composites modified by styrene-butadiene rubber (SBR) and fumed silica. The incorporation of SBR decreased the flexural strength, modulus of elasticity, and tensile modulus of the composites but increased the elongation at break, impact, and tensile strength. The best balance among strength, stiffness, and toughness was obtained using 10 wt% SBR and 3-phr-fumed silica.

In addition to the aforementioned application of rubber-waste, other authors have developed research such as Cordeiro et al.⁹ studied SBR photopolymer-plate waste products and used them as a filler blended into a thermoplastic polypropylene (PP) and ethylene vinyl acetate copolymer (EVA) resins at different loading percentages. It was found that the recycled materials enhance mechanical properties when blended with PP and EVA resins.

Flexography is a progressive printing technique that is highly applicable in the packaging industry and in the printing of different functional films and coatings; the flexographic printing-plate industry particularly uses significant quantities of the photopolymerized materials⁹. This flexography process includes different materials such as the photopolymer plate also known as cliché, containing the desired design for the printing.

Ethylene propylene diene monomer and SBR-printing plates have been recently remarketed because of their desirable resistance to various solvents¹⁰. However, their difficult and problematic recycling processes generate significant waste and considerable quantities of unwanted excess material globally^{9,10}. Maciel and Nascimento¹¹ used it as part of a composite with thermal and acoustic application, proving the possibility of a new destination for this residue.

In conclusion, although some studies have been conducted on the flexographic photopolymer-plate elastomer residues (ERs) incorporated in polymers, such as PP and EVA, studies on the waste generated in FRP composites based on polyester matrices are limited. Another advantage of the flexographic photopolymer-plate ERs is the production of cost-effective and eco-friendly products. Thus, the primary aim of this study was to examine the physical and mechanical behaviors of a glass FRP composite based on a flexographic photopolymer-

plate ER-modified polyester matrix, promoting a recycling alternative for this material.

2. Materials and Methods

2.1. Materials

The photopolymer-plate residues that were incorporated in the composites were obtained from the flexographic polymeric-packaging process and provided by Canguru Plásticos Ltda. (Criciúma/SC, Brazil). Orthophthalic unsaturated polyester resin with 30% styrene (P) and the MEKP catalyst based on methyl ethyl ketone peroxide (Butanox-M50), for the polyester matrix development, were purchased from Redelease Produtos Para Indústrias Ltda. (São Paulo/SP, Brazil). As indicated by the supplier, 1 wt% Butanox-M50 was used to promote matrix crosslinking. Glass fibers (supplied by Texiglass Indústria e Comércio Têxtil (Vinhedo/SP, Brazil)) with 200 g/m² areal weight were used as the bidirectional fabric (GF.F), and a fiberglass with an areal weight of 300 g/m² was used as the multidirectional non-woven blanket (GF.B). Before use, the fibers and elastomer residues (ER) fillers were dried in a thermal oven at 60 °C for 4 hours. According to the supplier specifications, the glass fibers used were compatible with the unsaturated polyester resins. Figure 1 shows the micrograph that is obtained by the optimal microscopy (OM) of the two fiberglass surfaces used in this study. Components of the photopolymer plates were previously separated using toluene obtained from Dinâmica Química Contemporânea Ltda. (Indaiatuba/SP, Brazil).

2.2. Methods

2.2.1. Recycling of flexographic photopolymer plates

The rubber microparticles (also referred to as ERs) were obtained by recycling the photopolymer plates⁹. The process separated the polyester-base layer from the elastomeric component via solvent exposure, and thus a higher-purity material was obtained.

Subsequently, the ERs were mechanically ground using a Mecanofar MF160 knife mill with a Ø-10-mm sieve. The ER was then micronized cryogenically using a Netzsch Atritor high-energy mill operated at 1400 rpm for a grinding time of 3 h in a cryogenic atmosphere with liquid nitrogen. Resultant particles with sizes smaller than 24 mesh (0.5 mm) were obtained. Figure 2 illustrates the different stages of the ER recycling process.

