International Journal of Adhesion & Adhesives 103 (2020) 102701

Contents lists available at ScienceDirect

International Journal of Adhesion and Adhesives

journal homepage: http://www.elsevier.com/locate/ijadhadh

Study of the properties of an epoxy adhesive with additions of a residue from the biodiesel production process

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ARTICLE INFO

Keywords:
Residues reuse
Epoxides
Dynamic mechanical analysis
Lap-shear

ABSTRACT

In order to increase fuel supplies and reduce the gaseous emissions responsible for global warming, the production of biofuels has been stimulated. However, Biodiesel production leads to a huge amount of residues. The inappropriate discard of these residues generates an environmental problem. An important co-product of the transesterification process for biodiesel production is glycerol (also known as glycerin), representing 10% of the processed mass. Therefore, there are studies being carried out for the proper destination of glycerol. From these studies, one application of glycerol that deserves special attention is in the production of polymers. In this sense, this work studies the possibility of incorporating crude glycerol in epoxy adhesives, where fractions of 1.0%, 2.5%, 5.0% and 10.0% were added into epoxy. The tests carried out were tensile tests, adhesion tests and dynamic mechanical analysis (DMA), in order to evaluate the mechanical, adhesives and thermal behavior of the epoxy with these additions. In conclusion, small fractions of glycerol, like 1.0%, showed satisfactory results, in general, not decreased in adhesive properties; in opposite it leads to a small improvement with need more studies. With 2.5% and 5.0% of addition, more investigation is advisable. However, it was observed indications of improvement in properties, although less than those obtained with 1.0%. With 10.0% of addition there were sharp deterioration of properties, showing that use with 10.0% is not recommendable.

1. Introduction

In order to increase fuel supplies and reduce the gaseous emissions responsible for global warming, the production of biofuels has been stimulated. This growth of production also provides more opportunities in rural communities and develops a plan to replace fossil reserves, which are finite [1]. Biofuels can be used directly or through changes in internal combustion engines. Also, they partially or totally replace fossil fuels in other forms of energy generation.

Biodiesel is produced mostly from soybeans in Brazil. However, the sustainability of this production is questionable. The large landowners dominate the production of soy, to the detriment of small producers, concentrating the land [2]. These landowners have been responsible for the increasing deforestation of Brazilian native forests. Still, soybeans are grown with a high pesticide load, which results in contamination of the water [2]. On the other hand, in the quest to make biodiesel production more sustainable, we highlight the demand for the reuse of its waste. As a consequence of the increase in the production of biodiesel, there is a high amount of resulting co-products. Among the residues, we highlight the generation of crude glycerol, and the researches in search of ways to add value to this co-product [3]. The current market for the consumption of glycerol is specific and restricted, so it is essential to intensify the consumption of glycerol to make biodiesel production competitive [4]. Therefore, it is necessary to look for new technological and product routes for glycerol, besides its transformation, since the usual processes of purification are too expensive [5].

The high impurity level of crude glycerol limits its application, mainly in food and pharmaceutical industries, in which the glycerol from biodiesel can't be used for this reason. However, there are a growing number of studies for the application of glycerol as a raw material for polymer blends, mainly as plasticizing agent [4].

Polymers and polymer blends have been increasingly replacing traditional engineering materials such as metals, glass and wood. In addition, glycerol can be polymerized so as to form a thermoset polymer [5]. In this sense, it is justified to investigate possible applications for this by-product of biodiesel production. In the present work, this is done by incorporating the residue, the crude glycerol, into an adhesive based on epoxy resin. Epoxy-based adhesives have been modified to meet

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https://doi.org/10.1016/j.ijadhadh.2020.102701

market requirements, which seek to improve the properties of the final product. In this way it, as examples, it can be cited some works like FU et al. [6], also MARTINS et al. [7] and MARTINS et al. [8] and many other studies like SOARES et al. [9], BARCIA et al. [10], NGUYEN et al. [11]. In addition, one of the main use is the production of epichlorhydrin, which can be seen in SANTACESARIA et al. [12], KOČÍ1 AND LOUBAL [13] and production of liquid epoxy resin from epichlorohydrin.

