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COB-2019-1139 ANALYSIS OF THE INFLUENCE OF THE CURE AND POST-CURE PROCESSES ON FIBERGLASS COMPOSITES

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Abstract. *Composite materials have become increasingly relevant in different applications, especially in the aerospace field. They are increasingly present in the production of internal components and external ribs of wings in aircraft, leading edge, flaps, landing gear doors, radome of cargo aircraft, structural parts of fighters jet, among others. This article aims to present the analysis of the influence of the cure and post-cure processes of specimens made with glass fiber and polyester resin; to perform post-cure in certain types of specimens; to perform mechanical tests and to analyze the results of the specimens. Four types of specimens will be manufactured, differentiated by the cure and post-cure processes. To manufacture the specimens and subject them to the curing process, a laminating bench with temperature control and vacuum use was developed. An autoclave developed at the University of Brasilia was used to perform post-cure treatment. The tests showed different mechanical performances of the specimens and showed that cure and post-cure provide increased mechanical properties of the material.*

Keywords: *Composites Materials, Cure, Post-Cure, Fiberglass, Laminate.*

1. INTRODUCTION

Composite materials, manufactured by curing and, in many cases, post-curing processes, have increasingly been considered for applications, mainly in the aerospace industry because of their properties. The use of these materials in the aerospace segment has been growing about 5% a year since the 60's, since this type of material allows lighter and more resistant structures, besides guaranteeing flexibility to a certain chosen part. Continuous fiber composites with thermorigid matrix are being used to obtain internal and external components, wing ribs in Boeing aircraft, landing gear doors, leading edges, flaps, cargo plane dome, fighter structural parts, among others (Resende 2000). Therefore, studies that can broaden knowledge and greater mastery of these materials and their nature, such as the present work, are relevant.

Composite material is a new class of materials derived from the chemical and structural combination of at least two mutually insoluble materials. Once combined, these materials have distinct properties of their components when analyzed separately. In microscopic scale the constituents are inherently heterogeneous, but they can be considered homogeneous in macroscopic scale (ASTM D3878, 2018).

Such materials consist of a matrix, which is responsible for grouping the components and can be ceramic, metallic or polymeric, and a reinforcement that can be ceramic, metallic or polymeric, synthetic or natural (Strong, 2008). The present work uses polyester resin matrix and fiberglass reinforcement (synthetic reinforcement).

In the manufacture of composite materials it is necessary to subject them to cure and post-cure processes. Curing is the stage of laminate consolidation, in which temperature variations occur, changes in physical state, changes in density viscosity and the appearance of residual stresses, in which the reaction is performed by the hardener. The thermal profile to which it is subjected is called the curing cycle, having several heating phases (Martinez, 2011). The post-cure process is a variation of the curing cycle and aims to achieve the final properties of the polymeric matrix with the highest degree of curing (Martinez, 2011), in addition to using the temperature, which becomes common in this type of process, in order to achieve the mechanical and chemical stability of the material (Martinez, 2011).

In this paper, which focuses on the curing and post-cure processes, the influence of these curing and post-cure processes of specimens made of glass fiber and polyester resin was analyzed. In order to achieve the objectives, it was necessary to develop a methodology for the manufacturing process of the samples. For such, it was necessary to perform the curing in certain types of specimens on a lamination bench developed by the authors, and post-cure in certain types of specimens in an autoclave developed at the University of Brasilia. The analysis of the influence of curing and post-cure were made from the results of tensile tests applied to four configurations of specimens. An impulse excitation assay was also performed, but the results were inconclusive, so they were discarded from the analyses.

2. MATERIALS AND METHODS

2.1 Laminate cure

Curing is the stage of laminate consolidation, in which temperature variations occur, changes in physical state, changes in density viscosity and the appearance of residual stresses, in which the reaction is performed by the hardener. The thermal profile to which it is subjected is called the curing cycle, having several heating phases (Martinez, 2011). The post-cure process is a variation of the curing cycle and aims to achieve the final properties of the polymeric matrix with the highest degree of curing (Martinez, 2011), in addition to the use of temperature, which becomes common in this type of process, in order to achieve the mechanical stability of the material (Martinez, 2011).

