Revista Colombiana de Materiales

ISSN 2256-1013, No 14, pp.117-128, 2019.

EVALUATION OF MECHANICAL PROPERTIES IN CARBON/EPOXY COMPOSITES WITH DIFFERENT CURING PARAMETERS

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ABSTRACT

The intense research on the parameters involved in the composite materials grows every day due to the use of these materials in areas such as automotive, military and aeronautics. The use of composite materials is due to the possibility of combining desirable properties with the joining of metals, ceramics and/or polymers. The aim of this work was to evaluate the different conditions of improvement of the fabrication and curing process of the carbon fiber composite and epoxy resin, verifying if the differences are significant with the variations of resin volumetric fraction and curing process. For this purpose, the curing occurred due to the action of the vacuum, with the aid of the hydraulic press at a pressure of 1tnf with no pressure, all for 5 hours. For each type of curing process, the volumetric fraction of the resin was varied in 15%, 25%, 35% and 45%. Then, mechanical tensile, flexural and DMA tests were carried out and the results showed that there is an influence of the curing processes on the performance of the composites. The increase of the volumetric fraction of resin with hydraulic press and with no pressure showed a tendency of increase in the properties of tensile and flexural, respectively. In addition, for the vacuum process, it showed a better thermal stability. With this, depending on the application, one can choose the most suitable curing process.

Keywords: Composites, carbon fiber, epoxy, properties.

1. INTRODUCTION

Nowadays, the importance of composite materials has been increasing due to the requirements of more efficient product with better mechanical properties [1,2]. With the advancement of technology, the interest in the composites properties has been growing with time, and today their market is well spread including transport, civil construction, marine, electric, wind industry, consumer products and aeronautical market [3–5].

In general, a composite can be considered as any multiphase material that exhibits a significant proportion of the properties of both constituent phases, such that the best combination of these properties is obtained [6,7]. According to this principle of combined action, better combinations

of properties are created by a combination of two or more distinct materials, in which the reinforcement and the matrix are obtained [8,9].

The material used in the reinforcement is responsible for attenuating the mechanical and chemical properties, in general [10,11]. Among the main reinforcements of composites, carbon fiber has important applications. Depending on how it is processed, it can present a wide range of physical, chemical, electrical and thermal properties [12,13]. Among the several functions of the matrix, it is responsible for the preservation of the reinforcement in its determined place protecting the material from possible unexpected conditions [14].

There are several parameters that influence the properties of the composites such as fiber orientation, fiber volumetric fraction, number of layers, stacking sequences, thickness of the layers, treatments used, technique and manufacturing process, and materials used [15,16]. The use of the combined epoxy resin with carbon fibers is very common when a good relation of mechanical properties is required and it is desired to apply as structural materials [17–19]. The possible manufacturing methods are so important as the combinations among the materials because they are related to the quality control of the production process, that is, the final quality of the composite [20,21].

However, within the manufacturing process, the properties of the final laminates may differ as a function of several curing parameters [22]. In addition, it is defined as laminate the composite that is manufactured alternating the stacking of the different materials [23]. These properties can be obtained by mechanical tests in order to determine the ideal set of parameters of this process in which the higher values of relative properties are obtained, guaranteeing a higher quality of the laminated composite [24–26].

Thereby, the aim of this work was to evaluate the tensile, flexural and mechanical dynamics properties of a carbon fiber laminate with epoxy resin in different curing parameters (hydraulic press, vacuum and no pressure) by varying the volume percentages of resin (15%, 25%, 35% and 45%).

2. EXPERIMENTAL PROCEDURE

2.1. Materials

The main raw materials used in this research were carbon fiber and epoxy resin. The fiber is bidirectional fabric type 200 g/m². The resin is of the epoxy type ES260, suitable for manual laminations, and its catalyst is type E35. The manufacturer of these raw materials is Advanced Vacuum. The density of the fiber was obtained according to the manufacturer (ρ_f = 1.8 g/cm³) and the density of the resin was obtained experimentally through the mass and the volume of a test piece only of the resin manufactured for this purpose (ρ_r = 1.2 g/cm³). In addition, peel ply and acrylon were used as resin absorptive fabrics and liquid mold release agent.