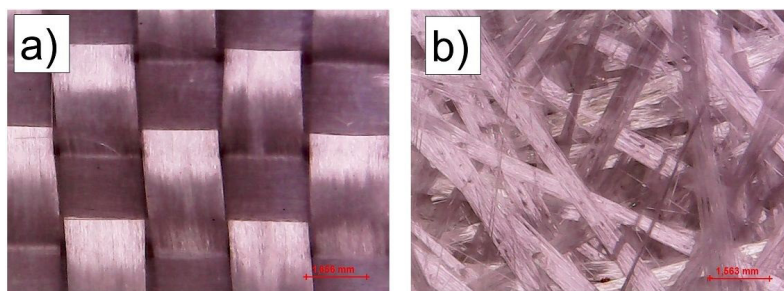


Figure 1. Micrographs obtained by OM of the glass fiber sample surfaces of (a) fabric and (b) blanket.

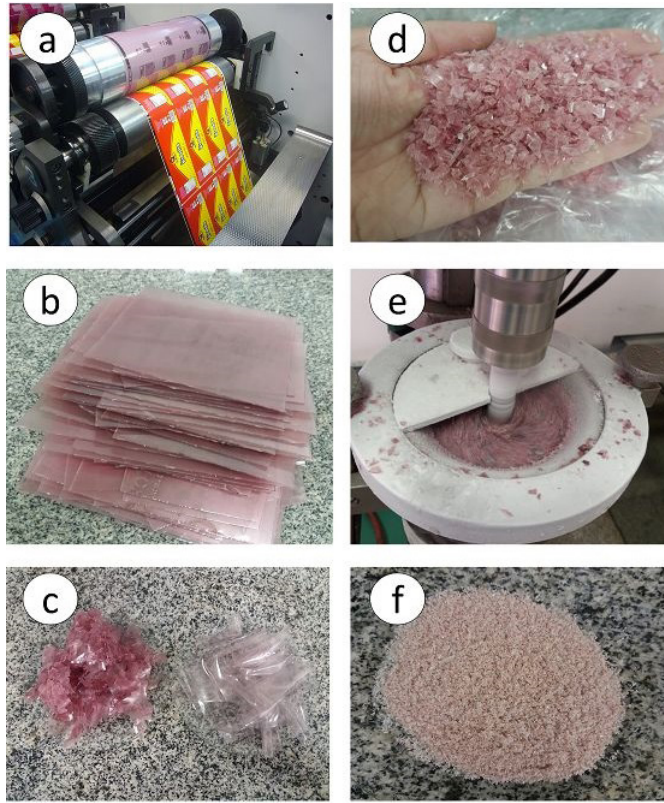


Figure 2. Images of the (a) photopolymer plate used in flexography, (b) photopolymer-plate residues, (c) photopolymer-plate residues after phase separation, (d) mechanical grinding of the residues, (e) cryomilling of the ERs, and (f) particulate ER to be incorporated into the polyester/glass fiber composite.

2.2.2. Preparation of ternary composite polyester/fiberglass/ER

Different ER contents were added to the polyester resin (0, 2.5, 5, and 10 wt% based on the mass fraction of the polyester resin present in the formulation) and a polyester-to-fiberglass ratio of 30:70 (w/w) for all configurations was adopted. It was used 4 layers of the fiberglass fabric and 3 for the fiberglass blankets. The composites were prepared via manual lamination (hand lay-up), while a metal mold coated with non-stick silicone (to facilitate the extraction of composites) (200 mm × 150 mm × 25 mm) was used for sample production. After lamination, the composites were placed in a thermal oven at 70 °C for 30 min and subsequently maintained under a controlled temperature of 23 °C for 72 h in a desiccator. Table 1 displays the composition fractions and nomenclature of the samples used in this study. The mass fraction of polyester resin, catalyst and glass fiber was kept constant, varying the type of glass fiber and the percentage of ER in the composition

2.2.3. Characterization

Morphological analysis of the ER was performed via scanning electron microscopy (SEM) using JEOL JSM-6390. The samples were covered with a thin gold film via sputtering using a Denton Desck IV vacuum metallizer. OM analysis of the glass fibers was performed using an Olympus BX41M-LED microscope.