The curing process of the matrix is an exothermic reaction of free radical polymerization. The resin changes from a liquid to a vitreous state due to the formation of cross-links during chemical reaction. Addition of heat during curing catalyses the process, increases the speed of polymerization and favors the formation of cross-links (Strong, 2008). During this process, gases are released that generate bubbles between the fiber and resin layers. The existing voids caused by the bubbles act as tension concentrators and compromise the strength of the material, due to this fact is commonly the use of vacuum bag for the process associated with temperature application (Callister, 1991).

The post-cure process is a technique that has the purpose of raising the degree of curing of the laminate by adding heat and pressure (in some cases). This process enhances the polymerization reaction, that is, there is a greater formation of cross-links that consequently decrease the amount of residual monomers, which in turn increase the crystallinity of the polymeric matrix and make it more rigid. The resin is the part of the composite that is exposed to the elements of the environment and is responsible for absorbing mechanical stress and transferring it to the reinforcement. This way if the polymeric matrix becomes more crystalline, the laminate becomes more resistant (Strong, 2008).

The increase in the level of cure of the composite implies greater stiffness to the laminate, greater chemical and mechanical stability, greater bending stiffness and higher glass transition temperature (Spigosso, 2017). For this purpose, it is necessary to carry out post-curing in an autoclave that is a greenhouse that can be pressurized or not, in which the material is introduced into a chamber and exposed to a controlled and homogeneous atmosphere at the glass transition temperature of the polymer (Strong 2008).

2.2 Autoclave

Initially, the dimensions of the autoclave were defined so that various types of samples can be cured, with respect to different geometries and volumes, that is, the equipment must be safe, cheap and with considerable volume. After defining the dimensions of the chamber, it was decided to use a 35-liter industrial pressure cooker (Fulgor Express) for adaptation as an autoclave, eliminating the need to manufacture a pressure vessel.

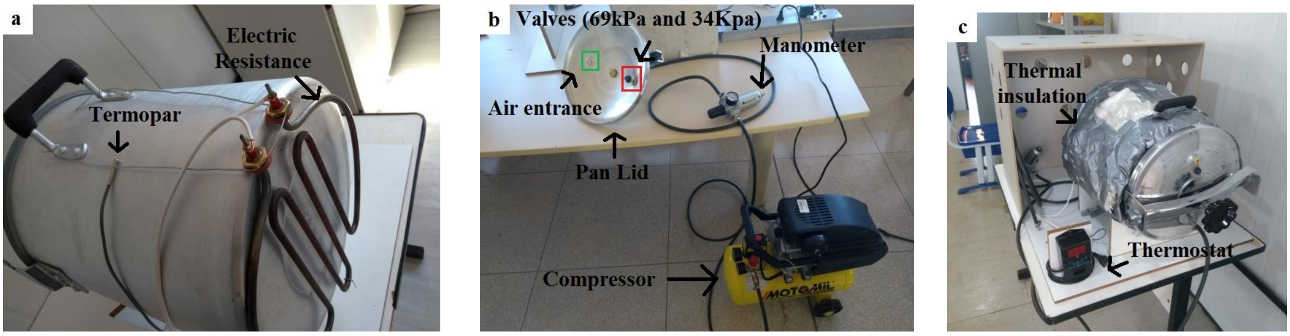

Figure 1: (a) Electrical resistance fixed to the pressure pot. (b) Autoclave pressurization system. (c) Autoclave Adapted from Dias, 2018.

The pan, seen in Figure 1a has a pressure support system between the lid and the body, has four safety valves that work at different gauge pressures: two that operate with 69 kPa, and one that operates at 103 kPa and the work valve that supports up to 34kPa. According to the manufacturer's information, its maximum supported pressure without damage is 172 kPa. Then, the operating range of the pan was set with an upper gauge pressure limit of 69 kPa and a temperature of 120 ºC. In order to adapt the industrial pot for use as an autoclave, it was necessary to assemble all the required systems to make the equipment a pressure vessel capable of carrying out curing processes with efficiency, control and safety. The main systems to be developed are: heating, pressurization and insulation.

