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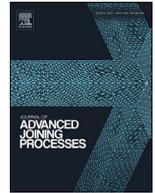
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Adhesion of epoxy and polyurethane adhesives in pultruded composite material

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ABSTRACT

An increase in applications of pultruded composite material has been noted in the last decades. The material is usually employed in sectors that require high mechanical properties as well as chemical and corrosion resistance. The pultruded profiles can be joined by mechanical fasteners (i.e. bolts) or adhesive bonding. Bonded joints present many advantages, such as low weight, design flexibility and corrosion resistance. However, an inherent property of the polymers is the low superficial energy, which tends to be detrimental to the adhesion quality of the structure. In this sense, is important to perform a proper surface treatment and select an adequate adhesive.

The present work investigates the influence of the adhesive type and surface treatment on the adhesion of pultruded composites. Three polyurethane adhesives and an epoxy adhesive were evaluated using tensile tests in bulk specimens. Then, the composite bonded joint was tested using single lap joints (SLJs). Two methods of mechanical abrasion were applied as surface treatment: unidirectional abrasion (UA) and bidirectional abrasion (BA). The treated samples presented rougher and more homogeneous surface profiles. Results revealed that the surface treatments improved the adhesion quality of the bonded materials compared to untreated samples. The high surface roughness produced by the BA together with a ductile adhesive resulted in the best improvement of the joint strength.

Introduction

Fiber reinforced polymers (FRPs) are composite materials made of a polymeric matrix reinforced with continuous fibers. The matrix is generally a thermoset or thermoplastic resin reinforced with synthetic fibers (glass, carbon or aramid) (Joshi, 2012) and, more recently, natural fibers such as jute, kenaf, hemp, coir bamboo, sugar palm and sisal. Studies has been developed in order to minimize the main drawbacks of biocomposites (Biswas et al., 2019; Sahu and Gupta, 2019). The fiber treatment of a pultruded biocomposite made of vinylester matrix reinforced with jute fiber improved the mechanical properties of the material (Ray et al., 2002).

The pultrusion is a continuous manufacturing process of FRP profiles with a constant section usually reinforced with glass fibers (GFRP) (Smith, 2007). These materials are applied in different sectors of industries, such as civil construction, aerospace and automobile, as an alternative for conventional materials (Stazi et al., 2015; Sundriyal et al., 2020). Fig. 1 shows applications of pultruded composites in structures. Fig. 1a shows an industrial shed for salt production with a total area of 21,000 m² (Pultrusão do, 2020). Mooring pillars and beams are made of

concrete and other elements are made of pultruded composites. Fig. 1b show stairs, guardrail and floor grid made of pultruded composites of a fertilizer factory in Brazil. No damage was found after three years of service life (Pultrusão do, 2020). Compared with steel, the high specific strength, light weight, corrosion resistance, low electrical and thermal conductivity represent substantial advantages. The main limitations of pultruded profiles are the initial costs that is usually higher than a similar structure made of steel (Stazi et al., 2015) and the lower fire and heat resistance. However, the higher costs can be compensated by the longer service life and the no maintenance required in the composite structure.

The correct joining method between pultruded profiles is an important factor in a project (Ungureanu et al., 2018a). Mechanical fastening and adhesive bonding are the main joining methods applied in GFRP structures (Ascione et al., 2016). Bonded joints present important advantages, such as a more uniform stress distribution between the substrates, high corrosion resistance and low weight (Heshmati et al., 2015; Ascione, 2009; Ascione et al., 2017). Additionally, the fibers are preserved, which favors the mechanical performance of the material.

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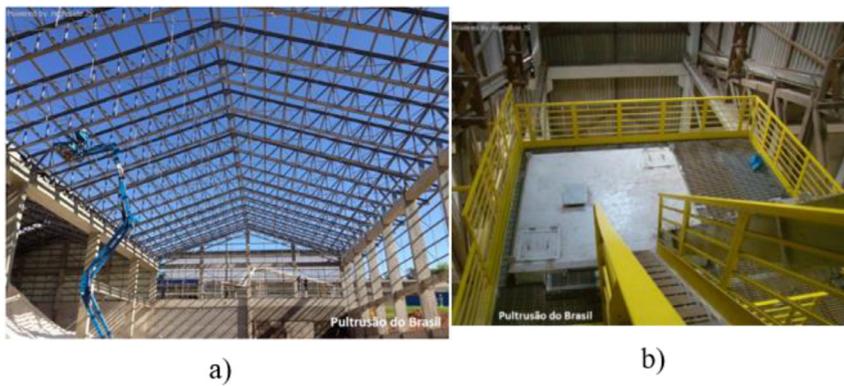


Fig. 1. Examples of pultruded structures: (a) elements of an industrial shed, (b) stairs, guardrail and floor grid (Pultrusão do, 2020).

