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Effects of the Vacuum Moulding Process on the Mechanical Properties of Cotton/Epoxy Composite

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Abstract
A study on the effect of the vacuum assisted resin transfer moulding (VARTM) manufacturing process on the mechanical properties of cotton/epoxy composite is presented in this investigation. Woven cotton was used as reinforcing material embedded in epoxy resin. The woven cotton was treated with sodium hydroxide for one hour at concentrations of 0% to 20%. The tensile test showed that the untreated material had the highest ultimate strength and Young's modulus. Observation of the fracture surface by scan electronic microscopy (SEM) was compared to the hand lay-up process as well as the results of the tensile test. The comparison showed that the VARTM process presents better mechanical properties than the hand lay-up process due to the reduction in discontinuities observed by means of optical microscopy.

Key words: *natural fibre, composite, cotton, tensile, fractography.*

Introduction

Composites are a mixture of materials with different properties, one of which has the capacity to support loads, providing strength and stiffness, known as the reinforcement, embedded in a brittle material, which maintains the position and orientation of the reinforcement, known as the Matrix [1]. Polymer matrix composites (CMP) are the most commonly known and used because of their ease of processing and manufacturing. CMPs consist of a polymer matrix (thermoset or thermoplastic) reinforced with fibres, tissues or particles (synthetic or natural) [2].

Unfortunately the use of CMP reinforced with synthetic fibres becomes a problem once they have fulfilled their useful life. For this reason, it was chosen to study composite materials from renewable resources [3-5]. The use and study by industry and academia of reinforced CMPs with natural fibres have grown in recent years because they offer advantages over synthetic fibres, such as low density, high mechanical strength, low cost and environmental contributions [6]. Frequently the most natural fibres used as reinforcement are jute, hemp, sisal, cotton, banana, bamboo, flax, coconut, etc. [6, 7].

The manufacturing methods most used for the composite between natural fibres and thermosetting resins are hand lay-up, pressure moulding, protrusion, resin transfusion moulding (RTM) and vacuum assisted resin transfusion moulding (VARTM). The hand lay-up manufacturing process is most commonly used at the industrial level due to its speed and ease, but is more susceptible to errors and defects in the material [8-10]. The VARTM process provides clean, economical manufacturing of the composite which reduces contact with materials and the emission of volatiles. In addition, the process decreases manufacturing defects such as voids, impurities and bubbles, providing a homogeneous material and increasing its mechanical properties [11].

Although cotton is the most popular natural fibre in the world due to its use in the textile industry, during the literary review, insufficient papers were found showing the use of cotton as a reinforcement in composite materials. The objective of this research was to demonstrate the use of cotton as a reinforcement of a versatile composite material, as well as to analyse the effect on the mechanical properties of the VARTM and hand lay-up processes. A study of the mechanical properties of the cotton/epoxy composite was performed by means of tensile tests, and analysis of the effect of the VARTM process was performed using scanning electron microscopy (SEM).

Material and methods

Materials

The composite material was made up of an epoxy system composed of the COLREPOX 6090 epoxy resin and 1956 B hardener. ***Table 1*** shows the main mechanical properties of the epoxy system, which was reinforced by unidirectional woven cotton known as "genus", produced by the intermingling of mercerized yarns, thus forming a weft and warp, which correspond to the longitudinal and transverse fibres, respectively. The use of this fabric was totally textile. ***Table 2*** shows the main mechanical properties of the cotton yarns. ***Figure 1*** shows an SEM photograph of the woven cotton, where the warp (greater number of threads and fabric direction) and weft can be identified.

Methods

Fibres mercerization

The fibres were cut parallel to the warp of the fabric (orientation of the fibres), obtaining a 0 ° orientation of fibres. The size of the cuts for the tensile test specimens were 20 x 30 cm, guaranteeing at least 10 specimens per moulding.