Temperature control was carried out by a thermostat (Droyd brand). Its control is done from the on or off system with an electric relay that closes and opens the circuit from the temperature measured at the thermocouple and the operating temperature can range from -20 °C to 120 °C. For the determination of the resistance power it was considered a variation of the ambient temperature of about 25 °C up to the maximum working temperature of 120 °C with a heating ramp of 3 ºC per minute to cure an object of 25 kg of mass, so it was chosen a commercial resistance with maximum power of 2.5 kW (Dias, 2018).

The design of the pressure cooker used as an autoclave is in accordance with the ASME standard section VIII division 1 of 1986, which specifies calculations of hull thickness subject to internal pressure. The pressurization system was assembled by adapting the pan from its pressure regulating valve and its safety valves. To increase the pressure inside the pan, an air compressor with a maximum pressure limit of 1000 KPa (Motomil brand) was used, connected by a hose to a pressure regulator air filter (Worker brand) that aims to precisely adjust the pressure value applied to the system. The compressed air reaches the pan through the duct of its working pressure regulating valve (34 kPa gauge pressure), located near the central part of the lid, and the piping that exits the compressor is connected to this inlet. If the pressure in the autoclave is increased undesirably, there are two safety valves that operate at gauge pressures of 69 kPa and one of 103 kPa that can be actuated. The working pressure is then limited to 69 kPa.

The insulation was divided into two parts, one fixed to the outer wall of the pot hull and was built from a structure formed by a sandwich with an outer surface of fiberglass blanket (1 mm thick and 0.12 g/cm2 granulometry) fixed with heat-resistant adhesive tape plus a rock wool core with 50 mm thick (Dias, 2018). The other part of the insulation is also a sandwich structure, similar to the first, better adapted to the lid of the pan, being positioned on the inside.

2.3 Lamination bench

To perform the curing, a laminating bench was built with temperature control and vacuum use. The bench has been assembled from low cost materials so that they can be easily purchased in order to reduce the cost of construction and maintenance. The dimensions were defined from the autoclave so that curing could be carried out on the bench and, if necessary, after curing in the autoclave. Initially, the countertop was built from a glass plate in conjunction with a marble base to support the resistance. The material, however, proved inadequate as the glass broke due to exposure to the heat emitted by the resistance.

For the construction of the definitive structure, two galvanized zinc plates of 500 mm x 600 mm x 3.5 mm were used. Zinc was chosen because it has great stiffness, low thermal conductivity and high specific mass when compared to other metals available in the market, having adequate characteristics to store energy and be used on a surface that needs to have structural strength and a homogeneous temperature distribution. The bottom plate was attached to an MDF (medium-density fibreboard) of the same dimensions and thickness of 10 mm, which besides being an accessible material and having a low thermal conductivity, has structural stiffness to be used as a base. In order to create a heated environment through the use of an electrical resistance and facilitate its handling, four large threadable screws were used as spacers and placed through holes in the corners of the bottom plate, which were fixed by nuts. The upper plate was placed on the lower plate in the bolt housing, and was also immobilized by nuts. A height of 60 mm was left between them to allow natural convection heat transfer and radiation. A third galvanized zinc plate was introduced to the gap between the first two just to serve as a support for the resistance.

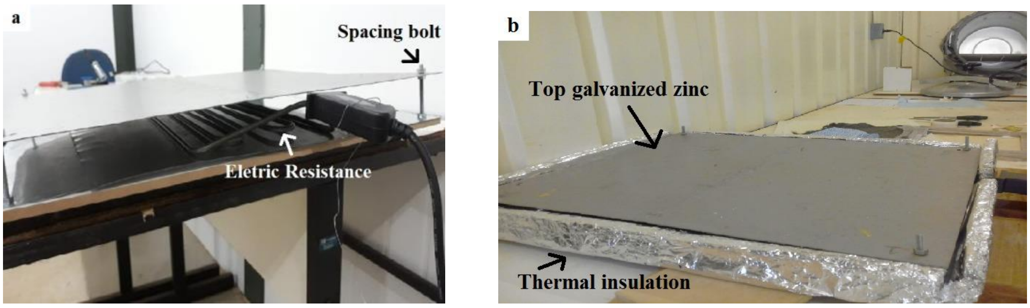

Figure 2: (a) Electrical resistance between galvanized zinc plates. (b) Thermal insulation.