Comparing with mechanical fasteners (i.e. bolts), bonded joints require lower investment (Keller and Tirelli, 2004; Zhang et al., 2009). However, the manufacturing process may induce residual stresses to the adhesive layer depending on the joint design (Günther et al., 2020). Different types of adhesives can be applied in bonding pultruded profiles, the most common being the epoxy adhesives (Stazi et al., 2015; Ungureanu et al., 2018a,2018b). The typical stiff and brittle behavior of epoxy adhesives allows them to withstand higher stresses while polyurethane adhesives usually tolerate lower stresses, present ductile behavior and inferior stiffness (Vallée et al., 2017).

In the manufacturing of pultruded profiles, some components are mixed to the resin matrix. The mold release is included in the resin in order to avoid friction with the oven, reducing the adhesion properties (Ascione, 2009). Therefore, surface treatments are indicated to improve the adhesion performance of the material. Plasma, acid etching and machining are some surface treatments used in polymeric substrates (Sundriyal et al., 2020). The mechanical interlocking is the adhesion mechanism more observed in pultruded bonded joints. The increase of the surface roughness provided by mechanical abrasion is shown to improve the intensity and density of the interlocking adhesion forces (Ungureanu et al., 2018a; De Barros et al., 2012). Since the superficial veil decreases the adhesion performance of the material, manufacturers recommend the use of a surface treatment (Ghumatkar et al., 2016). These works show that different surface treatments have significant effect on the mechanical behavior of composite bonded joints. Simple and reliable solutions for the optimization of the performance of bonded structures can be achieved by new combinations of different materials and surface treatments.

This work aims to investigate the influence of the adhesive type and surface treatment on the adhesion of pultruded composites. Three polyurethane adhesives and an epoxy adhesive were evaluated using tensile tests in bulk specimens. Then, the composite bonded joint is tested using single lap joints (SLJs). Two methods of mechanical abrasion were applied as surface treatment: unidirectional abrasion (UA) and bidirectional abrasion (BA). The performance of the bonded joints is evaluated.

Materials and methods

The materials and manufacturing process of bulk adhesive samples and single lap joints are described in this section.

Bulk adhesives

The mechanical properties of four different adhesives were evaluated using tensile tests: the epoxy P 110 and the polyurethanes E LTX, E 300 and E 1500, provided by *Masterpol*. Six bulk specimens of each adhesive were manufactured according to the standard ASTM D 638 (D638-01, 2002). Fig. 2 shows the manufacturing process, specimen geometry and

test set up. The section in the center of the specimens has 13 mm of width and 2.85 mm of thickness. The adhesives were produced according to the manufacturer instructions and cured for 24 h at room conditions. Tensile tests were conducted in an *Instron* test machine model 5966 with a load cell of 10 kN. Tests were performed at a constant displacement of 1.0 mm/min for a relatively slow (quasi-static) fracture.

Bonded joints

Single lap joints (SLJs) were manufactured in order to study the mechanical behavior of pultruded composite bonded joints using different adhesives. The composite plates were provided by *Pultrusão do Brasil*, made of an acrylic resin as matrix and glass fibers as reinforcement. The composite substrates were cut from a plate with 3 mm thickness, in accordance with standards ASTM D 1002 (D1002-10, 2019) and ASTM D 3163 (D3163-01, 2014). The substrates have 25 mm width and 101 mm length, as shown in Fig. 3a. Two different surface treatments were manually applied with sandpaper (grit 80): unidirectional abrasion (UA) in parallel with the glass fibers, and bidirectional abrasion (BA) with an additional abrasion in the perpendicular direction. After the surface treatment and prior to bonding, the substrate surfaces were cleaned with acetone in order to remove any grease or release agent remained from the previous processes. An adhesive thickness of 0.2 mm and an overlap length of 12 mm was applied, as illustrated in Fig. 3a. Fig. 3b shows the specimens manufacture and Fig. 3c shows the test set up. For each surface condition and adhesive five specimens were manufactured. Tensile tests were performed using an *Instron* test machine model 5966 with a load cell of 10 kN and a constant displacement of 1.3 mm/min, as recommended in ASTM D 1002 (D1002-10, 2019).

Results and discussions

This section presents the adhesive properties obtained from tensile tests in the bulk adhesive specimens, as well as the roughness resulted from the surface treatment of the pultruded composites. Then, the influence of the different adhesives and surface treatments in the performance of the bonded joints is evaluated in the SLJs.