The ER density was determined by helium-gas pycnometry using Quantachrome equipment (Ultracyc 1200e), while the composite-sample densities were measured according to the ASTM D792-00 standard¹². The samples were weighed on an analytical balance, immersed in ethanol, and subsequently weighed. Each test value was calculated as the average of seven independent measurements (seven specimens of each formulation). The density calculations were performed according to Equation 1:

$$\rho = \frac{(a \times b)}{(a - c)} \quad (1)$$

Where ρ is the sample density (g/cm³), a is the sample mass (g), b is the ethanol density (g/cm³), and c (g) is the sample mass following its immersion in ethanol.

The void content of the samples was determined according to the ASTM D2734-16 standard¹³, by obtaining the relative difference between the theoretical and measured composite densities. The theoretical densities of the composites were calculated using Equation 2.

$$\rho_T = (M \cdot \rho_m) + (G \cdot \rho_g) + (E \cdot \rho_f) \quad (2)$$

Where ρ_T is the theoretical density of the composite (g/cm³), M is the content of the polymer matrix in the composite (wt%),

ρ_m is the density of the polyester matrix (g/cm^3) (1.17 g/cm^3), G is the content of the fiberglass in the composite (wt%), ρ_g is the density of the fiberglass (g/cm^3) (2.55 g/cm^3), E is the content of the ER in the composite (wt%), and ρ_f is the density of the ER (1.12 g/cm^3). The composite-void contents were again determined from the relative difference between their experimental and theoretical densities according to Equation 3:

$$V = \left(\frac{\rho_T - \rho_E}{\rho_T} \right) \times 100 \quad (3)$$

Where V is the void content, ρ_T is the theoretical density of the composite (g/cm^3), and ρ_E is the experimental density of the composite (g/cm^3).

The flexural strength test of the composites was conducted according to the ASTM D790 standard¹⁴ using an Emic universal testing machine (model DL10000) at a test speed of 1.8 mm/min and a distance of 48 mm between the supports. Each test value was calculated as the average of seven independent measurements (seven specimens of each formulation).

Izod impact strength testing was performed with a CEAST Resil 25 pendulum using unnotched specimens, according to the ASTM D256 standard¹⁵, with individual-test values calculated as the average of minimum seven independent measurements (seven specimens of each formulation).

Data were analyzed in GraphPad Prism software version 5.00 spreadsheets. The results were expressed as mean and

standard deviation. Inferential analyzes were performed with a significance level of $\alpha = 0.05$, ie, 95% confidence. Comparison of means found between groups was performed by applying the one-way ANOVA test, followed by the post hoc Tukey test when statistical significance was observed.

The dynamic mechanical test (DMA) was performed by the dual cantilever method, using a TA Instruments equipment (model DMA T800) at a heating rate of $3 \text{ }^\circ\text{C/min}$ from 25 to $150 \text{ }^\circ\text{C}$, a frequency of 1 Hz, and a deformation amplitude of 0.1%.

3. Results and Discussion

Figure 3 presents the SEM micrographs of the ER after recycled from the flexographic plates. The particle sizes are observed to be in the range of $100\text{--}500 \text{ }\mu\text{m}$. A homogeneous distribution of particle sizes is observed. It can also be evidenced by the SEM micrograph, that the particles have significant surface roughness, which can positively contribute to the physical adhesion with the polymeric polyester matrix.

Table 2 presents the observed density and void content of the studied compositions. By comparing the two types of fiberglass reinforcements used (fabric and blanket), it was observed that the composites manufactured with fabric possessed higher density values, suggesting a better wettability of the fiber by the resin and fewer voids, since the observed mass fractions of the samples were identical and only the type of fiberglass was varied. The density of the fabric-reinforced is also associated to the resin volume content of the composite, due the fact that the fabric has

Table 1. Nomenclature and mass composition (grams) of the samples produced in this study.