2.2. Preparation of composites

The composites of carbon fiber and epoxy resin in this work were obtained taking into consideration three curing processes, varying the resin volumetric fractions of 15%, 25%, 35% and 45%. This variation in the resin volumetric fraction in steps of 10% (starting from 15% by decision of the authors aiming a smaller number of total samples) was to guarantee more fiber than resin in the composite and also to evaluate the effect of curing versus the resin content. The

composite was formed of 4 layers of fibers, in same orientation [(0º, 90º)] interspersed with 4 layers of resin. A rectangular mold was used to support the laminate for the cases of curing with no pressure and with vacuum action. A plastic bag to wrap it was also used. For the case where the cure occurred due to the action of the hydraulic press, metallic plates were used, usually used in compression tests. In all three cases, prior to starting the process, the mold release agent was used 3 times intercalated for a drying period of about 30 minutes in order to facilitate the release of the laminate from mold or the dishes.

Initially, manual rolling was performed and the cure occurred under pressure of 1 tnf determined in the hydraulic press. In the next process, the cure was given under the action of vacuum with the aid of the vacuum pump. And finally, the cure naturally occurred at ambient pressure.

Two factors are common to the three processes: manual lamination was the starting point and in all composites four reinforcement layers were used. In addition, the curing process was carried out for all laminates in a 5 hours period, suggested by the epoxy resin manufacturer, which indicates its cure between 4 and 6 hours. The symbology of each sample is shown in detail in Table 1 according to the variation of the resin volumetric fraction and the curing process.

Table 1. *Symbology of the samples according to their variations.*

Samples	Volumetric fraction of resin (%)	Curing process
A1	15	Hydraulic press
A2	25	
A3	35	
A4	45	
A5	15	Vacuum
A6	25	
A7	35	
A8	45	
A9	15	No pressure
A10	25	
A11	35	
A12	45	

2.3. Characterization of the tests

The samples submitted to the tests of this research had their dimensions determined according to ASTM D3039 standards for tensile strength tests in composite materials and ASTM D7264 for flexural tests in composite materials. A Universal Mechanical Testing Machine (EMIC CCE 100KN) with velocity of 2 mm/min and 1 mm/min was used for the tensile and flexure tests, respectively. For tensile tests the standard recommends the use of tabs of about 20 mm in order to reduce the difficulty of attaching the specimen to the test machine claws.

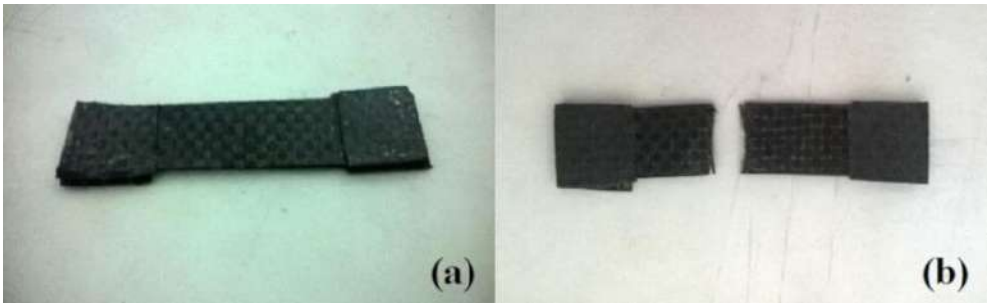

Figure 1. *Specimen for tensile test (a) normal and (b) fractured.*

Figure 2. *Flexural test.*

Samples for the DMA analyzes were based on the characteristics of the equipment itself, since there is no standard for this, using a DMA Q 800 (TA Instruments). Some data were used for this analysis, such as amplitude (15 μm), frequency (2 Hz) and heating rate (5 °C/min). The data obtained through this test were analyzed by the elastic module, which are directly related to the resistance to deformation and the glass transition average temperature (Tg), the initial glass transition temperature (Tg_i) and the final glass transition temperature (Tg_f). The damping modules can also be obtained, but these will not be considered this analysis.

Table 2 shows the dimensions of the test specimens for each test, considering that the thicknesses varied according to the manufacturing process, but in all cases, four layers of fiber were intercalated with four layers of resin. Thus, 12 different laminates were obtained and from each of them, five specimens were obtained to be submitted to tensile tests, five for flexural tests and one for DMA analysis, 132 specimens in total. Equipments such as vacuum pump, hydraulic press and mini grinding were used for the other processes.

