Characterization of Composites Based on Natural and Glass Fibers Obtained by Vacuum Infusion

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ABSTRACT: The mechanical properties of composites based on different natural fibers and glass fibers using unsaturated polyester and modified acrylic as matrix are evaluated. In spite of the several works done in natural fiber composites, there are very few results on acrylic as matrix. Fabrication of the composites is done by means of vacuum infusion. Flexural, tension, and impact test are conducted on the composites. Ignition, thermal degradation, and water absorption are determined. Jute composite with unsaturated polyester resin as matrix showed the best results on flexural and tensile strengths and the lowest in impact energy, because of the strong interphase developed. Flax composites show higher impact energy than the other natural fiber composites, due to the existence of the effective energy dissipation mechanisms, like pull-out and axial splitting of the fibers. Scanning electron micrograph confirmed this fact. None of the samples resisted the five-second exposition to the flame on the ignition test. All of them were completely consumed, and flax composites burned the longest.

KEY WORDS: RTM, vacuum infusion, natural fibers, glass fibers, mechanical properties, ignition test, water absorption.

INTRODUCTION

NATURAL FIBERS-REINFORCED POLYMERS have gained importance in technical applications such as the automotive industry, where mechanical properties have to be combined with low weight [1–8]. Natural fibers have a number of advantages as a reinforcing factor, including low weight, low cost, availability from renewable resources,

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low density, high specific properties, nonabrasive processing characteristics, and lack of residues upon incineration. The disadvantages are moisture absorption leading to fiber swelling, low thermal resistance, and local or seasonal variations in quality.

Manufacturing methods of natural fibers thermoset composites have been layup, press molding, pultrusion, resin transfer molding (RTM), and vacuum infusion molding. The RTM process consists of the introduction of a liquid resin or thermoset into a closed mold, which contains dry fiber mats under an applied pressure gradient. The pressure difference required for resin flow may be created by applying a vacuum to the mold, an external source at elevated pressure, such as gravity feed, or more often a positive displacement pump or pressure vessel.

Thermosetting resins are used in this processing technique. Epoxy resin with sisal fibers was studied by Oksman et al. [8]. They determined the morphology and mechanical properties of sisal fibers composites manufactured by RTM with a fiber content of 30–50% volume fraction. It was shown that the specific modulus of sisal–epoxy composite was 17 GPa g cm^{-2} compared to 18 GPa g cm^{-2} for glass–epoxy composites and 29 GPa g cm^{-2} for enzyme-retted flax. The specific tensile strength of sisal–epoxy composites was 186 MPa compared to enzyme retted flax 210 MPa and 470 MPa of the glass fibers composites while the absolute value was 219 MPa versus 280 MPa and 817 MPa.

Munikenche Gowda et al. [9] evaluated the mechanical properties: flexural, tensile and impact tests of woven jute fabric-reinforced composites. The technique used was hand layup.

Bidirectional mat provides a laminate with better in-plane properties than a composite with unidirectional mat. In addition to better damage resistance under biaxial loading, the interlacing of the yarns in each layer of woven mat also allows better delaminating resistance, and hence, a superior impact performance compared to the composites with unidirectional mat [10].

Matemilola and Stronge [11] have studied the impact behavior of carbon fiber composites made by the RTM process. Various types of fractures, as well as the level of damage in each specimen, were assessed from photomicrographs of sections removed from the region near the impact point. The development of damage in the specimen is related to factors such as nose radius and density of missile, size of specimen, and kinetic energy of impact. Damage initiation depends on the ratio of missile nose-radius to plate-thickness. Karbhari [12] described the different regions of impact response of composites fabricated by RTM. Region I was defined as purely elastic in nature with a one-to-one ratio between the incident and returned energy. Region II is the actual dynamic response characterized by a linear relationship between the incident and the returned energy and region III is the region where small perturbations can significantly influence the returned energy levels. It was found that the first damage mode is those of intrabundle. Once these cracks propagate to the bundle surface, they can act as initiation sites for interbundle cracking. The intrabundle cracking is a consequence of the fibers debonding, and it is a result of the produced deformation from the transverse tensile strains and bundle compaction under load. Both intra and interbundle cracking can absorb significant levels of energy, without showing the gross damage levels seen in delamination that results from a specimen fabricated by prepreg.

Bledzki and Gassan [13] in a review of different composites reinforced with cellulose-based fibers show that the increase in modulus in flexure produces a decrease in impact energy for the unsaturated polyester with glass fibers and flax obtained by SMC. The measured properties were divided by the density (specific properties). Glass-fibers-reinforced

material attains higher characteristic values, except for tensile strength and impact energy showing a slightly higher value of modulus in flexure.