Sample	Unsaturated polyester with 30% styrene	MEKP catalyst	Glass fiber bidirectional fabric	Glass fiber multidirectional blanket	Elastomer residue
P / G.F.F / ER 0	70	0.7	30	-	0
P / G.F.F / ER 2.5	70	0.7	30	-	1.75
P / G.F.F / ER 5.0	70	0.7	30	-	3.5
P / G.F.F / ER 10	70	0.7	30	-	7.0
P / G.F.B / ER 0	70	0.7	-	30	0
P / G.F.B / ER 2.5	70	0.7	-	30	1.75
P / G.F.B / ER 5.0	70	0.7	-	30	3.5
P / G.F.B / ER 10	70	0.7	-	30	7.0

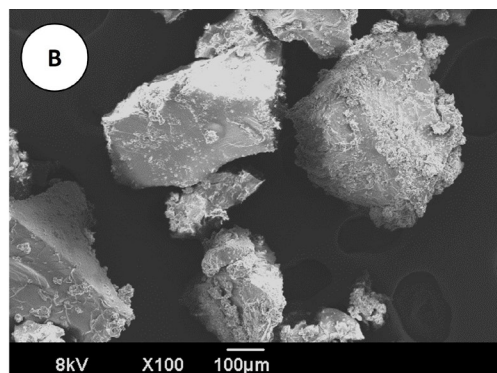
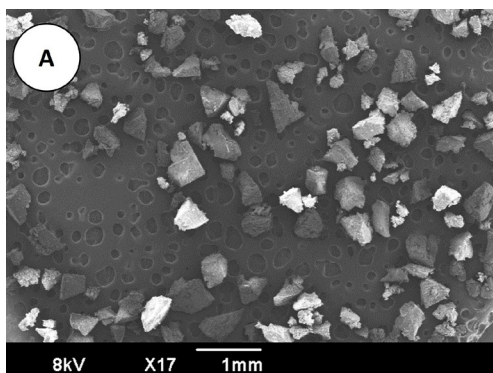


Figure 3. Micrographs obtained by the SEM of the rubber particles after processing.

less thickness than the blanket, so it needs more volume of polyester resin to complete the mold. The observed value was confirmed by the void content and ANOVA test.

The presence of voids is commonly associated with hand lay-up lamination. According to Salasinska et al.¹⁶, hand lay-up is a primary technique for lamination, which facilitates the production of finished goods using predominantly inexpensive tooling. However, it is challenging to obtain high-quality products having limited structural defects as the presence of voids is the main disadvantage of this method.

Comparing the two types of fiberglass that were used, the composites that were produced using the multidirectional fiberglass blanket had a higher void content. During molding under the same processing conditions, the impregnation of the polyester resin in the glass fiber blanket was visibly more difficult, which corroborated with the observed void content.

The contents of up to 5% ER in the sample did not cause a significant change in the void content compared to that caused by the ER absent sample. However, for both compositions (P/GF.F/ER 10 and P/GF.B/ER 10) with 10% ER, an increase

in the void content coincided with an increase in the filler content, suggesting a compositional limit of ER saturation in the composites, which led to greater bubble formation. Density showed no statistically significant difference in the different amounts of ER ($p > 0.05$).

Table 3 shows the flexural strength results of the studied samples. For the samples containing 0% of the ER (P/GF.F/ER0 and P/GF.B/ER0), the flexural strengths exhibited different results. The flexural strength, maximum force, and maximum deformation of the bidirectional fabric of the glass fiber samples (P/GF.F/ER0) were approximately 10%, 26%, and 7% lower than those of the multidirectional fiberglass non-woven blanket sample (P/GF.B/ER0), respectively. Even though the composites that were produced using the multidirectional fiberglass blanket possessed a higher void content, the observed mechanical response of the flexural strength was superior.

Zhang et al.¹⁷ reported that many factors influences the composite material properties, such as fiber orientation, fiber geometry (including shape and length-to-width ratio), and interface properties. The bidirectional fiberglass fabric

Table 2. Density and void content of P/GF.F and P/GF.B composites with different rubber powder contents.