Adhesive properties

Representative load-displacement curves of the adhesives are shown in Fig. 4. The tested adhesives presented remarkably different mechanical behavior. Individual load-displacement curves are then presented in Fig. 5. The P 110 epoxy adhesive (Fig. 5a) presented a fragile behavior and high mechanical properties, with the maximum stress of 17.17 MPa. The E LTX polyurethane adhesive (Fig. 5b) presented a ductile behavior and intermediate properties, with maximum stress of 11.73 MPa. The E 300 (Fig. 5c) and E 1500 (Fig. 5d) polyurethane adhesives presented ductile behavior but stresses significantly lower than the E LTX and the P

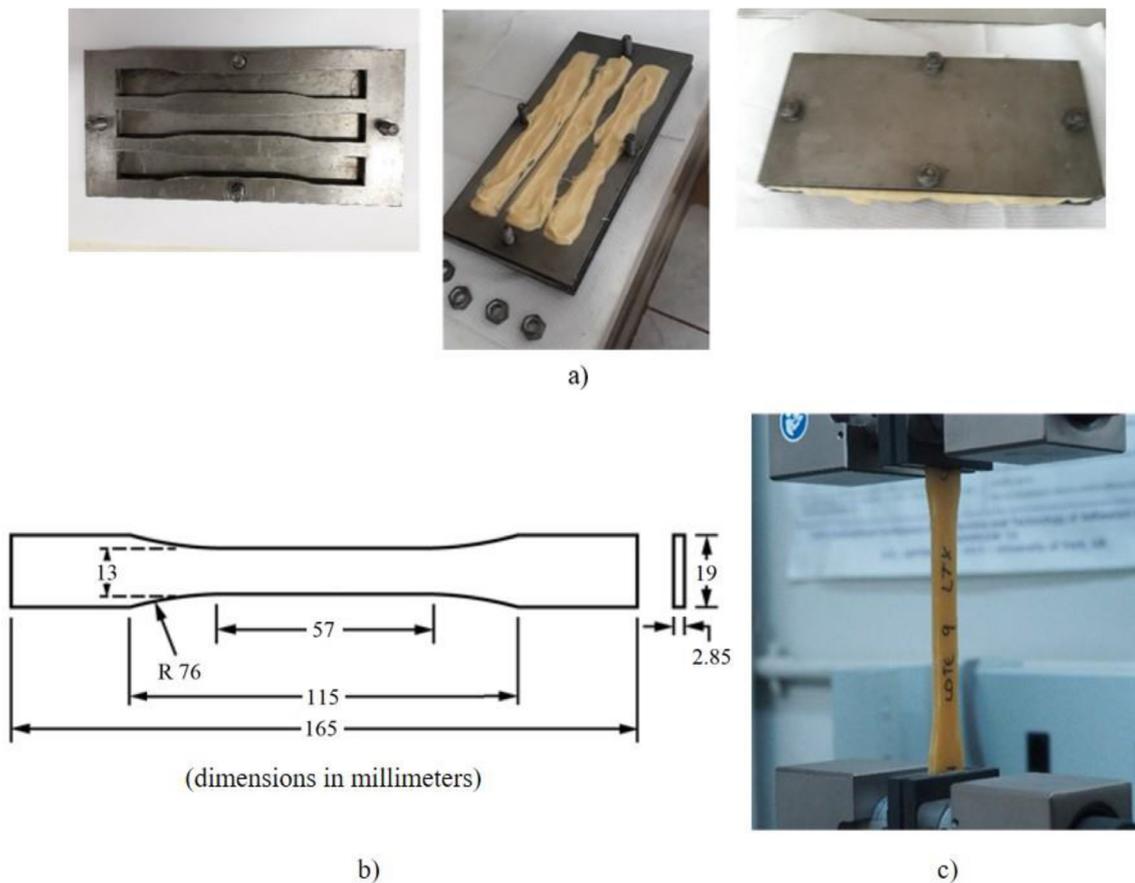


Fig. 2. Tensile tests in bulk adhesives: (a) specimens manufacturing, (b) geometry (dimensions in millimeters) and (c) test set up.

Table 1
Adhesive properties.

Adhesive	P 110 (epoxy)	E LTX (polyurethane)	E 300 (polyurethane)	E 1500 (polyurethane)
Maximum Stress (MPa)	17.17 ± 2.58	11.73 ± 1.01	1.128 ± 0.097	2.548 ± 0.093

110. The maximum stress is 1.128 MPa for the E 300 adhesive and 2.548 for the E 1500 adhesive. Therefore, the adhesives E 300 and E 1500 will not be tested in the bonded joints. The adhesive properties are shown in Table 1. No voids were observed in the tested bulk adhesive specimens. Samples that presented defects were discarded.