In spite of the large number of published results in natural fiber composites, there are very few results on acrylic as matrix. This type of resin is usually used for pultrusion and RTM process. The aim of the present paper is to characterize the composites based on two types of matrices: unsaturated polyester and acrylic, reinforced with natural and glass fiber and to compare their mechanical properties.

EXPERIMENTAL

Composite specimens consist of unsaturated polyester and unsaturated polyester modified with acrylic resins and natural fibers.

Castanhal Textil CIA, Brazil, kindly supplied woven jute. Finflax, Finland supplied flax mat. Vetrotex, Argentina supplied non-woven glass mat. Figure 1 shows the mat used in each case. Table 1 shows the cost and production of each kind of fiber [14–16] and Table 2 shows the fiber composition [17]. Single filament tensile tests were performed according to the ASTM D 3379-75 standard, using a dynamometer Instron 4467. Single filaments were mounted along the center-line of a slotted tap. Tests were carried out using a 20-mm gauge length and a crosshead speed of 5 mm/min. The cross-sectional area of the jute fiber was considered circular and it was measured by using an optical microscope. The average

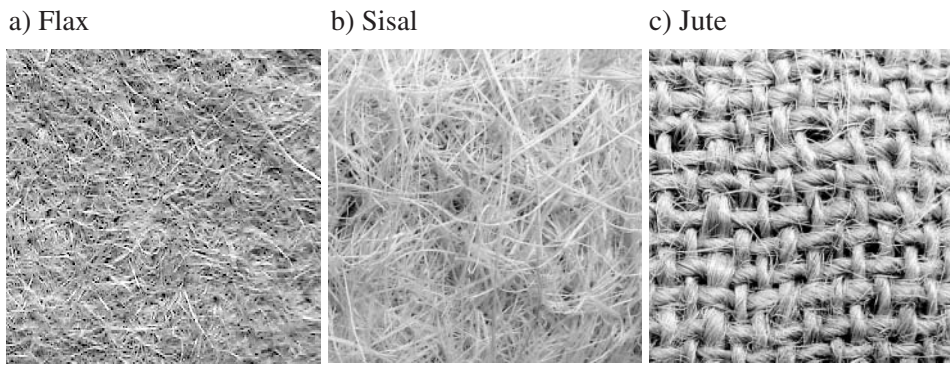


Figure 1. *Photographs of the different natural mats used in the present paper.*

Table 1. Comparison between the type of fiber and production.

Type of fiber	Comparison of the fiber cost in function of glass fiber cost [%]	Production (1000 ton)
Jute	18	3600
E-Glass	100	1200
Flax	130	800
Sisal	21	500
Banana	40	100
Coir	17	100

Table 2. Composition of different natural fibers Ref (Gassan J., Bledzki AK., Die Angew Makromol Chem., (1996)).

Component (%)	Jute	Flax	Sisal
Cellulose	64.4	64.1	65.8
Hemi-cellulose	12.0	16.7	12.0
Pectin	0.2	1.8	0.8
Lignin	11.8	2.0	9.9
Water soluble substances	1.1	3.9	1.2
Wax	0.5	1.5	0.3
Water	10.0	10.0	10.0

Table 3. Properties of the used fibers and their mats.

Fibers	Young's modulus (GPa)	Tensile strength (MPa)	Specific density (δ/δ_{water})	Specific modulus (E/δ_{sp}) (GPa)	Mat surface density (kg/cm^2)	Mat thickness (mm)
Jute	30	500	1.3	23	25.5	0.45
Sisal	15	510	1.3	11.5	86.0	3
Flax	50	344	1.5	33.3	53.0	1.21
Glass	72	3400	2.5	28.8	44.5	0.70

diameter was used to calculate the properties of each fiber. Tensile test of fibers as well as the thickness of each mat used are shown in Table 3.

Ashland Chemicals supplied unsaturated polyester resin, Aeropol FS 6912, and resin-modified acrylic resin, Modar 835 S. Unsaturated polyester was mixed with 2% methyl ethyl ketone peroxide (MEKP, Fluka) and 0.4% DMA. The modified polyester resin was mixed with 2 wt% of benzoate peroxide (BPO). The fibers were used after drying for 24-h at 80°C. The density of the composites was determined by means of pycnometry (p) and floating and sinking (f) methods.