Sample	Apparent density (g.cm ⁻³)	p-value [†]	Theoretical density (g.cm ⁻³)	Void Content (%)
P/GF./ER 0	1.53 ± 0.02	0.228	1.58	3.40
P/GF.F/ R 2.5	1.55 ± 0.07		1.60	3.34
P/GF.F/ER 5.0	1.59 ± 0.04		1.62	2.04
P/GF.F/ER 10	1.56 ± 0.03		1.66	6.15
P/GF./ER 0	1.41 ± 0.04	0.695	1.58	10.9
P/GF.F/ R 2.5	1.42 ± 0.07		1.60	11.4
P/GF.F/ER 5.0	1.45 ± 0.03		1.62	10.6
P/GF.F/ER 10	1.45 ± 0.10		1.66	12.7

[†]Value obtained after applying the one-way ANOVA test.

Table 3. Flexural strength and impact strength values of composites with different ER contents

Sample	Flexural strength (MPa)	p-value [†]	Flexural Testing			Impact strength (kJ/m ²)	p-value [†]
			Maximum force (N)	p-value [†]	Deformation (mm)		
P/GF./ER 0	162.3 ± 15.5 ^{a,c,d}	<0.001	89.8 ± 12.9 ^a	0.012	6.7 ± 0.3 ^a	68.4 ± 6.7	0.125
P/GF.F/ E 2.5	228.4 ± 33.5 ^{b,d}		134.7 ± 31.4 ^b		5.1 ± 0.3 ^b	55.6 ± 5.4	
P/GF.F/ER 5.0	197.5 ± 20.1 ^d		97.5 ± 18.0 ^{a,b}		5.1 ± 0.4 ^b	56.4 ± 10.1	
P/GF.F/ER 10	150.2 ± 21.1 ^c		87.0 ± 21.0 ^a		5.8 ± 0.2 ^c	52.6 ± 16.1	
P/GF./ER 0	180.5 ± 15.1	0.334	121.7 ± 20.2 ^{a,b}	0.006	6.2 ± 0.8 ^a	25.0 ± 2.2 ^a	<0.001
P/GF.F/ R 2.5	200.4 ± 25.5		160.6 ± 13.2 ^a		5.4 ± 1.2 ^b	16.4 ± 3.7 ^b	
P/GF.F/ER 5.0	178.2 ± 15.0		98.9 ± 8.8 ^b		5.4 ± 0.7 ^b	18.0 ± 4.3 ^b	
P/GF.F/ER 10	188.0 ± 20.0		105.0 ± 31.8 ^b		7.3 ± 0.9 ^c	15.8 ± 4.1 ^b	

[†]Value obtained after applying the one-way ANOVA test. ^{a,b,c,d} Different letters represent statistically significant differences after applying Tukey's post hoc test ($p < 0.05$).

was composed of long, intertwined strands arranged in a pattern (Figure 1). The multidirectional fiberglass non-woven blanket was composed of short strands in a random configuration.

It was observed that the ER promoted significant changes in the bidirectional fiberglass fabric composites. Increases of 40% and 20% were observed in the flexural strengths of samples P/GF.F/ER 2.5 and P/GF.F/ER 5.0, respectively, compared to that of the 0% ER sample (P/GF.F/ER0). The addition of ER possibly promoted an increase in the interfacial adhesion between the resin and fiberglass, thereby improving the distribution and transmission of energy by the composite. However, with the higher concentrations of ER (P/GF.F/ER 10), the flexural strength was similar to that of the sample with 0% ER (P/GF.F/ER0). Considering the standard deviations, the samples did not clearly exhibit significant variation. The samples with 10% ER concentration possessed higher void content, which potentially contributed to mechanical property saturation in this composition.

For composites containing multidirectional non-woven blanket fiberglass, variations in the flexural strength were not significant (considering standard deviations). The properties of the fiberglass material possibly prevailed, with no relevant interference of the ER concentration on the flexural strength of the final composite. The variety of flexural strengths values along the samples, can also be linked with the manual manufactured process, that generates irregular proportions of ER in each test specimen.