Table 2. *Dimensions of specimens for each type of test.*

Type of test	Length (mm)	Width (mm)
Tensile	70	15
Flexural	120	13
DMA	40	5

3. RESULTS AND DISCUSSIONS

3.1. Tensile testing

It can be observed that the modulus of elasticity, rupture tension, and ultimate tensile strength, have their values in ascending order as the resin volumetric fraction increases in the curing process with hydraulic press under a pressure of 1tnf. The highest values were found for the sample A4 (45% of resin) with 48.98 GPa, 801 MPa, 842.44 MPa, for the modulus of elasticity, rupture tension, and ultimate tensile strength. These values represent an average out of 5 measurements for each sample. The standard deviation (SD) of the Ultimate Tensile Strength is also showed in Table 3. In the process in which the curing occurred under vacuum, with the aid of a vacuum pump, there was a decrease in the tensile properties as the volume percentage of resin increased, with exception for the sample A5.

The best values for the sample A6 (25% of resin) for the modulus of elasticity, rupture tension, and ultimate tensile strength were 49.21 GPa, 799.16 MPa, 864.9 MPa, respectively. In relation to the previous process (hydraulic press), it was possible to observe an advantage in the values of the properties, since the samples cured in the vacuum presented values still slightly higher than the best values of the samples cured in the hydraulic press, except for the rupture tension. In this case, it was observed that increasing the resin by 35% or 45% does not bring advantages, that is, the values of 25% are enough for the composite to have high mechanical properties and superior to the previous curing process.

In the process in which the curing occurred naturally at ambient pressure, the highest values of mechanical properties for the samples A10 (25% of resin) relating to the modulus of elasticity with 48.12 GPa, and A12 (45% of resin) for rupture tension and ultimate tensile strength were 681.81 MPa and 791.37 MPa, respectively. In a comparison of the three curing processes, it can be seen that with the cure in the press and at ambient pressure, the best results were presented with 45% of resin and with curing in the vacuum, the best results were shown for the samples with 25% of resin. Of all the tests, the sample A6 (25% of resin) in the vacuum curing process had the best performance relating to these properties.

Table 3. *Mechanical properties for the samples in tensile tests.*

Samples	Curing process	Modulus of elasticity (GPa)	Rupture tension (MPa)	Ultimate tensile strength (MPa)	Standard deviation (MPa)	Strain (m)
A1	Hydraulic press	36.64	648.82	688.97	0.82	0.02167
A2		45.79	674.48	808.70	0.60	0.02112
A3		47.66	698.59	840.69	0.77	0.01945
A4		48.98	801.00	842.44	0.79	0.02635
A5	Vacuum	35.76	472.97	560.16	0.71	0.02145
A6		49.21	799.16	864.90	0.77	0.02061
A7		48.36	778.72	855.36	0.61	0.01889
A8		33.62	576.93	640.38	0.78	0.02579
A9	No pressure	31.70	325.83	447.67	0.79	0.02191
A10		48.12	653.12	773.95	0.24	0.01968
A11		41.99	575.74	679.15	0.39	0.01975
A12		47.58	681.81	791.37	1.99	0.01817

3.2. Flexural tests

In the flexural tests, with curing in hydraulic press, the best results of maximum flexural tension and rupture tension were 891 MPa and 753.92 MPa, respectively, for the sample A3 (35% resin). In relation to the modulus of elasticity, the sample A2 (25% of resin) presented the best value, that is, this type of sample had a higher resistance to deformation, and a greater elastic stiffness. However, the maximum flexural tension value presented by sample A3 is much higher than the values of the other samples. The standard deviation (SD) of the Maximum flexural tension is showed in Table 4.

When observing the values obtained under the action of vacuum, a behavior similar to that occurred in the previous process. The highest values of modulus of elasticity, rupture tension and maximum flexural tension were found for the sample A7 (35% of resin) with 116.45 GPa, 695.75 MPa, 927.79 MPa, respectively. The values obtained for this sample containing this volumetric fraction exceed the values for the other samples. In this case, it also occurred that the deformation of the specimen for the submitted load was the smallest.

Comparing the best result of each process, as in the tensile tests, the vacuum is more efficient than the cure in the press. Flexural tests for the samples that cured at ambient pressure exhibited a different behavior than the previous procedures. In this case, as the volumetric fraction of resin increased, the values of the properties cited also increased. The deformation behavior is inversely proportional. Therefore, sample A12 (45% of resin) presented the highest values with 98.17 GPa, 795.04 MPa, 1178.85 MPa, for modulus of elasticity, rupture tension, and maximum flexural tension, respectively. Comparing with the best results among the processes, the sample A7 (35% of resin) presented the highest modulus of elasticity with vacuum cure, and the sample A12 (45% of resin with no pressure) presented the highest values for rupture tension and maximum flexural

tension for the sample. The choice of the sample will depend on the application of the composite, since it does not have a better performance in the properties contained in a single process.