The mold used in injection processing is shown in Figure 2. Injection was done by means of the vacuum infusion technique. The resin enters through two injection points that are located in the corners and the resin flows to the center of the mold. The volume of the fibers was 30%. Molded plaques were cured at 60°C for 2 h, and post-cured at 110°C for 3 h.

Mechanical tests were performed using a dynamometer Instron 4467 at a crosshead speed of 2 mm/min for three-point bending (ASTM790). An average value of at least five specimens was determined. Tensile test were also done by ASTM D3039-00.

Impact tests (ASTM D256-84) were performed by means of a falling weight Fractovis Ceast. The speed of the test was set at 1 m/s, and the striker minimum mass (3.6 kg) was used.

Water absorption of different composites was measured. Specimens of 3–4 × 25 × 76 mm^3 were cut from laminates along the direction of the fiber axis and dried in a vacuum oven at 80°C until constant weight was obtained. Specimens were immersed in distilled water during 24 h at room temperature (ASTM D570-81). Specimens were wiped with filter paper before weighing and the water absorption was calculated as:

$$\%\text{water absorption} = \frac{(w(t) - w_0)}{w_0} \times 100$$

Optical micrographs of the composites were taken using a Olympus SZH-10 optical microscope equipped with a Sony camera adaptor CMA-D2. It was used in order to determine the degree of pull-out of each composite by means of fractured surface. The used magnification was 5.3×. The void content and size were determined by optical microscopy. In order to make a contrast on the image, calcium powder was introduced into the voids. The analysis of the image was done by means of Image-Plus from Media cybernetics software.

Scanning electron micrographs were also obtained in order to see the fractured surface of composites.