Table 1. *Mechanical properties of epoxy resin COLREPOX 6090 [12].*

Properties	Value	Unit
Density	1100	Kg/m^3
Young's modulus	3000	MPa
Shear modulus	4200	MPa
Poisson rate	0,38	–
Tensile strength	35	MPa
Compressive strength	100	MPa
Shear strength	13,78	MPa

Table 2. *Mechanical properties of cotton yarns [13].*

Properties	Value	Unit
Density	1.54	g/m^3
Tensile strength	400	MPa
Young's modulus	4.8	GPa

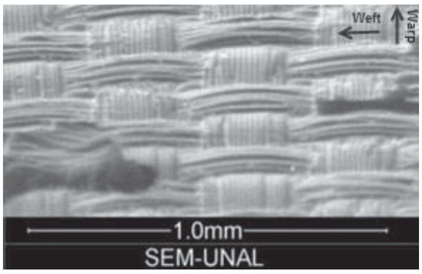

Figure 1. *SEM photography of woven cotton.*

The mercerisation process was carried out with 99% of NaOH purity caustic soda. The fibres were immersed in a liquid solution of caustic soda at concentrations of 5, 10, 15 and 20% for 1 hour; these were chosen due to preliminary studies performed on cotton and other natural fibres [14, 15]. Afterwards they were washed with water containing 1% acetic acid to neutralise the excess of sodium hydroxide, and final washing was performed several times with water to ensure the removal of all chemical residues [16]. Finally the drying of the fibres was carried out in an oven at 110 °C for 1 hour [17]. The average moisture content of the cotton fabrics after oven drying was 7% ± 0.06. After the chemical treatment, the fibres were ironed in order to eliminate existing wrinkles and protected from the environment by means of airtight bags.

Composite manufacturing

The composite material was constructed using VARTM. The process consists in providing a sealed space by means of a flexible membrane on a rigid mould [18] a comprehensive Vacuum Assisted Resin Transfer Molding (VARTM). First a rubber tape is placed on a glass moald, outlining a space of 35 x 23 cm, then spiral tubes are placed in front of the resin inlet and outlet. The architecture used in the location of the fabrics is shown in ***Figure 2***, consisting of a bleeding fabric (absorbent) in the middle of two releasing agent fabrics located on the surface of the mould, followed by four layers of aligned woven cotton, and finally a bleeding fabric in the middle of the two releasing agent fabrics. Once the fabrics are placed, the distribution mesh is placed and finally the mould is sealed a vacuum bag with sealing tape. To check the seal, a vacuum is made at 67729 Pa ± 1000 for 10 minutes, close to atmospheric pressure. The pressure was measured with an analogue manometer, after which time no pressure change should be observed.