The main objective of this work is to study the effects of the addition of biodiesel residue, glycerol in the epoxy resin, verifying the impact on its mechanical, thermomechanical and adhesive properties. For that, a methodology of characterization of these materials was used, with respect to its properties showing promising results with 1.0% of addition.

2. Materials and methodology

2.1. Epoxy resin based adhesive

The following materials were used, to prepare the specimens to tests:

- Base: MC 109 of EPOXYFIBER manufacturer;
- Hardener: FD 131 from EPOXYFIBER manufacturer;
- Fractions of biodiesel residue (glycerol).

The procedure for the manufacture of the epoxy resin is to mix the base, in this case the diglycidyl ether of bisphenol A (DGEBA), with hardener, the aliphatic amine, mixed ratio of epoxy and hardener 5:1, this fraction recommended by supplier - for the beginning of the polymerization process - for approximately 2 min with the aid of a spatula. The mixture occurs slowly, preventing the inclusion and entrapment of gases in the resin. This hardener has relatively rapid curing, which occurs at room temperature and low toxicity, according to the supplier. As said before, the mixing ratio is 5: 1 resin to hardener by weight. After mixing the clear viscous liquid base with the hardener, the biodiesel residue was added, noting that glycerol is miscible in epoxy forming a homogeneous mixture. The percentages of crude glycerol had been calculated in relation to the adhesive mixture (i.e. resin + hardener). Then, the mixture was poured into the molds. The dimensions of the test specimens in the DMA are 25 × 7 × 2 mm, in accordance with the standard established by the manufacturer of the equipment to be used. Attention should be paid to the regularity of these measures, since the analysis is very sensitive to variations. Also, samples may not contain: burrs, pores or any other types of structural defects [14]. The tensile test samples follow ASTM D 638, type V [15]. Prior to carrying out the tests, sanding of these samples is carried out in order to remove the burrs presented.

2.2. Preparation of biodiesel from soybean oil

Soybean biodiesel was obtained from the transesterification reaction. A reaction in which the triacylglyceride contained in the soybean oil is reacted with a preferably short chain alcohol in the presence of a catalyst. Thus, alkyl esters - biodiesel - and glycerol are produced. Generally, the reaction is conducted in excess of alcohol, to shift the equilibrium to the formation of the esters. The reagents used were: commercial refined soybean oil, 99.8% methanol from VETEC and, as catalyst, potassium hydroxide lentils with a purity of 85% from VETEC. The reaction was carried out at the Laboratory of Industrial Processes and Nanotechnology (LPIN) of UEZO, in an IKA RCT BASIC magnetic mixer reactor with integrated temperature control and agitation speed adjustment. The reaction procedure is explained as follows: 100 g of soybean oil was added to the reactor, maintaining stirring at 500 rpm and controlled heating to the temperature of 45 °C. At room temperature, 1% m/m of KOH was dissolved in methanol, the alcohol/oil molar ratio being 6, and then the solution was added to the reactor. Once a reaction time of 60 min was reached, the reactor was turned off for cooling. The mixture was transferred to a separating funnel and kept standing for 12 h, forming two phases. The upper phase (approximately 90%) contains mainly biodiesel and the lower phase (approximately 10%) is a glycerol phase. The glycerol phase that has been formed, also called crude glycerol, may contain 30–50% glycerol, and the remainders are impurities consisting of unreacted alcohol, catalyst and soap. Crude glycerol, being objective of the present work, was separated and adequately stored for later use.

Table 1
Physicochemical characteristics of glycerol [17]].

%C	%H	%N	%O	%S	%H2O	%CINZAS
32.8	8.6	0.2	54.6	0.9	12.5	3.8

Fig. 1. The plates positioned on the device.

The chemical formula of pure glycerol is $C_3H_8O_3$, [16]. Table 1 show physico-chemical characteristics of glycerol according to Ref. [17].

2.3. Crude glycerol and epoxy resin

For the purpose of this study, it is necessary to determine proportions of biodiesel residue to be added to the epoxy resin in order to obtain the final adhesive mixture. Thus, the quantities 1.0%, 2.5%, 5.0% and the maximum amount of 10.0% crude glycerol were added to the epoxy resin. Subsequently, the influence of each fraction of added residue on the mechanical properties of the adhesive was investigated.