Table 4. *Mechanical properties for the samples in flexural tests.*

Samples	Curing process	Modulus of elasticity (GPa)	Rupture tension (MPa)	Maximum flexural tension (MPa)	Standard deviation (Mpa)	Strain (m)
A1	Hydraulic press	42.92	95.95	178.74	0.41	0.01206
A2		92.88	150.77	493.97	0.73	0.01494
A3		77.79	753.92	891.00	0.25	0.01434
A4		79.88	150.78	383.81	0.85	0.01046
A5	Vacuum	49.44	178.19	328.99	0.36	0.01234
A6		77.11	278.33	695.85	0.26	0.01121
A7		116.45	695.75	927.79	0.63	0.00963
A8		44.98	301.57	630.54	0.55	0.01777
A9	No pressure	42.01	178.19	246.73	0.48	0.01679
A10		61.27	150.77	370.11	0.71	0.01514
A11		75.14	246.73	452.35	0.30	0.01488
A12		98.17	795.04	1178.85	0.59	0.01389

3.3. DMA analysis

In the DMA analysis, relevant data were extracted as elastic modulus (E), initial glass transition temperature (Tg_i), average glass transition temperature (Tg), final glass transition temperature (Tg_f), and modulus of damping (tan δ). The curves of samples A2 (25% of resin), A3 (35% of resin) and A4 (45% of resin) showed the same type of behavior.

The curve that refers to sample A1 showed an atypical behavior and should not be analyzed. The justification for this behavior is the total no cure of the specimen. Regarding the modulus of elasticity, sample A2 (25% of resin) presented better results both in relation to its own value (15.78 GPa) and in relation to the higher thermal stability of the sample. The sample A4 (45% of resin) had lower elastic modulus value and sample A3 showed the lowest thermal stability of the properties. Within this analysis with this process, sample A2 showed a better performance.

There was no atypical behavior of any of the vacuum cured samples. Sample A8 (45% of resin) presented a higher elastic modulus value with 42.09 GPa, however, it had the worst thermal stability. Sample A5 (15% of resin) presented the highest thermal stability. The sample A6 (25% of resin) was considered interesting by the look of the thermal analysis, since it had the second largest elastic modulus and also presented a good stability of its thermal properties, very close to sample A5.

Comparing the curing processes in the press and in the vacuum, an influence of the elastic modulus process was observed. In vacuum, these values are quite above the values obtained in the press. There is a closeness in the values of the module of elasticity of samples A9, A10 and A11.

However, sample A9 also exhibited the highest thermal stability. The range of values presented for the module of elasticity for this ambient pressure cure process lies between the values of the curing process in the hydraulic press and the values of the curing process in vacuum.

Thus, it has been observed that curing in the vacuum is more indicated when a greater elastic modulus and greater thermal stability of the samples analyzed are required. It is also important to emphasize that one property does not replace the other one. The interesting thing is that there is a compatibility. It can be observed that curing in vacuum is the process that presents larger module of elasticity for all the resin volumetric fractions. In addition, the lower the amount of resin the greater the value of the glass transition temperature. The cure in the press is the case that presented the lowest values also for all resin fractions.

Table 5. *Modulus of elasticity and glass transition temperatures of samples.*

Samples	Curing process	Modulus of elasticity (GPa)	Glass transition temperature Tg (ºC)		
			Initial	Average	Final
A1	Hydraulic press	14.13	60.22	81.66	101.98
A2		15.78	72.24	79.64	90.26
A3		14.53	64.34	75.25	88.86
A4		12.12	70.24	75.25	86.77
A5	Vacuum	31.22	80.65	87.87	86.88
A6		35.52	75.25	83.07	92.17
A7		28.95	73.44	80.65	89.07
A8		42.09	62.43	70.45	80.06
A9	No pressure	18.29	70.24	80.65	94.08
A10		19.76	68.84	77.65	88.26
A11		19.36	63.03	74.84	86.86
A12		14.18	71.65	78.06	78.06

The most relevant of the damping modulus curves is their beginning and in this case, the values are so close that no sample is highlighted. By the peak of the curves it would also be possible to determine the average glass transition, initial glass transition and final glass transition temperatures. However, it is more reliable to obtain them by the method of tangents in the curves of the modulus of elasticity, as was done in this analysis. Figure 3, 4, 5 showed the curves of the damping modulus for the samples cured in the hydraulic press, under vacuum and no pressure, respectively. Both presented similar behavior to the first one, exhibiting very close values at the beginning and with the glass transition starting in the 60 and 70ºC range.