Surface roughness

In order to evaluate the effectiveness of the mechanical surface treatment applied, the surface roughness was analyzed using a 3D rugosimeter (Talyscan 150, Taylor Hobson). An area of 2 mm x 2 mm with a spacing of 1 μm (axis x) and 10 μm (axis y) was considered. The parameter Ra describes the average roughness of the profile, it means, the average between peaks and valleys (Rudawska, 2019). Fig. 6 shows the roughness profile of an untreated surface (Fig. 6a), and surfaces treated with unidirectional abrasion (Fig. 6b) and bidirectional abrasion (Fig. 6c). The bidirectional abrasion resulted in a more homogeneous profile. A similar length of the peaks in the different areas tends to enable a soft transition between the regions and improve the surface adhesion (Ungureanu et al., 2018a). The surface treated with BA presented considerably higher roughness compared to the UA and the untreated surface, as shown in Table 2.

Table 2
Average surface roughness.

Surface Condition	Ra (μm)
Untreated	0.961 ± 0.147
UA	1.433 ± 0.402
BA	4.005 ± 0.905

Single lap joints

The influence of adhesive properties and surface treatment on the strength of the pultruded composite bonded joints are discussed in this section.

Representative load-displacement curves of the tensile tests in SLJs are shown in Fig. 7. The SLJs curves load-displacement for untreated and UA treated samples shown a similar behavior and an increase in the maximum load with the treatment. For the SLJs treated using BA, the joints bonded with the P 110 adhesive (Fig. 7a) showed loadings higher than the untreated joints but lower than the joints treated using UA. In the case of SLJs bonded with the E LTX (Fig. 7b), the BA treatment resulted in higher loadings than the UA treatment.

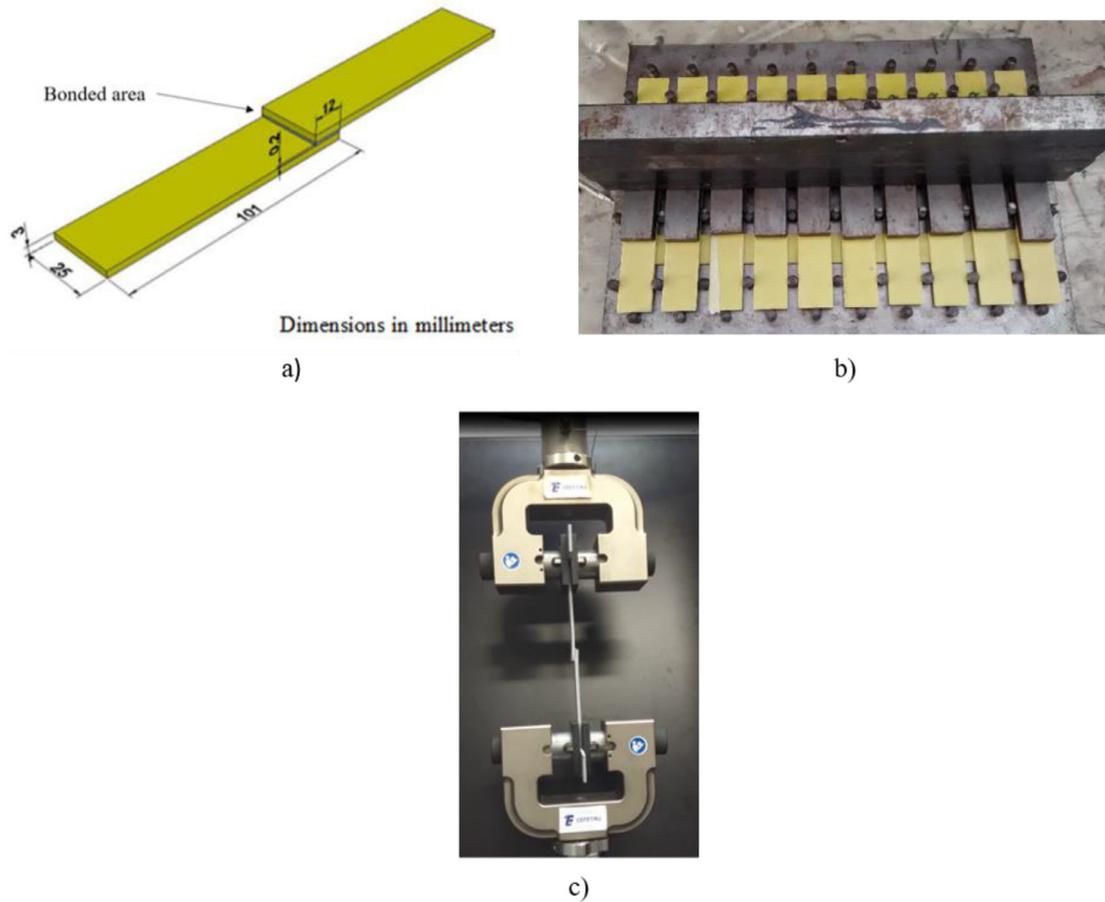


Fig. 3. SLJ (a) geometry (dimensions in millimeters). (b) bonding process and (c) test set up.