2.4. Preparation of test specimens for adhesion test

The sample manufacturing methodology, previously described, was limited to the tensile and DMA tests, since the test specimens for adhesion test require special treatment. First, it was necessary to cut a 1/6″ steel sheet (approximately 1.59 mm) according to ASTM 1002 D [18]. Next, the chopped sheets were blasted with AG glass microspheres - between 53 and 105 μm in diameter at a pressure of 9 bar and at an angle of 45° for 30 s. The blasting machine used was the BBV7570/8 from *Blastibrás*. Subsequently, the plates were cleaned with acetone to remove residues. Then, one of the plates was positioned in the device shown in Fig. 1, so that the gluing was performed in an aligned manner.

The samples were prepared as described above (methodology identical to the samples from the tensile tests and DMA). However, instead of being subsequently placed in a curing mold, the elaborate adhesives were placed on the surface of the plates affixed to the device. Subsequently, another sheet was placed on the adhesive, so as to finalize the lap shear adhesion test specimens conforming to ASTM D1002 for metal substrates. This device, shown in Fig. 1, ensures uniformity of the thickness of the adhesive joint, a prime factor for a correct analysis by the adhesion test.

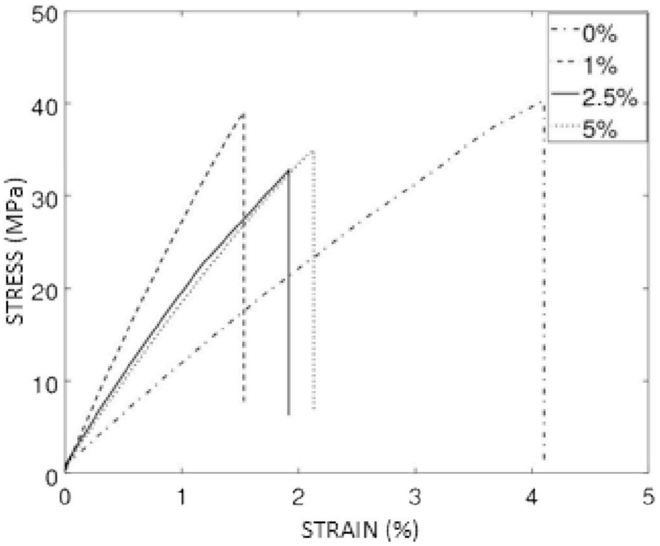

Fig. 2. Comparison of the different results of tensile tests: epoxy with different amounts of glycerol.

3. Tests carried out

For the analysis of the mechanical properties of the epoxy adhesives with and without addition of glycerol, the following tests were performed:

- Tensile test;
- DMA (Dynamic-Mechanical Analysis);
- Adhesion Test (“Lap Shear").

3.1. *Universal Testing Machine*

Two distinct types of tensile tests were performed using the Universal Testing Machine, brand INSTRON, model 5966, at the CEFET/RJ Adhesion Laboratory. For that, a 10 kN cell was used. The first type of test was the tensile test, according to ASTM D 638, Standard Test Method for Tensile Properties of Plastics. These tests were conducted until fracture of the samples. The second was the adhesion test, following the specification of ASTM 1002 D, using simple shear joints (also known as Single Lap Joints). These tests were also conducted until the fracture of the samples, in this case, the fracture of the adhesive contained between the sheets of steel.

3.2. *Dynamic mechanical analyzer (DMA)*

The equipment in question is the PerkinElmer DMA 8000 model, available at the COPPE/UFRJ LAVI Laboratory. The Tests were carried out to obtain the glass transition temperature (Tg) The glassy transition occurs in amorphous materials, which is the reversible transition from a rigid (vitreous) state to a soft, rubbery state [19].

Temperature sweeps from the room temperature (25 °C) to 100 °C were performed. A heating rate of 3 °C/min and a frequency of 1 Hz was used. The glass transition temperature of a material is of paramount importance because it is a property that defines their application [20]. The following proportions of glycerol were analyzed: 1.0%, 2.5% and 5.0%.