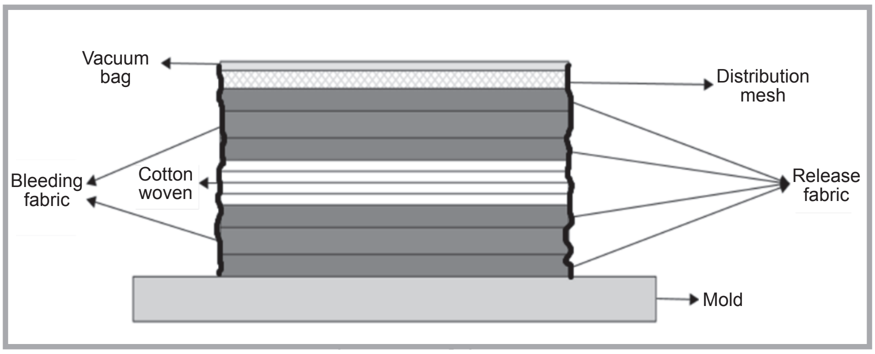

Figure 2. *Architecture of the VARTM process.*

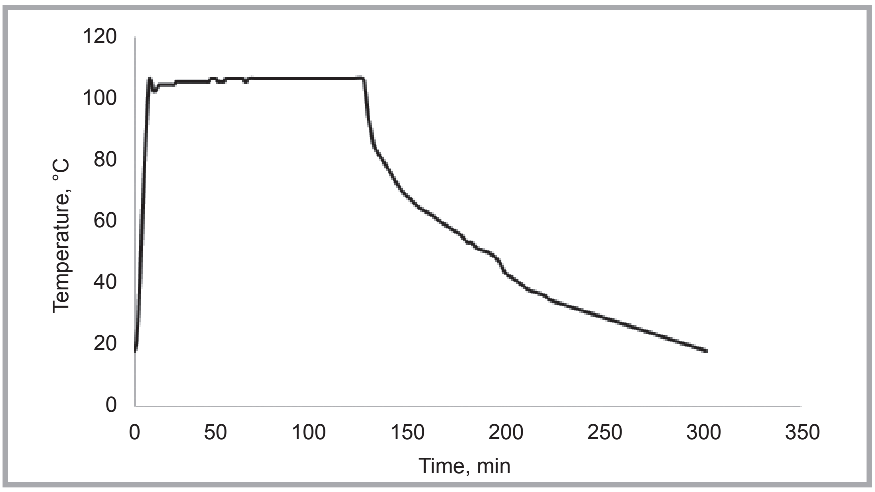

Figure 3. *Real straight ramp-up.*

After checking the vacuum in the mould, the thermoset resin was added, which was mixed with its hardener at a 5: 1 ratio, where the percentage of resin used was 250% of the total weight of the fabrics. The resin was added at a vacuum pressure of 67.7 kPa ± 1 for an approximate time of 20 minutes. After completion of the resin infusion process, the mould (with the composite material thereon) was put into an oven, where the material was cured at a temperature of 105 °C for 120 minutes. ***Figure 3*** shows the temperature ramp used for curing the material.

When the composite material was completely cured, samples were cut, ensuring the elimination of stress concentrators that may affect material testing, where each cut was made with a guillotine for plastic, and all the edges were inspected visually and smoothed with sad paper, obtaining 10 specimens by moulding, the general dimensions of which were as follows: long (15 mm ± 0.3), width (150 mm ± 2), thickness (1 mm ± 0.06), in agreement with ASTM D3039 [19]. Finally 20 specimens were made by chemical treatment.

Measurements

Tensile measurements of the composite material were performed using optical microscopy and SEM of the fracture surface of the test specimens. Other studies were not performed because equipment for the manufacture of samples was not available.

The tensile test was performed using a Shimadzu Autograph AG-IS 5 KN machine (Japan), in which the calibrated length for all the tests was 150 mm, and the width and thickness of the specimens were measured for each of them. The tensile test was performed using Standard ASTM D3039 [19]. A study of the fracture surface was done using SEM FEI QUANTA 200 equipment (USA), and to make the shots the specimens were coated with gold by means of sputtering and located at a 70° angle in front of the microscope [20].

Results and discussion

Tensile test

Figure 4 shows the stress – strain graph for each of the chemical treatments, where it can be observed that the composite with the greatest modulus of elasticity and ultimate strength is that without chemical treatment: ultimate strength – 79.3 MPa, Young's modulus – 2.8 GPa, and deformation percentage – 9.0%. ***Table 3*** shows data obtained by ANOVA analysis for each of the chemical treatments. The reduction in mechanical properties produced by the chemical treatment is caused by the degradation of the fibre due to the high contents of sodium hydroxide, as presented by Koyuncu et al., The degradation is due to the reduction in cellulose and increase in Hemicellulose and lignin [21].

Table 4 shows a comparison of the tensile strength, modulus of elasticity and manufacturing process for CMP reinforced with natural fibres and the cotton/epoxy composite studied. It can be observed how the manufacturing process VARTM significantly improves the mechanical properties of the composite in relation to the hand lay-up process.