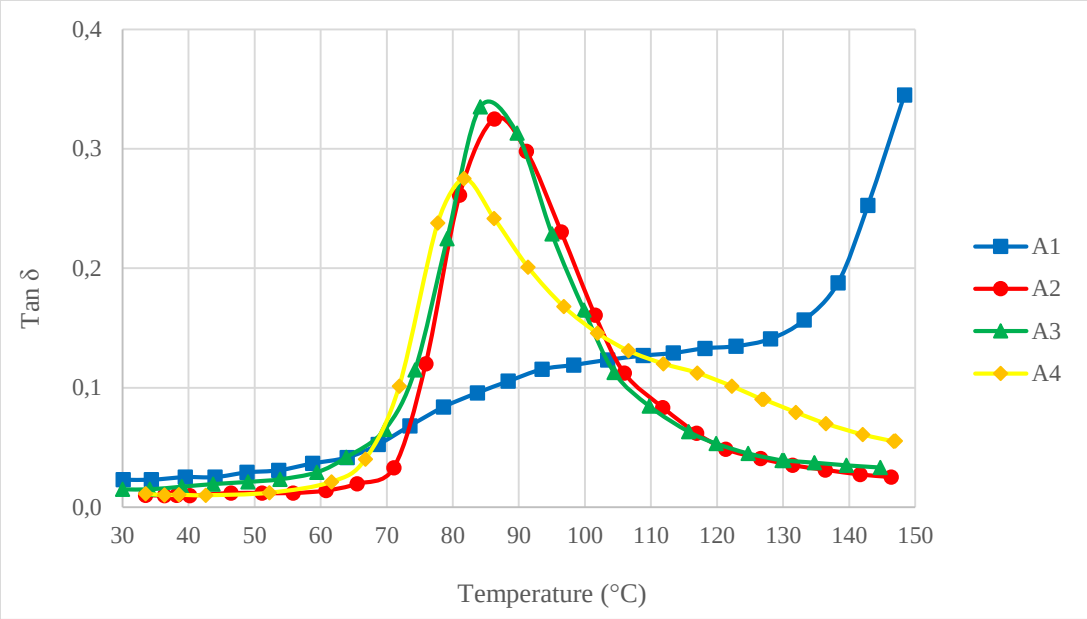

Figure 3. *Damping module as a function of temperature for samples cured in hydraulic press.*

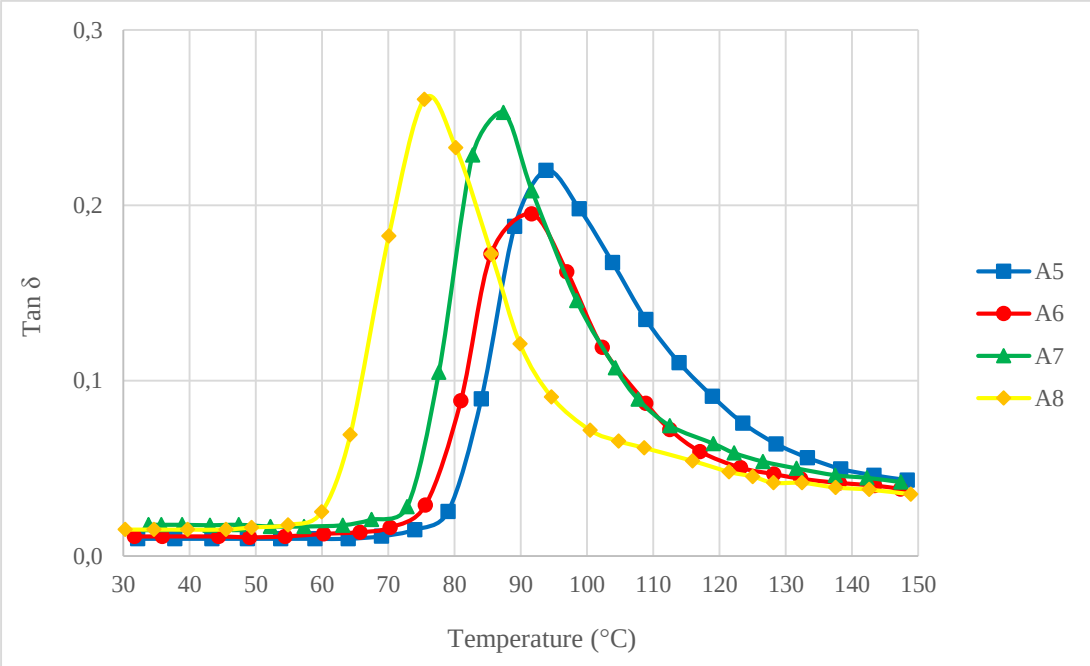

Figure 4. *Damping module as a function of temperature for samples cured in vacuum.*