Table 3
Average test results of the tensile tests in SLJs.

Adhesive	Surface Condition	Maximum Stress (MPa)	Failure Mode
P 110	untreated	4.04 ± 0.88	Adhesive
	UA	10.4 ± 0.52	Cohesive and delamination
	BA	7.68 ± 0.68	Cohesive and adhesive
E LTX	untreated	4.48 ± 1.08	Adhesive
	UA	9.77 ± 0.96	Cohesive
	BA	11.5 ± 1.82	Cohesive

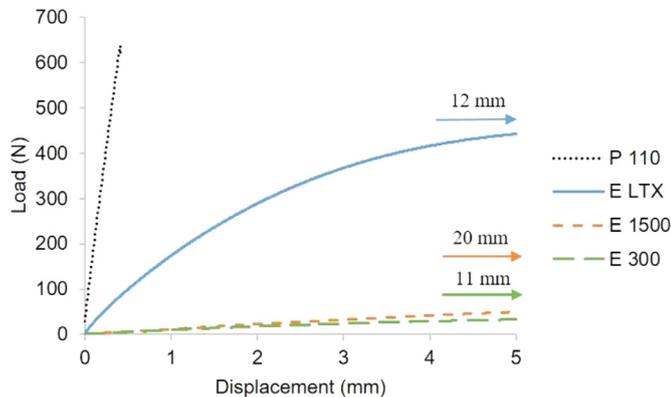


Fig. 4. Representative load-displacement curves of the tested adhesives.

adhesives treated using UA presented an increase in the shear strength of 157% and 118%, respectively. For the BA, the maximum stress increased 90% in joints bonded with the P 110 adhesive and 156% for joints with the E LTX adhesive. Results of the P 110 epoxy adhesive were lower than expected while the E LTX polyurethane adhesive presented superior results than usually found in the literature (Ungureanu et al., 2018a).

Fig. 8 shows the fracture surfaces of specimens bonded with P 110 adhesive. Samples manufactured with untreated surfaces (Fig. 8a) presented adhesive failure, indicating poor adhesion of the bonded joint (Ungureanu et al., 2018a). In the case of UA (Fig. 8b), cohesive failure is observed as the main failure mode. The cohesive failure is considered the ideal in tests of SLJs once the mechanical properties of the adhesive are considered in the analysis (Ungureanu et al., 2018a). Moreover, delamination failure occurred in the layer of the pultruded profile adjacent to the bonded interface. This failure mode is usually observed in the fracture of composite bonded materials (Arouche et al., 2021). For the samples treated using BA, the presence of adhesive failure in some areas decreased the joint strength.

Table 3 presents the average joint strength and the failure mode of each tested configuration. Specimens bonded with the P 110 and E LTX