The first objective was to obtain uniform heating, so the temperature was checked at different points on the plate. For the temperature measurement, two instruments were used: a laser thermometer (Benetech brand) and a thermocouple of a thermostat (Droyd brand). The sensor element of the thermostat was kept in the center of the plate and the laser thermometer took measurements in the center and in peripheral parts of the system. The resistance was turned on in the thermostat, adjusting the temperature to 40 ºC. For better control of the resistance power (2.5 kW maximum power), it was connected to a potentiometer. A scale was adapted to the potentiometer and it was kept adjusted to an intermediate power of about 1.25 kW.

In order to better homogenize the temperature of the entire system and achieve a permanent regime, all sides were closed with a protected insulating material to reduce the risk of combustion (16 mm thick polystyrene wrapped in 0.1 mm thick aluminium sheets). The temperature tests were remade and a temperature difference of about 4 °C was obtained between the measured points of the perimeter and the center, which represents a satisfactory value given the uncertainty of measurement of the temperature measuring instruments used.

A one way valve to prevent contamination of the vacuum system with lamination residue has been constructed with a glass container with a lid. Two holes were drilled in this cover and two hoses were pulled. One of the holes was connected to the vacuum pump (Suryha brand) and the other was connected to the zinc plate system. Its function is to prevent the resin from passing into the pump, preventing its malfunction.

After the integration of the subsystems, shown in Figure 3 (structure, vacuum system, temperature control and thermal insulation), tests were performed in order to validate the bench, in which vacuum and constant temperature were obtained. The one way valve system efficiently prevented resin residues from entering the vacuum pump.

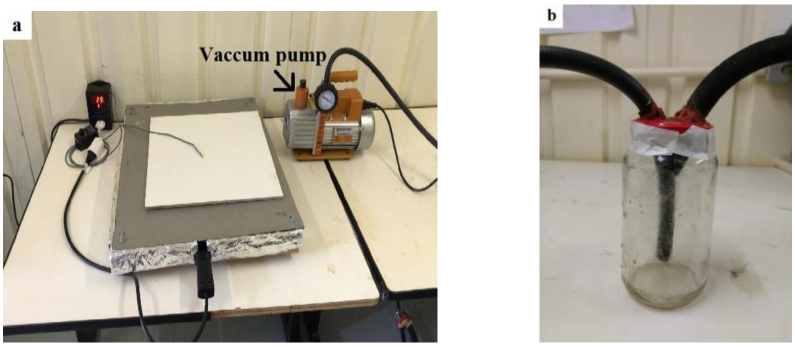

Figure 3: (a) Lamination bench. (b) One way valve.

2.4 Manufacture of specimens

The specimens were divided into four groups (type A, type B, type C and type D) according to the curing and post-cure processes to which they were submitted. Type A was made from a lamination in 6 hours of exposure to vacuum (vacuum pressure of 15 kPa), and curing process during 24 hours in ambient conditions (25 °C of temperature), without post-cure. Type B was performed in 3 hours of vacuum exposure (vacuum pressure of 15 kPa) with bench temperature around 40 °C and curing for 24 hours under ambient conditions (25 °C temperature), without post-cure. Type C was performed in a vacuum (vacuum pressure of 15 kPa) at an ambient temperature of 25 °C for 6 hours, and after autoclave curing at a temperature of around 80°C and a pressure of 69 kPa for 2 hours. Type D was performed in 3 hours of vacuum exposure (vacuum pressure of 15 kPa) with bench temperature around 40 °C and post-cure in the autoclave with temperature around 80 °C and pressure of 69 kPa for 2 hours. Table 1 elucidates the conditions of the curing process and post-cure submitted.

Table 1: Pressure and temperature during the curing and post-curing processes.