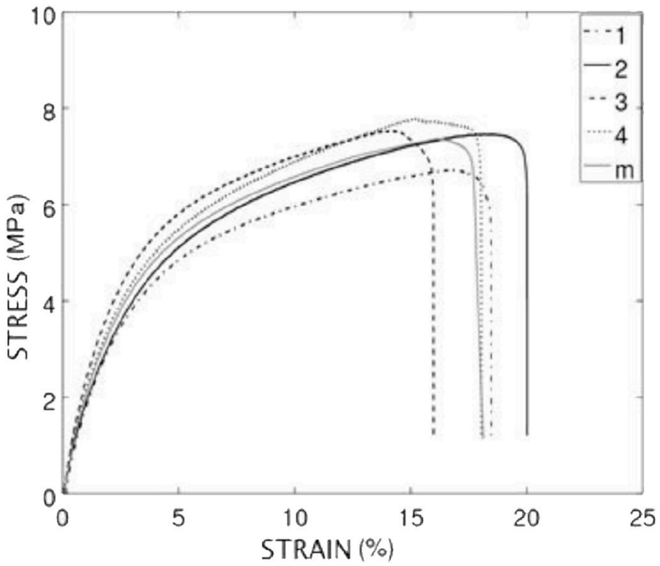

Fig. 3. Stress versus strain of epoxy +10.0% (glycerol), specimens 1, 2, 3, 4 and the average of the 4 results (m).

Table 2
Mechanical properties of the epoxy-based and crude glycerol modified Adhesive.

Materials (Epoxy + % Glycerol)	Elastic Modulus (GPa)	Elongation at break (%)	Strength Limit (MPa)
Epoxy + 0.0%	1.44 ± 0.11	4.10 ± 0.15	40.30 ± 0.34
Epoxy + 1.0%	2.85 ± 0.16	1.53 ± 0.16	39.10 ± 4.93
Epoxy + 2.5%	2.03 ± 0.32	1.92 ± 0.49	32.79 ± 4.38
Epoxy + 5.0%	1.87 ± 0.03	2.13 ± 0.05	34.96 ± 0.22
Epoxy + 10.0%	0.35 ± 0.04	16.15 ± 1.89	7.37 ± 0.46

4. Experimental results and discussions

4.1. *Tensile tests*

Results of the tensile tests are presented with the stress versus strain curves in the following Figs. 2 and 3. These were performed at a constant velocity of 2 mm/min in the equipment described above using the 10 kN load cell at room temperature. Samples were analyzed without crude glycerol addition and with: 1.0%, 2.5%, 5.0% and 10.0% of crude glycerol. Adhesive without the addition of the residue from now on will be called pure. Firstly, three epoxy samples with 1.0% glycerol were tested, for subsequent averaging of the curves obtained. Then, four epoxy samples with 2.5% glycerol were tested, from which the mean was also obtained. Subsequently, samples were tested with 5.0% glycerol. Finally, epoxy samples with 10.0% crude glycerol were tested, which the average was also performed. The results were Compared and presented Fig. 2, which represents the mean of the results of the pure epoxy adhesive and the adhesive with crude glycerol addition until 5.0% of addition. However, very large difference were observed in relation to specimen with 10.0% of crude glycerol, there are a marked decrease in the strength limit of the material (from 40.30 to 7.37 MPa) and a significant increase in elongation at break (about 4%–16%). Therefore, this result was not included in the graphical comparison of the adhesive for to better visualize of the other adhesives with different fractions of crude glycerol. From the curves obtained with the tests of the samples with different fractions of crude glycerol, the mechanical properties of the adhesives obtained are later presented in Table 2.

From the analysis of Figs. 2 and 3 and Table 2, the following observations are made:

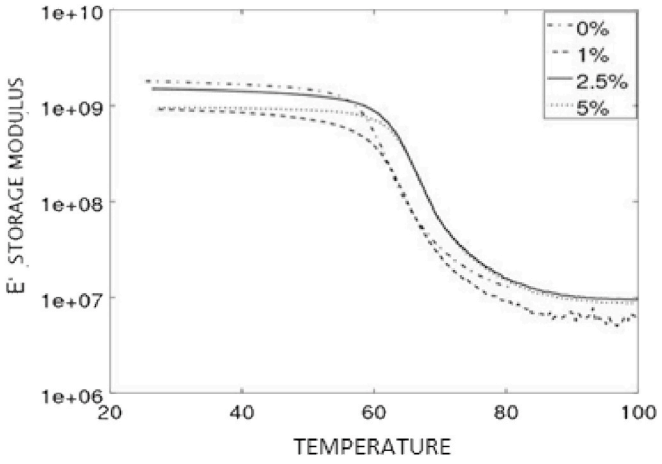

Fig. 4. DMA test, storage modulus E′ versus temperature.