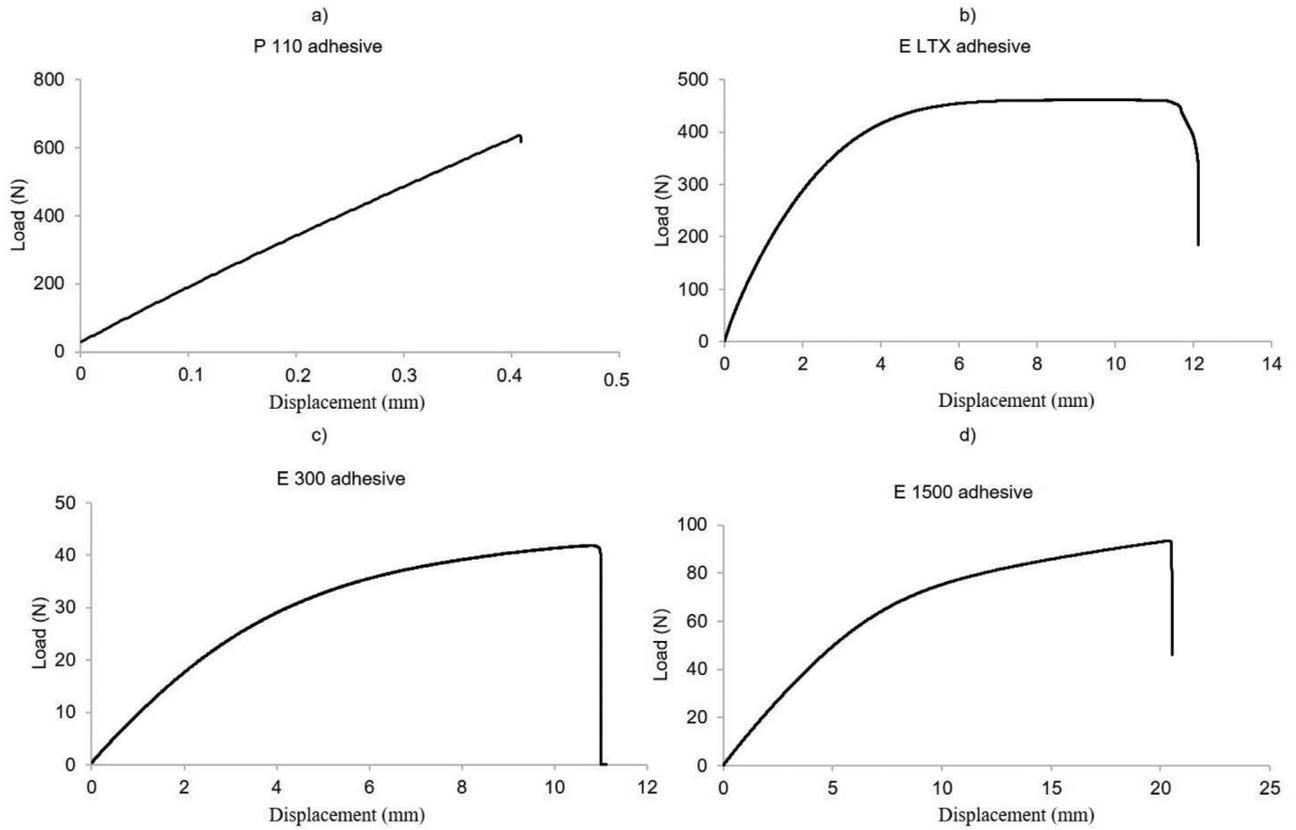


Fig. 5. Individual load-displacement curves of the (a) P 110 and (b) E LTX, (c) E 300 and (d) E 1500 adhesives.