Specimen	Curing vacuum pressure [kPa]	Curing temperature [°C]	Curing time [hours]	Post-cure manometric pressure [kPa]	Post-cure temperature [°C]	Post-curing time [hours]
A	15	25	6	0	25	24
B	15	40	3	0	25	24
C	15	25	6	69	80	2
D	15	40	3	69	80	2

The production of the specimens took place through the production of rectangular laminate with four layers of fiberglass with dimensions 250mm x 25 mm x 4 mm (braided with fibers arranged in 0° and 90° orientation and grammage 0,12 g/cm^2) with orientation 0° and 90° of the blanket alternately, each face of the blanket was coated with resin, being utilized the Polyester resin 2001-BB and the hardener 3154 Polyester, both from the brand Anjo. According to the manufacturer, the resin mixture with the hardener must be made before the process. After the mixture is made, it is estimated a period of 30 minutes to be able to pass it in the fiber, being a proportion of 25 droplets of catalyst for a cure temperature higher than 25°C and 50 droplets when the cure temperature is lower than 25 °C for each 100g of polyester resin. The specimens were cut from the laminate in the dimensions 25 mm x 250 mm x 2.5 mm, as shown in Figure 4.

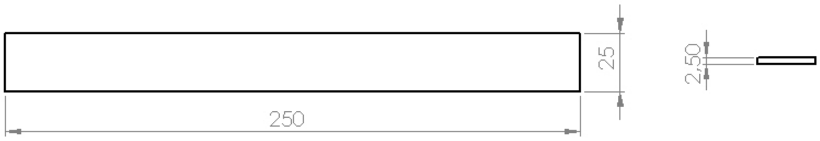

Figure 4: Specimen dimensions

The lamination of the material was done with the bench mentioned in the topic above, on this bench was positioned an MDF plate of 400mm x 400mm x 10mm, with which the laminate is made, because it facilitates the handling of the composite, besides the possibility of being used in high temperatures or at room temperature. It is necessary to apply two layers of release agents to facilitate the removal of the laminate.

To perform the vacuum exposure process, a procedure with four specific materials was used: peel ply, perforated film, absorbent film and vacuum bag, overlaid respectively in this order. The dimensions of each material were 350 mm x 350 mm x 0.15 mm, except for the vacuum bag, which was 400 mm x 400 mm x 0.15 mm to assist in handling when placed on the MDF plate. Tacky tap commercial adhesive was used to fix the vacuum bag, and the other materials do not need to be fixed by the adhesive. A rubber tubing is positioned on the one way valve and cannot come into contact with the laminate to remove the air without direct contact with the vacuum pump.

2.5 Tensile test

The tensile test was performed in the materials characterization laboratory, in the equipment INSTRON 8801, available in the materials laboratory of the University of Brasilia. The test consists of pulling the specimen that is attached vertically to the clamps of the machine according to Figure 5. The ascent speed of the upper clamp to pull the sample was 2 mm/min as suggested by the ASTM D3039 standard. From equation 1, the stress/deformation graph was constructed using the test results, and the modulus of elasticity was calculated using equation 2 with the force and strain values obtained, in which σ is the normal stress, F is the force measured in the test, A is the cross-sectional area of the specimen and ε is the specific strain.