The impact resistance results are presented in Table 3, where the composite P/GF.F/ER0 exhibited a 60% superior result than that P/GF.B/ER0 did. As previously mentioned, the orientation and dimensions of the glass fibers are determining factors in the properties of the composites, as the fibers bear the maximum portion of the impact energy exposed to the composite material. Fiber orientation can also alter the strength and stiffness of the composite. Short, randomly oriented fibers possessing a small aspect ratio were easily introduced into the matrix and exhibited a relatively isotropic behavior in the composite. Long or even continuous unidirectional fiber arrays displayed anisotropic properties with favorable strength and stiffness parallel to the fibers. Therefore, this result might be correlated to the distribution and weight of the glass fiber used in the composite as the multidirectional non-woven glass fiber blanket was randomly distributed, forming a composite with strong interfacial adhesion.

For composites containing bidirectional fiberglass fabric, a decrease of approximately 15% in impact strength was observed with ER addition, regardless of concentration. Notably, in composites containing multidirectional non-woven blanket fiberglass, a decrease of ~34% in impact strength following ER addition was observed. This result indicated a low adhesion of the ER containing resin to the glass fibers, since in composite materials, the matrix transmits force to the fibers, which tended to have a superior resistance than mechanical stress. However, force transmission was ineffective when interfacial adhesion was insufficient, which promoted the formation and propagation of undesirable cracks and irreversible failures. Additionally, the presence

of voids during processing might have contributed to the lower impact strength of these samples (Table 2).

The variation in the dynamic mechanical properties as a function of the temperature of the glass fiber bidirectional fabric-reinforced polymer composites and composites containing multidirectional non-woven blanket fiberglass and ER reinforcements are shown in Figure 4.

There was a prominent increase in the storage modulus (Figure 4a) of the P/GF.F composites with the incorporation of ER over the entire region. This may correlate with an increase in the stiffness of the polyester/glass fiber composite with the reinforcement effect imparted by the ER, allowing stress transfer at the interface to occur. This result corroborates those observed for the flexural properties presented in Table 3 for P/GF and F/ER composites. The observed temperature increases coincided with the storage modulus decrease, with a decline observed near the glass transition region. This behavior may be attributed to an increase in the molecular mobility of the resin chains above the glass transition temperature^{18,19}. Additionally, ER contributed to a reduction in the composite glass transition temperature, as shown in Figure 4c. The ER particles were softer than the glass fibers, which promoted segmental mobility of the polymeric chain at lower relaxation temperatures.

Figure 4b displays the variation in the loss modulus for the glass fiber bidirectional fabric polyester composites with temperature, whereby incorporating the ER particles broadened the loss modulus peak. Peak broadening can be attributed to the inhibition of the relaxation process in the composite. This may be associated with a higher free volume upon the addition of ER. The composites also exhibited two transition peaks corresponding to the ER and polyester phases. The loss modulus values for the composites with ER were higher than corresponding composites without ER addition. This phenomenon may be due to the increase in internal friction caused by the ER particles, which enhances energy dissipation.

Figure 4d shows the variation in the storage modulus as a function of temperature for multidirectional non-woven blanket fiberglass/polyester composites with ER loading. The addition of 2.5 wt% ER particles reduced the composite stiffness in the glassy and rubbery states. Conversely, upon the addition of 5 wt% and 10 wt%, the storage modulus increased in the glassy and rubbery states according to the graph curve. Higher quantities of ER particles are potentially necessary to increase the composite stiffness, in this case to overlap the effect of multidirectional non-woven blanket fiberglass when compared to glass fiber bidirectional fabric/polyester composites.

The variation in the loss modulus for the multidirectional non-woven blanket fiberglass/polyester composites with change in temperature is shown in Figure 4e, whereby ER particle incorporation flattened the loss modulus peak. This behavior may correspond with an increase in the polymer chain mobility associated with ER addition, likely generating a decrease in mechanical properties, as observed in Table 3. As observed in Figure 4f, the presence of ER also contributed to the reduction in the composite glass transition temperature.