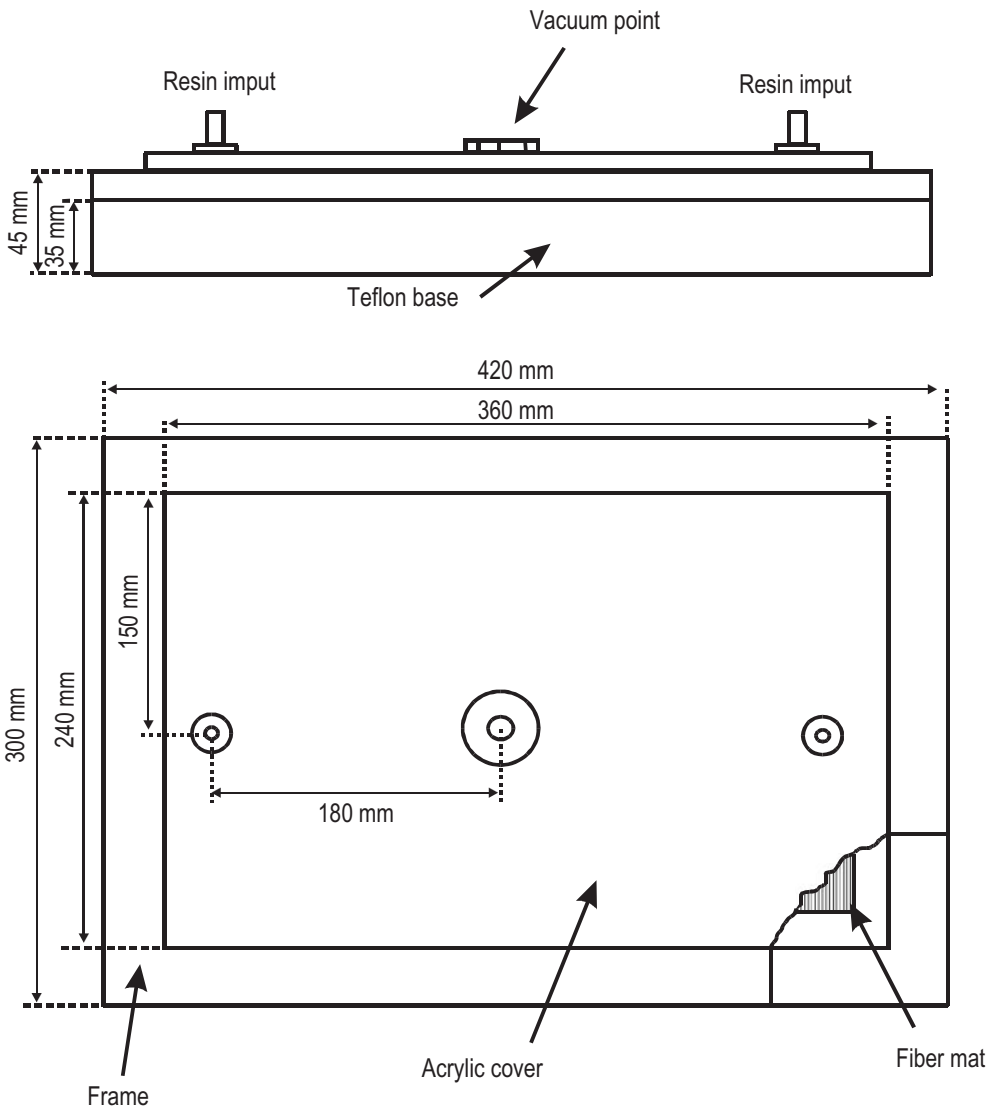


Figure 2. *Scheme of the used mold for vacuum infusion technique.*

Thermogravimetric analysis (TGA) was carried out using a TGA Shimadzu 50/50 at a heating rate of 10°C/min under nitrogen.

Ignition tests were performed according to ASTM D3713-78, ''Measuring Response of Solid Plastic to Ignition by a Small Flame.'' The chamber was built in the laboratory. Test results were expressed in terms of how long the composite resists contact with a small flame without ignition (ignition response index, IRI). Maximum resistance time considered in this test was 60 s. Three specimens of each sample were evaluated. Sample dimension were $3–4 \times 13 \times 63\,mm^3$.

RESULTS AND DISCUSSION

Figure 3 shows the results of flexural test for all the composites. The specimens show a nonlinear behavior because the crack initiates on the tension side of the beam and slowly propagates in an upward direction. Different mechanisms are involved in the fracture of the composite material. The composite displayed debonding between the fiber and the matrix due to the lack of adhesion, and as a consequence pull-out can occur. Pull-out is another mechanism of fracture energy, because it produces the sliding of the fiber into the matrix and friction between them. Natural fibers usually break in two different ways: transversal to the fiber length (as in glass fiber) and longitudinal to the fiber length or axial splitting of elementary fibers because each fiber is in fact a bundle of elementary fibers bonded together with pectin and lignin [18].

The flexural modulus and strength values obtained from several fiber-reinforced compounds are shown in Figure 4. Glass composites show the highest modulus value and strength. The highest modulus value for natural fiber composites was obtained with

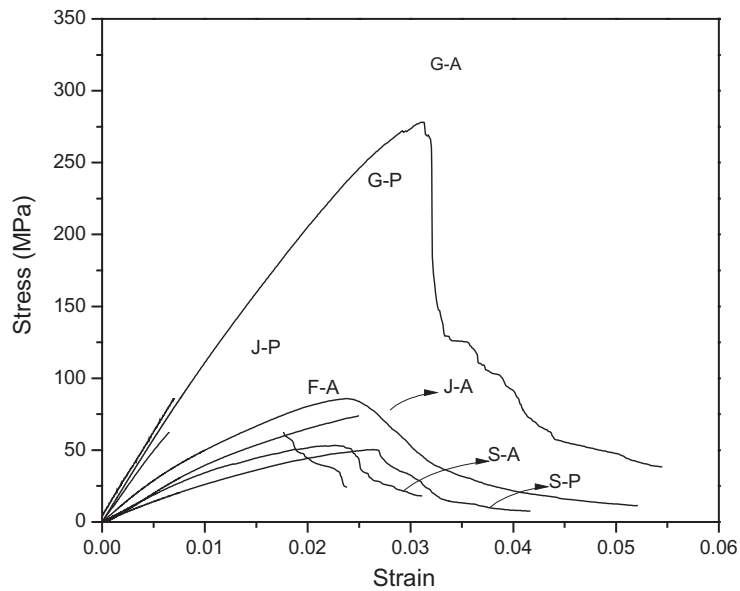


Figure 3. *Flexural test results for all of the composites: G-A (glass + acrylic); G-P (glass + polyester); J-P (jute + polyester); J-A (jute + acrylic); F-A (flax + acrylic); S-P (sisal + polyester); and S-A (sisal + acrylic).*