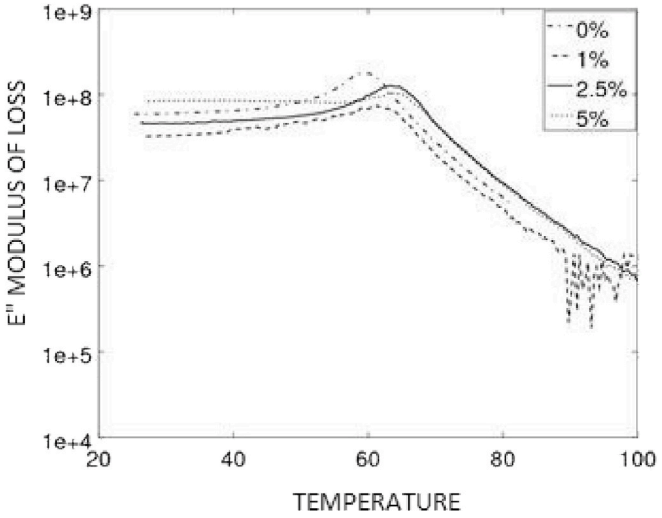

Fig. 5. DMA test, loss modulus E″ versus temperature.

- Epoxy with 1.0% glycerol: The reduction of the tensile strength is insignificant when compared to pure epoxy. However, there is an increase in the modulus of elasticity, that is, the material becomes less deformed with the same applied load. The proportion of glycerol placed resulted in an increase in stiffness of the adhesive;
- Epoxy with 2.5% glycerol: There was a decrease in the value of the strength limit in tensile test. Also, the elastic modulus is greater than that of pure epoxy and less than with 1.0%. Also a positive result was obtained; giving indications that it is feasible to use up to this fraction.
- Epoxy with 5.0% glycerol: The modulus of elasticity continues to decrease, but is still greater than that of pure epoxy. In comparison with the 2.5% ratio, there is an increase of the tensile strength limit and also the elongation of rupture. However, it should be interesting to have more number of tests with this fraction.
- Epoxy with 10.0% glycerol: The value of the strength limit in tensile test has a marked decrease when compared to any other analyzed proportion. The proportionality region becomes reduced and the modulus of elasticity is very small in relation to the others. However, there is a considerable increase in elongation at break.

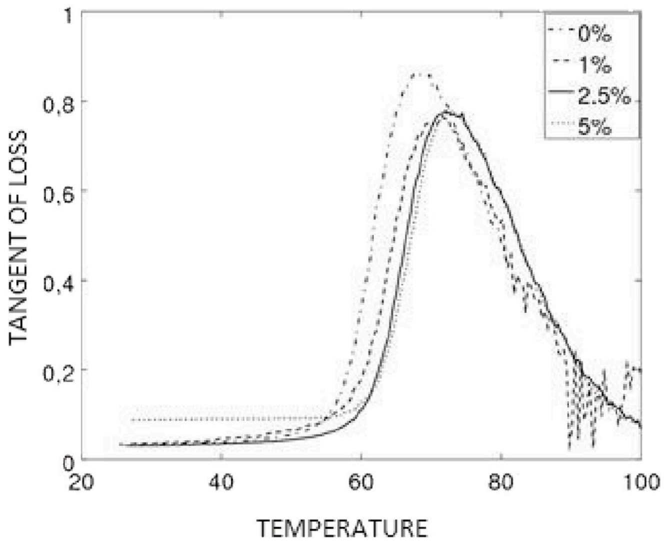

Fig. 6. DMA test, loss tangent versus temperature.

Table 3
Results obtained from the DMA Tests.