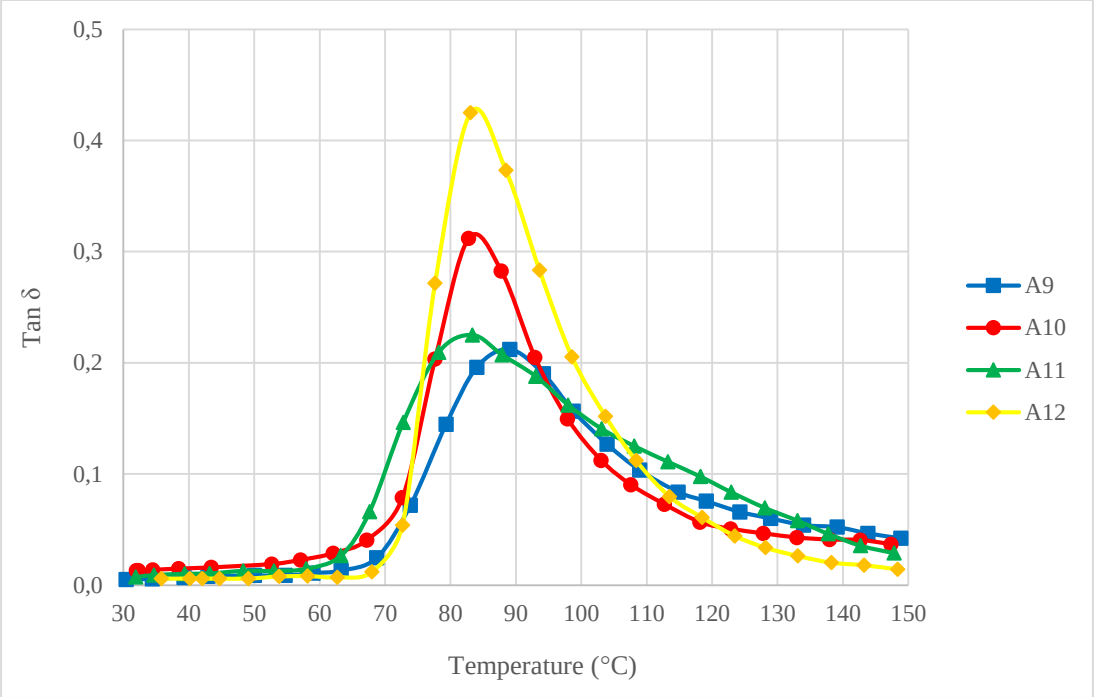

Figure 5. *Damping module as a function of temperature for samples cured in no pressure.*

4. CONCLUSIONS

It can be affirmed that there is a reliability of the results presented in this research, since this number is recommended by the standards used and cover losses due to failures in the manufacturing process and/or in the test. Both failures occurred throughout the tests, however, the values considered valid were sufficient for comparison and choice of the most suitable for presentation. Within each group of samples the values were, for the most part, consistent.

The results showed a tendency that the properties increased as the resin volumetric fraction increases in the hydraulic press cure process (in the tensile), no pressure (in the flexural), in general. In addition, it was found that the percentage of 45% (tensile) and 35% (flexural) of resin for hydraulic press, 25% (tensile) and 35% (flexural) of resin for vacuum, and 45% of resin (tensile and flexural) for no pressure, presented better performances in the properties. Increasing resin content can fill better the voids that occupy the composite in the hydraulic press curing process, resulting in better tensile properties. In the DMA analysis, it showed a tendency to have higher values of modulus of elasticity in the vacuum curing process, having as the lowest performance in the hydraulic press, not having a very significant variation in the growth of the resin content.

In addition, when looking for better thermal stability, the vacuum curing samples showed a trend of better performance, with the glass transition starting at 60 to 70 ºC for the curing processes, which were shown in the module curves of damping. Based on the tendencies found, it is possible to select a curing process and a volumetric fraction of resin for the reinforcement and the matrix in question, according to the main need of the project.

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