$$\sigma = F/A \tag{1}$$

$$E = \sigma / \varepsilon \tag{2}$$

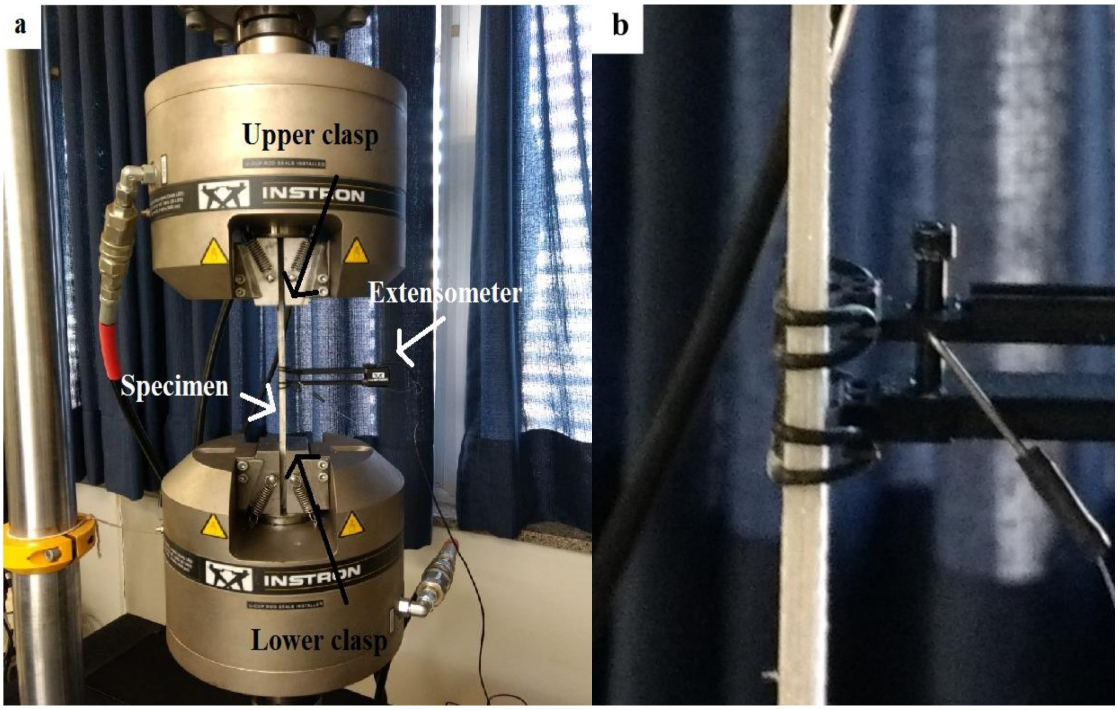

Figure 5: (a) Specimen attached to the clamps with a fixed strain gauge. (b) Strain gauge fixed on specimen.

The specific strain is measured by the strain gauge. The device is fixed by rubbers to the midpoint of the specimen. In order to carry out the measurement during the test it is necessary to remove the lock and calibrate the instrument in the software of the tensile testing machine. However, the marking at instant zero of the test is not null, so the value of the strain is zeroed to calculate the modulus of elasticity and construct the stress by strain graph.

3. RESULTS AND DISCUSSION

3.1 Qualitative Analysis

The method of cutting specimens is done manually. Because of this, the average specimens have the dimensions suggested by the standards, and the standard deviation associated with the dimensions is not significant in order to interfere with the results obtained in the tensile test. Table 2 shows the average dimensions of the specimens, in which 4 layers of fiberglass were used in all types of samples.

Table 2: Averages of the dimensions of specimens.

Specimen	Width [mm]		Length [mm]		Thickness [mm]	
	Average	Standard Deviation + instrumental error	Average	Standard Deviation + instrumental error	Average	Standard Deviation + instrumental error
A	24,9	0,051	250	0,55	2,5	0,051
B	24,5	0,052	249	0,552	4,1	0,055
C	24,9	0,051	249,3	0,552	3,8	0,051
D	25,3	0,05	248,9	0,551	3,8	0,051

During the production of the laminate it was observed that the ambient temperature influences the manufacturing process. On warmer days the resin starts its curing process in a shorter time interval which reduces the packing of the fiber layers. Due to this fact, the laminate has a higher final thickness when compared to the laminates produced in days with milder temperatures, therefore, the specimens were manufactured during the morning period. In addition to the ambient temperature, the quality of the vacuum bag seal and the wear of the vacuum system's one way valve sealing also interfere with the thickness of the final composite.

Type A samples were thinner due to the fact that they were made with low average room temperature (around 25 ºC). All samples were produced using four layers of fiber in the laminate, following the same pattern adopted in Spigosso's 2017 work. It was noted that specimens C and D were thinner than specimens B, showing that the pressurized post-cure process improves the compactation of the material, due to pressure in the oven.

3.2 Quantitative Analysis

The results of the impulse excitation test (non-destructive test) were disregarded because of inconsistencies in the results obtained. The divergence in the results may have occurred because the measurement system is not suitable for measuring the bending and twisting frequencies of laminated composite materials. Due to this, the analyzed results are the tensile test results and the analyzed parameter is the modulus of elasticity.