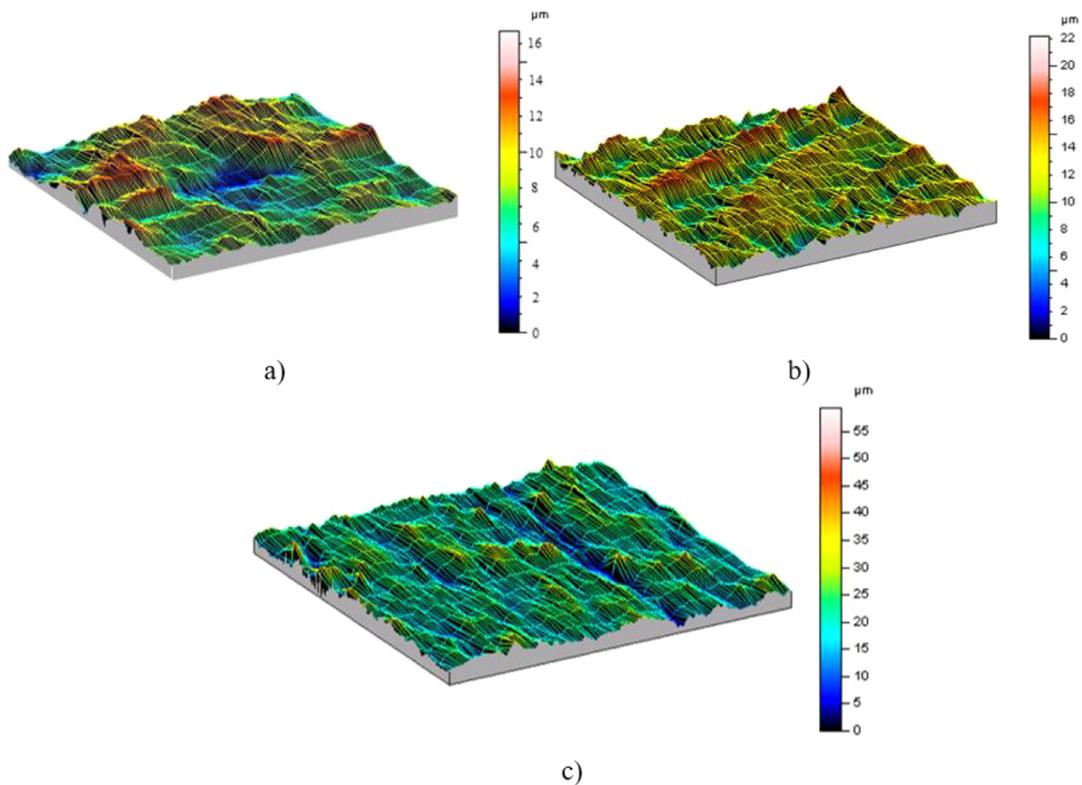


Fig. 6. Surface profile of (a) untreated surface, (b) treated with unidimensional abrasion (UA) and (c) treated with bidirectional abrasion (BA).

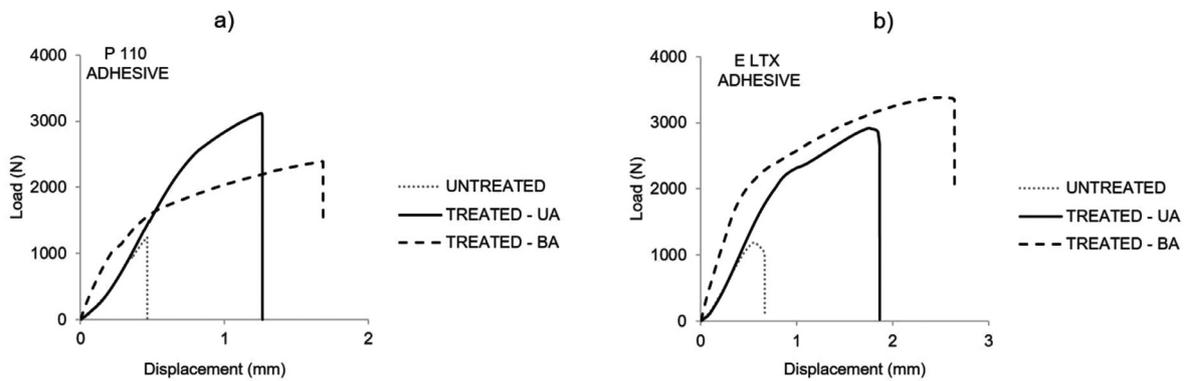


Fig. 7. Representative load-displacement curves of the tensile tests in SJLs with (a) P 110 and (b) E LTX adhesives.

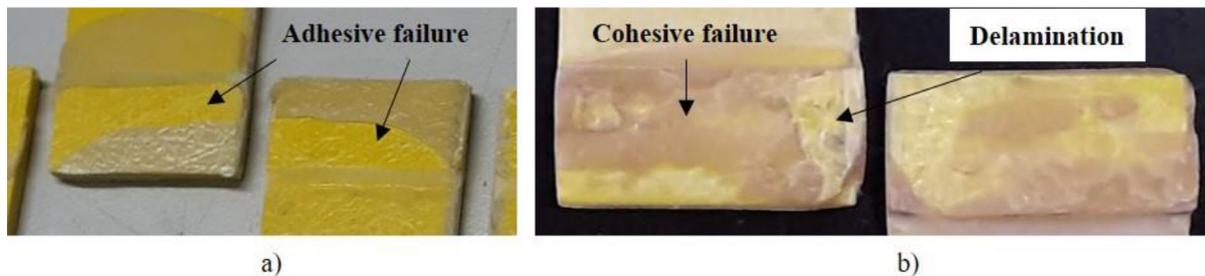


Fig. 8. Representative fracture surfaces of P 110 samples (a) untreated and (b) treated with UA.



Fig. 9. Representative fracture surfaces of E LTX samples (a) untreated and (b) treated with UA.

Fracture surfaces of E LTX samples are shown in Fig. 9. Specimens with untreated surfaces (Fig. 9a) presented adhesive failure, while the UA (Fig. 9b) revealed fracture surfaces with cohesive failure. These results are similar to the specimens bonded with P 110 adhesive and are considered a result of an efficient surface treatment (Ungureanu et al., 2018a; Ghumatkar et al., 2016; Guzanová et al., 2014). The surface treatment with UA improved the adhesion quality of the pultruded composites bonded joints, resulting in a change of failure modes and consequent improvement in the performance of the structure.

The fracture surfaces of the sample submitted to the UA and bonded with the P 110 adhesive was examined using an optical microscopy in order to analyze the delamination failure. Fig. 10 shows the fractography with a magnification of 6.3 times. The composite delamination occurred in the mat, closer to the longitudinal fibers.