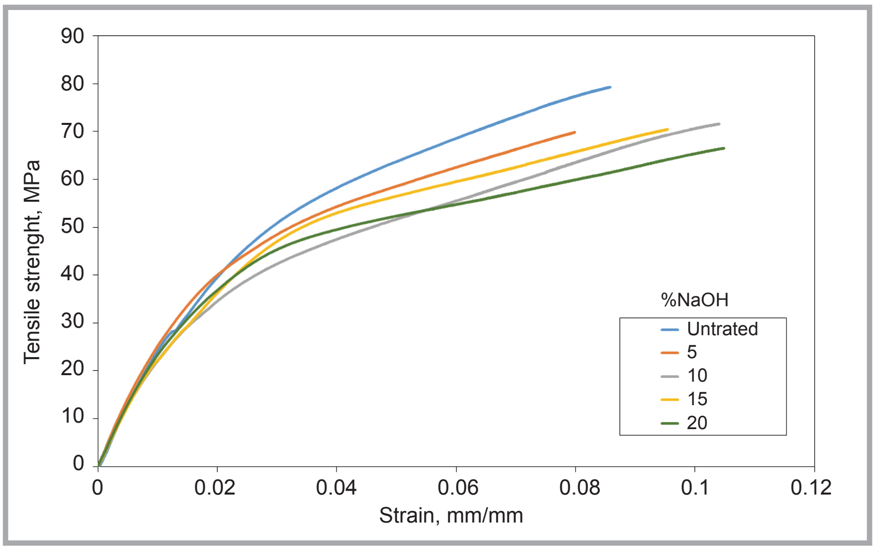

Figure 4. *Stress-strain curve for each of the composites with the different chemical treatments.*

Comparison between the hand lay-up and VARTM processes

Figure 5 shows the fracture surface for the tensile test specimens made by hand lay-up and VARTM. The hand lay-up composite was fabricated with the same fibre/resin proportion and same curing process as the VARTM composite. It can be observed how the VARTM manufacturing protocol provides better wetting between the fibre bundles, which is reflected in a homogeneous surface with less presence of pores and impurities. The average percentage of pores in the composites manufactured by hand lay-up is 5.09%, while that for the composites manufactured by VARTM is 1.5%. In addition, a surface without a clear pull out is observed.

Figure 6 shows a comparison of the ultimate strength and Young's modulus of the cotton/epoxy composite made by the VARTM and hand lay-up processes, where the composite fabricated with the hand lay-up process presented an ultimate strength of 38.3 MPa and Young's modulus of 2.2 GPa; thus it is seen how the VARTM process increases the properties of the composite. This is because the VARTM process guarantees a homogeneous and simultaneous pressure on all the material, which corresponds to the atmospheric pressure. In addition, a homogeneous and simultaneous pressure reduces the appearance of pores and the low wetting between bunches of fibres. Additionally, by generating a vacuum in the mould, moisture is reduced or eliminated. These considerations contribute to improving the mechanical properties of the composite material.

These results are in accordance with research conducted by Yuhazri et al., based on a comparison between the manufacturing processes for the kenaf/polyster composite and Yun-Hae et al. in their study of the jute/styrene composite, where the tensile strength and Young's modulus are greater for the VARTM manufacturing process compared to the hand lay-up, which is associated with the decrease in defects, such as voids and homogeneous compaction on the material [27, 28].

Conslusions

Chemical treatment with NaOH did not provide an increase in the mechanical properties of the cotton/epoxy composite, due to degradation of the fibre caused by the reduction in cellulose; only an increase in the percentage of deformation of the material is observed. The composite without chemical treat-

Table 3. *Tensile test properties of each cotton/epoxy composite, mean value (standard deviation).*

Fibre with chemical treatment	Youn's modulus, GPa	Ultimate strength, MPa	Deformation percentage, %
0% NaOH	2.769 (0.184)	79.347 (1.557)	9.037 (0.518)
5% NaOH	2.655 (0.170)	70.089 (2.526)	8.766 (0.522)
10% NaOH	2.460 (0.130)	71.620 (2.240)	9.975 (0.648)
15% NaOH	2.535 (0.137)	70.508 (2.309)	9.241 (0.632)
20% NaOH	2.532 (0.090)	66.209 (2.786)	10.187 (0.899)

Table 4. *Comparing the mechanical properties of some CMPs reinforced with natural fibres.*