The tensile test was not performed until the fracture of the material occurred, because the available tensile test machine has a maximum load restriction of 10 kN for safety reasons. Following the ASTM D3039, the test was performed only in the elastic region. In this case, it is required to reach at least a value of 0.002 of strain to calculate the elastic modulus of the specimen (using equation 2). From equation 1 and the corrected strain were obtained the average strain stress curves, shown in Figure 6, of each type of specimen and their respective average modulus of elasticity according to table 3.

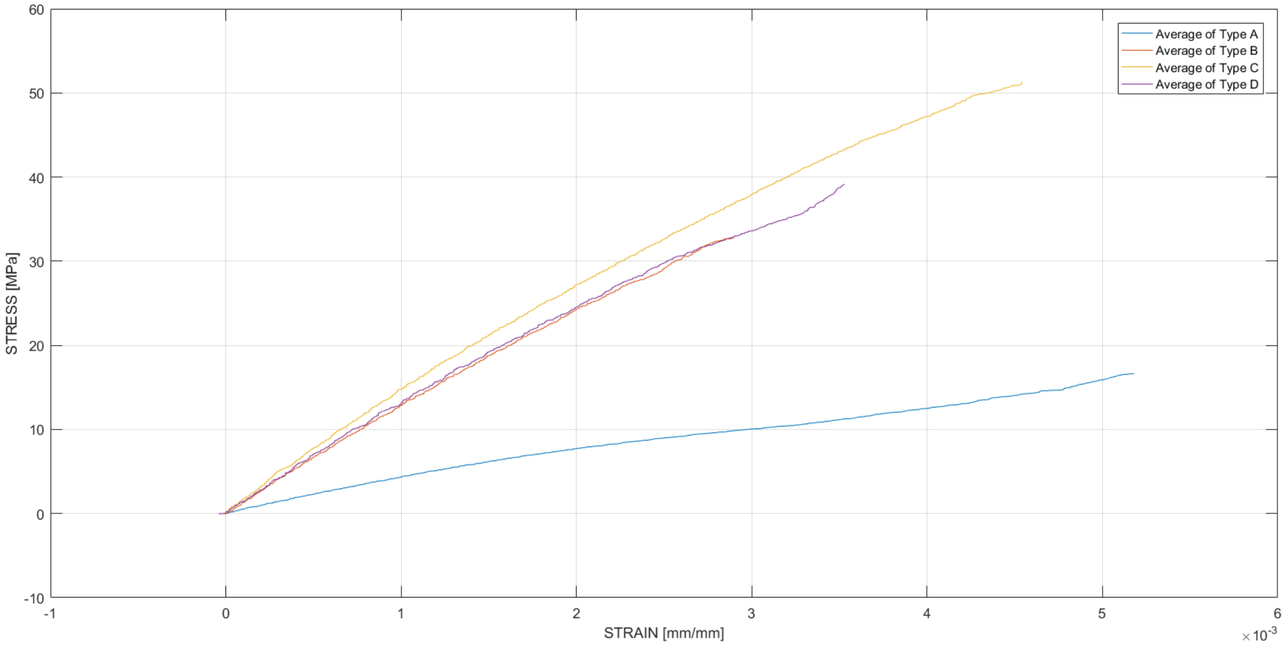

Figure 6: Average stress strain curves of each specimen.

Table 3: Modules of average elasticity of each type of specimen.

Specimen	Young Modulus (MPa)
A	3,85 ± 0,2
B	12,12 ± 1,62
C	13,51 ± 0,95
D	12,27 ± 0,98

The samples that went through the curing and/or post-cure process with exposure to a higher temperature than the room temperature (types B, C and D specimens) obtained greater stiffness because the temperature acted as a catalyst in the curing process of the polymeric resin. The temperature favored the polymerization reaction and the formation of cross-links of the polymer. This process ensured greater mechanical resistance to the composite.