Fracture surfaces of the bonded joints treated with BA are shown in Fig. 11. In these cases, cohesive failure was identified as the main failure mode. The presence of minor areas with adhesive failure in specimens bonded with the adhesive P 110 (Fig. 11a) decreased the joint strength. Specimens bonded with the P 110 adhesive produced an intermediate strength of 7.68 MPa, between the untreated samples and treated using UA. The fragile behavior of the P 110 (see Fig. 5a) may have contributed for a reduction of the mechanical anchorage in rougher substrates, as the presence of minor areas with poor adhesion is observed. In the case of samples bonded with the E LTX adhesive (Fig. 11b), tests resulted

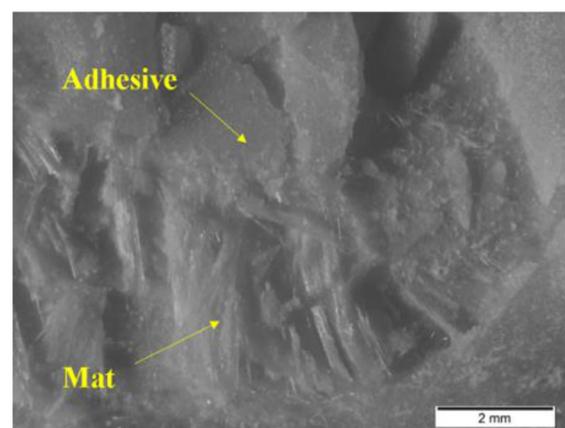


Fig. 10. Fractography (6.3x) of a delamination area in P110 sample.

in a joint strength of 11.5 MPa. The mechanical properties and ductile behavior of the adhesive (see Table 1 and Fig. 5b), together with the higher surface roughness produced by the BA, resulted in the highest strength among the tested configurations.

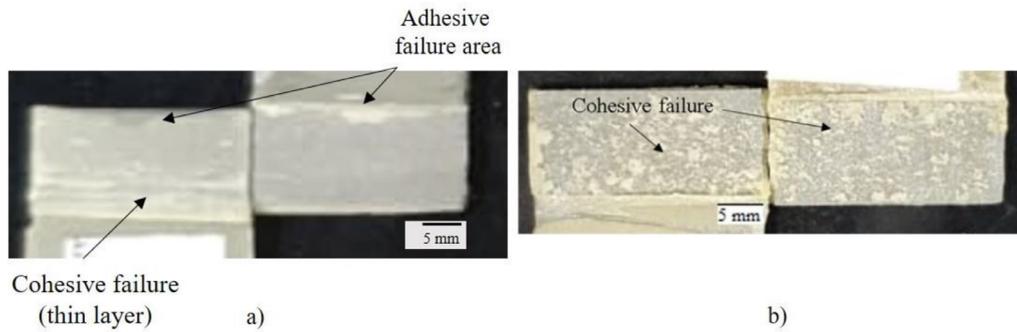


Fig. 11. Representative fracture surfaces of (a) P 110 and (b) E LTX adhesives treated with BA.

Stazi et al. (2015) obtained different results for SLJs bonded with epoxy and polyurethane adhesives. For bonded joints with an epoxy adhesive, results showed superior strength. In this research, a better performance of the joint was achieved with a polyurethane adhesive. This exemplifies how different parameters of substrate material, surface treatment and adhesive properties can affect the strength of a bonded joint (Stazi et al., 2015; da Silva et al., 2006).

Conclusions

The main objective of this study was to evaluate the influence of the adhesive type and surface treatment in the strength of pultruded composite bonded joints. The mechanical behavior of four adhesives were obtained by tensile tests. Unidirectional abrasion (UA) and bidirectional abrasion (BA) were applied as surface treatment. The roughness was evaluated using a 3D rugosimeter. Tests were carried out in SLJs and the strength and failure mode of the bonded joints were examined.

The P 110 adhesive presented a fragile behavior, while the E LTX, E 1500 and E 300 adhesives presented ductile behavior. The treated samples presented rougher and more homogeneous surface profiles. The increase of the surface roughness was more significant in samples treated using BA.

Tests on the SLJs revealed change in the failure mode with the surface treatment from adhesive to cohesive, indicating an increase in the adhesion quality. In bonded joints with the P 110 adhesive, higher stresses were achieved in the samples treated with UA, while bonded joints with the E LTX adhesive showed higher strength with BA. Both P 110 epoxy adhesive and E LTX polyurethane adhesive presented an increase higher than 150% of the joint strength. The E LTX adhesive showed better improvement than the P 110 adhesive as result of manual surface treatment. It is implied that the ductile behavior of the adhesive together with the higher surface roughness produced by the BA contributed to a more efficient mechanical anchorage of the E LTX polyurethane adhesive.

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