MATERIALS (epoxy + %glycerol)	Tg (°C)	Loss Tangent
epoxy + 0.0%	68 °C	0.86
epoxy + 1.0%	71 °C	0.77
epoxy + 2.5%	72 °C	0.78
epoxy + 5.0%	73 °C	0.78

4.2. Dynamic mechanical analysis (DMA) - glass transition temperature (Tg)

The following proportions of glycerol addition were analyzed: 0%, 1.0%, 2.5% and 5.0%.

The results of tests are presented in Figs. 4 and 5 e 6. Fig. 4 shows that the percentages 2.5% and 5.0% take longer to enter the transition region from the vitreous to the rubbery state than the other samples. This means a decrease in the rotational capacity between the chain sequences [21] introduced by these load quantities. The imaginary part of the complex modulus of elasticity, also called the loss modulus, is shown in Fig. 5.

By comparing the loss tangent curves of the adhesives tested, as observed in Fig. 6, certain analyzes are performed. Initially, it is important to note the presence of a single peak in all curves, including those of the loaded adhesive. This shows that, in the analyzed temperature range, there is no transition beyond the glassy one, that is, the material is behaving as a single phase material. The only peak - of each curve - observed in Fig. 6 is the glass transition temperature (Tg) of the adhesive.

Table 3 shows the values of Tg for the analyzed materials, as well as the value of the loss tangent in Tg.

From the analysis of the data in the previous Table 3, the impact of the amount of crude glycerol on the adhesive can be identified, and whether or not it acted as a plasticizing agent. The plasticizer is installed between the chains of a polymer so as to separate them, thus expanding the free volume of the polymer [19]. With this expansion, the degree of freedom for the movement of lateral groups of the polymer molecules grows, with rotational movements occurring [20]. Thus, the effect of a plasticizer on the glass transition temperature of a polymer, then, is its reduction. Also, the material becomes softer and more flexible. Observed in the tests, contrary to what is expected, a gradual increase in the glass transition temperature - although small. Therefore, it is concluded that, in these proportions, the glycerol did not act as a plasticizer, but may

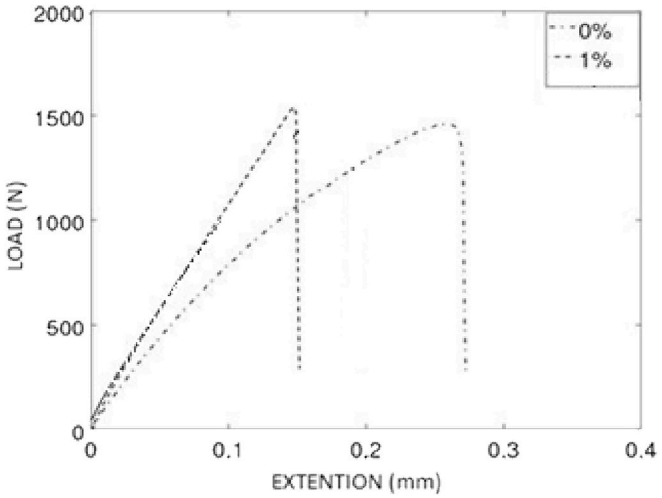

Fig. 7. Comparison of different results of the lap shear tests with 0% and 1.0% of crude glycerol.

have increased the density of the final product, which would explain the phenomenon observed. Glycerol addition also generated a decrease in the material loss tangent, which means a reduction in energy dissipation at this temperature.

4.3. Lap shear test

Lap shear test was performed at a constant speed of 2 mm/min at room temperature. Samples of: pure epoxy (epoxy+0%) and epoxy with the proportions 1.0% of glycerol were tested. Samples with 1.0% were chosen because with this addition were obtained better results in tensile tests.

The average values were compared as shown in Fig. 7. These graphics show the change in the behavior of the adhesives due to the addition of the additive (crude glycerol). While in the pure epoxy there is a flow before rupture, characteristic of a ductile fracture, the rupture occurs abruptly in the samples modified with crude glycerol.

There was prevalence of cohesive failure in both adhesive bonds, with 1.0% and without residue addition, as showed in Fig. 8.

From the results obtained in these tests, the adhesion strength limit of the pure epoxy adhesives and also with 1% of crude glycerol were obtained, as seen in Table 4.