Composite	Tensile strength, MPa	Young's modulus, GPa	Manufacture process	Reference
Flax (woven) – Epoxy	104	10	VARTM	[22]
Jute – Epoxy	112	14	VARI	[23]
Cotton – Epoxy	45	2	Hand lay – up	[21]
Cotton – Epoxy	45	2	Hand lay – up	[10]
Cotton – Epoxy	38.3	2.2	Hand lay – up	[24]
Kenaf – Epoxy	35	6	Hand lay – up	[25]
Sisal – Epoxy	183	15	RTM	[26]
Cotton – Epoxy	80	2.6	VARTM	Current investigation

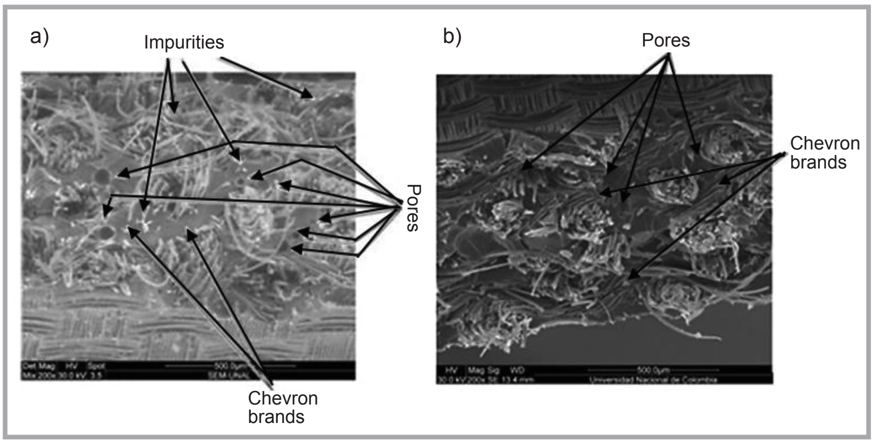

Figure 5. SEM image of cotton/epoxy composite: a) hand lay-up process [24], b) VARTM process.

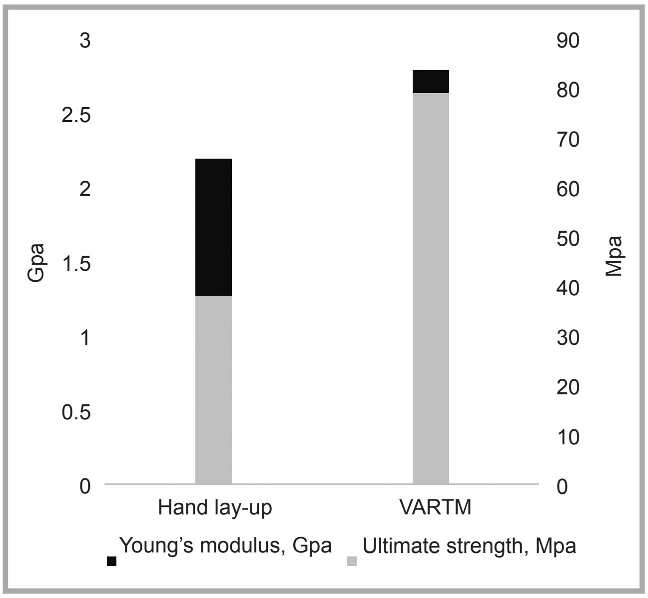

***Figure 6.** Comparison ultimate strength and Young's modulus of the cotton/epoxy composite for the hand lay-up and VARTM manufacturing processes.*

ment showed an ultimate strength of 79.3 MPa, Young's modulus of 2.8 GPa and deformation percentage of 9.0%, being the highest results for a cotton/epoxy composite.

When comparing the VARTM and hand lay-up manufacturing methods for the cotton/epoxy composite, a clear increase in the ultimate strength and modulus of elasticity was observed due to the better wettability offered by the VARTM process, which can be explained by SEM observation, where a reduction in defects such as voids, impurities and bubbles is evidenced. In addition, in the VARTM process there is no evidence of a pullout as a mechanism of fracture, consequently fibre stretching, known as fibre brindging, is observed.

In conclusion, with the results obtained, it was demonstrated that it is feasible to use cotton as a reinforcement of composite materials, as in this study it was possible to obtain a versatile material with optimal mechanical properties by applying the VARTM manufacturing process.

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