The type D specimen obtained a slightly higher modulus of elasticity than the type B specimen. The type D test specimen underwent the curing and post-cure processes on the bench and in the autoclave with a temperature higher than the environment and pressure difference in relation to the atmosphere. The type B test specimen, on the other hand, only went through the curing process on the bench, just like the type D test specimen, but the post-cure was in ambient temperature and pressure conditions. The curing process on the lamination bench with high temperature and pressure difference catalyzes the polymerization reaction of the material. It can be inferred that the lamination on the bench with significant difference in temperature and pressure in relation to the ambient conditions influenced in order to increase the stiffness of the material. The post-cure process did not have much influence because the increase in stiffness of the type D specimen in relation to B was small. This is because most of the chemical polymerization reactions occurred during the curing process.

Post-cure is more prevalent in the comparison of type A and C specimens. The curing of the type A and C specimens was done under the ambient temperature conditions, but the type C specimens went through the post-cure process with high temperature and pressure, which resulted in a higher stiffness than the other specimens. When only the curing process is performed at room temperature without following up with post-cure with a higher temperature and pressure difference, there is no maximum formation of polymerization cross-links in the matrix. Thus, by submitting the laminate to the post-cure process with higher temperature and pressure, the curing of the material intensifies and happens more quickly. It can be concluded that post-cure increases more quickly the stiffness of the material in which chemical polymerization reactions are still occurring, a partially cured material.

The post-cure process in the autoclave has the advantage of homogenizing the temperature and pressure on the specimen due to the controlled atmosphere, which results in a laminate with more homogeneous properties. This can be evidenced by the relatively small standard deviations of the modulus of elasticity of type C and D specimens. On the other hand, bench curing does not provide homogeneity of mechanical properties to the specimen due to two factors: different temperatures at various points on the bench surface and temperature difference between the heated surface of the specimen and the environment. These temperature variations promote faster curing in certain locations and, consequently, do not favor the homogeneity of the laminate, which can be observed by the large value of the standard deviation of the modulus of elasticity of the type B specimen.

Type A specimens had the most homogeneous laminate due to the fact that both cure and post cure were performed at room temperature, that is, the fiberglass plate was subject to practically the same temperature difference, causing all polyester resin to be cured at almost the same rate. This can be observed because the modulus of elasticity of type A specimens have the lowest standard deviation. However, the ambient temperature does not provide an effective polymerization, which resulted in a low stiffness of the type A laminate when compared to the other specimens exposed to a higher temperature and pressure difference during curing and post-cure.

In addition, the type C laminate obtained the highest resistance to elastic deformation because post-cure provides greater compaction between the reinforcement-matrix interface, thus there is a greater transfer of mechanical stress absorption from the resin to the fiber. Post-cure with partially cured resin at room temperature provides better polymerization and enables greater crystallization of polyester.

The lamination methodology employed showed that high temperature curing combined with post-cure under ambient conditions increase the material stiffness by about 68.2%, while high temperature and pressure curing and post-cure increase the stiffness by about 68.6% and vacuum curing and the ambient temperature associated with high temperature and pressure post-cure increase the stiffness by about 71.5%. This suggests that the equipment developed is efficient for the cure and post cure of composite material. It is also noted that the cure associated with post-curing at high temperatures and pressure differences increase material stiffness to relatively significant values with less time spent. Therefore, this study shows the potential of different cure and post cure combinations in order to increase stiffness and reduce the time and cost of manufacturing composite material laminates.

4. CONCLUSION

The methodology developed to manufacture composites proved to be efficient. It was noted that the climatic and/or local conditions in which the composite is intended to be fabricated interfere in its quality and mechanical properties. The controlled environment with uniform temperature and pressure provides greater homogeneity to the composite, that is, the laminate has the cure of the resin almost at the same rate. Vacuum curing at room temperature associated with post-cure at high temperature and pressure results in a greater increase in stiffness, around 71.5% in 8 hours of procedure. High temperature and pressure cure and post-cure had an increase in stiffness of about 68.6%, but with a time of 5 hours of procedure. This study achieved the stipulated objectives because it promoted the development and testing of devices for the manufacture of composite materials with low construction and maintenance costs. This study also evaluated the application potential of different curing and post-cure combinations in order to increase mechanical properties and finish quality of laminated composite materials, reducing manufacturing time and cost.

5. ACKNOWLEDGMENTS

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