Analyzing Table 4, we can observe that the results are close to each other. The difference between the strength limit in the epoxy lap shear test without the addition and with the addition of the glycerol waste is within the error track of the two results obtained, therefore, there is no noticeable impairment of the adhesive properties of the epoxy. We note, then, that the inclusion of glycerol does not present indicative changes in the limit of strength at adhesion with 1.0% of addition.

4.4. Discussion

In the present work, it was proposed to carry out the evaluation of the impact of the incorporation of a residue of the biodiesel production into an epoxy adhesive. Using the described methodology, experimental results were obtained, and from these, it is possible to consider the follow comments.

- 1.0% crude glycerol: the introduction of glycerol into the epoxy has made it stiffer, as said before. There was no significant drop in the strength limit of the pure adhesive. The Tg of the adhesive became larger, but not so much. In terms of the lap shear strength it did not lead to a considerable variation, which is a very positive result. It may even suggest a slight improvement, but that would have to be confirmed with further investigation.
- 2.5% crude glycerol: as observed, the adhesive strength limit was decreased compared to the result with 1.0% glycerol of addition; there was a decrease in the modulus of elasticity, which remains greater than that of the unfilled epoxy.
- 5.0% crude glycerol: The modulus of elasticity continues to decay, but it is still larger than that of epoxy without glycerol, showing a trend. The strength limit of this fraction is higher than 2.5%. However, it is still less than 0% and 1.0% of glycerol. In the DMA analyzes, the highest loss factor at room temperature is noted. A positive result is seen in the addition of 5.0% glycerol.
- 10.0% crude glycerol: This percentage of glycerol depleted too much the properties of the adhesive analyzed. A drastic drop in the adhesive strength limit was observed as well as in the modulus of elasticity. A high increase in maximum sample elongation was also observed. Due to these results in this proportion of residue addition, the other tests were not performed on it.

5. Conclusion

Based on the results obtained in the tests performed on the samples with each fraction of glycerol, we can conclude that:

One percent of crude glycerol addition to the epoxy did not decrease the adhesion ability capacity of the adhesive, which is very interesting in the reuse of this residue.

With two and a half and Five percent of crude glycerol addition in the epoxy resin presented increased the stiffness of the adhesive and decreased the ductility, and it can be undesirable when used to prepare some composite material. However, in others applications it may be interesting. Though, more investigation is advisable. With ten percent of crude glycerol addition in the epoxy resin there was a decrease in mechanical properties, showing that use with ten percent is not recommended. With this fraction, the adhesive begin to present decrease of mechanical properties and become an unfavorable factor to use in the composites preparation. Glass transition temperature increase is also a positive result, despite the relatively small increase, as it makes the adhesive more resistant to temperature increase.

Table 4
Strength limit obtained from the lap shear test.

Materials	Lap Shear Strength(N)
Epoxy + 0%	1459.8 ± 421.6
Epoxy + 1.0% of crude Glycerol	1540.5 ± 210.4

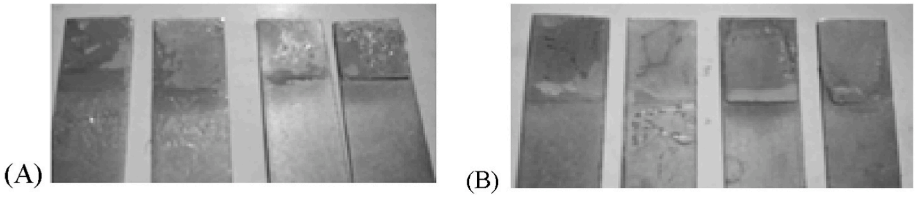

Fig. 8. Prevalence of cohesive failure in samples without addition (A) and with 1.0% of residue addition (B).

Acknowledgments

The authors wish to thank Professor Silvio de Barros (CEFET-RJ) and the student Brenno Duarte (CEFET-RJ) for their cooperation in the adhesion tests, and to Professors Lavinia Borges and Daniel Castello (COPPE/UFRJ), for the opportunity to use the DMA, also to the employees and students of the Laboratory of Industrial Processes and Nanotechnology (LPIN/UEZO).

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