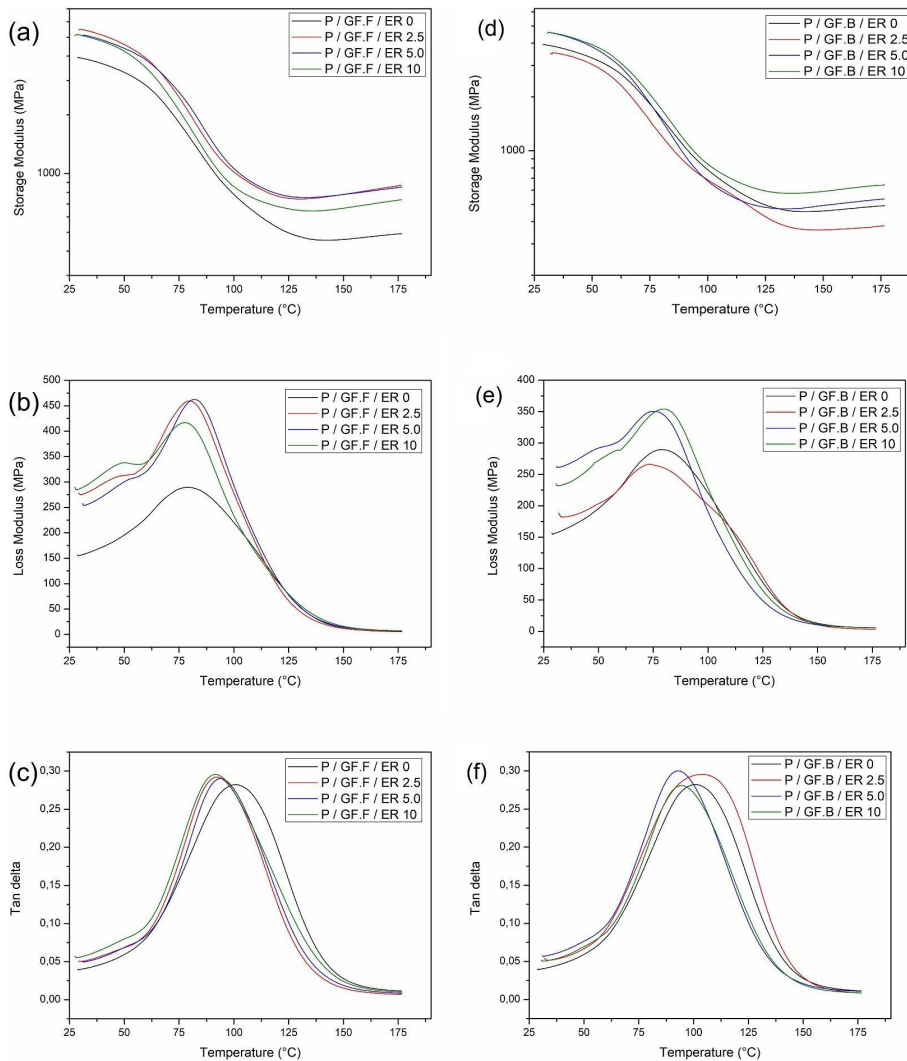


Figure 4. Glass fiber bidirectional fabric-reinforced polymer composites with (a) storage modulus, (b) loss modulus and (c) tan delta. Multidirectional non-woven blanket fiberglass composites with (d) storage modulus, (e) loss modulus and (f) tan delta.

4. Conclusion

In this study, the effect of the incorporation of the ER from flexographic photopolymer plates on the mechanical properties of two compositions of unsaturated polyester composites was evaluated. Contrary to previous observations, this residue did not noticeably affect the tenacity of the composites made by hand layup lamination. No significant variation in the void content was observed up to 5% ER. However, an increase in the void content was observed for 10% ER upon the incorporation of the filler. The lower impact strength of the samples associated to the presence of voids can be related to the manufactured process, which influence in the mechanical properties of the materials. The incorporation of the ER had a more pronounced effect on the composites produced with bidirectional fiberglass fabric than on those produced utilizing a multidirectional fiberglass blanket. In both compositions, a decrease in the impact strength was observed in the presence of the ER. No negative variations were observed in the flexural strength and dynamic mechanical analyses. DMA analysis

showed that the presence of the ER contributed to the composite glass transition temperature reduction because the ER particles were softer than the glass fibers, which promoted the segmental mobility of the polymeric chain at lower relaxation temperatures.

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