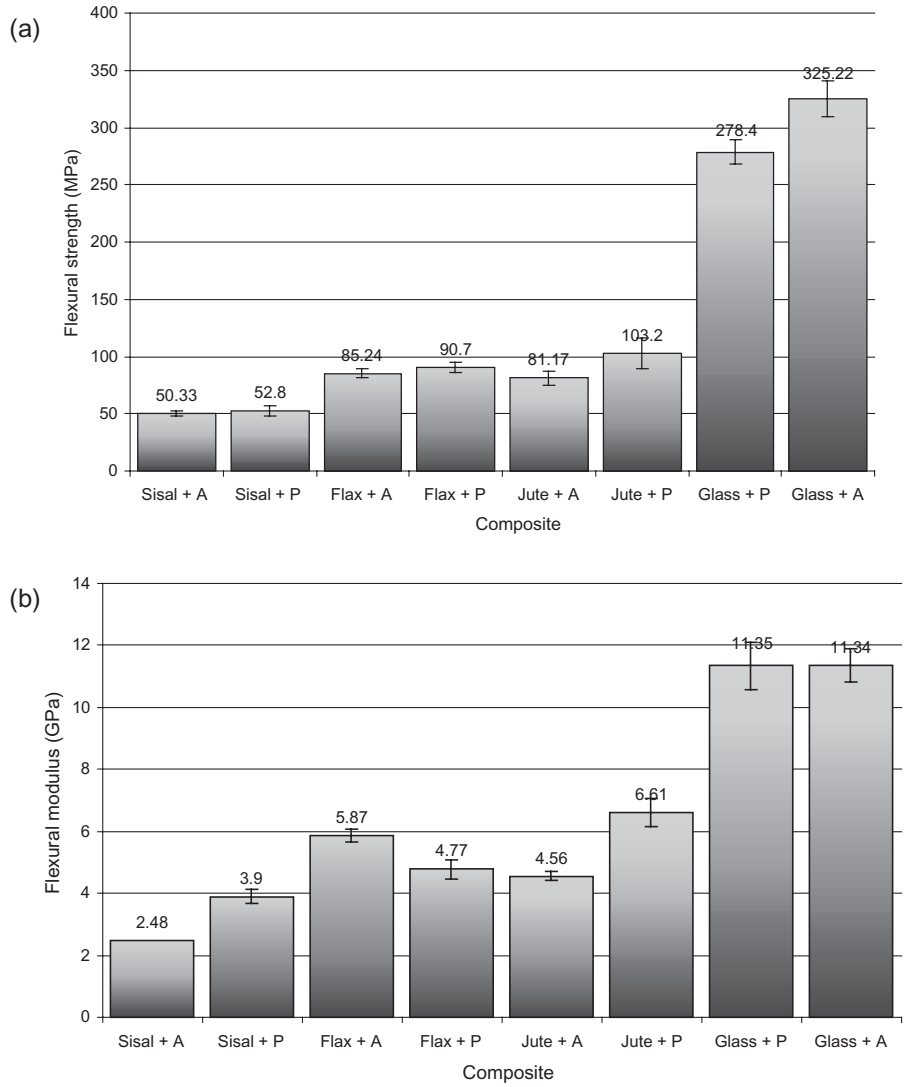


Figure 4. *Flexural properties for different composites: (a) flexural strength and (b) flexural modulus.*

polyester-jute. The composites based on sisal fibers show the lowest value. This can be explained by an incomplete wetting of the sisal fibers due to their low compatibility, or/and their high fiber diameter compared to other natural fibers. The composite with flax shows similar behavior than composite with jute. However, jute fibers are woven, and as a consequence it can produce better flexural behavior. Table 4 shows the specific modulus for each composite. Using natural fibers instead of glass fibers has a somewhat negative effect on the flexural performance. However, the difference is relatively small, and if the material cost is taken into account, jute and sisal natural fibers become competitive with glass fibers [19]. Single flax fibers showed better mechanical properties, but the composites based on flax fiber showed similar mechanical properties than the composites based on

Table 4. Composites flexural properties and their density obtained by pycnometry (p) or floating and sinking (f).

Composite	Flexural modulus (GPa)	Flexural strength (MPa)	Specific density (δ_{sp})	Modulus/δ_{sp} (GPa)	Strength/δ_{sp} (MPa)
Jute + Acrylic	4.6 ± 0.5	81 ± 6	1.254 f	3.64	64.7
Jute + Polyester	6.6 ± 0.8	103 ± 13	1.268 f	5.21	81.2
Sisal + Acrylic	2.5 ± 0.2	50 ± 2	1.197 f	2.07	42.0
Sisal + Polyester	3.9 ± 0.2	53 ± 5	1.192 f	3.27	44.3
Flax + Acrylic	5.9 ± 0.3	85 ± 4	1.215 f	4.83	70.0
Flax + Polyester	4.8 ± 0.1	91 ± 4	1.213 f	3.93	74.8
Glass + Acrylic	11.3 ± 0.5	325 ± 16	1.710 p	6.63	162.8
Glass + Polyester	11.4 ± 1.2	278 ± 11	1.637 p	6.94	198.6

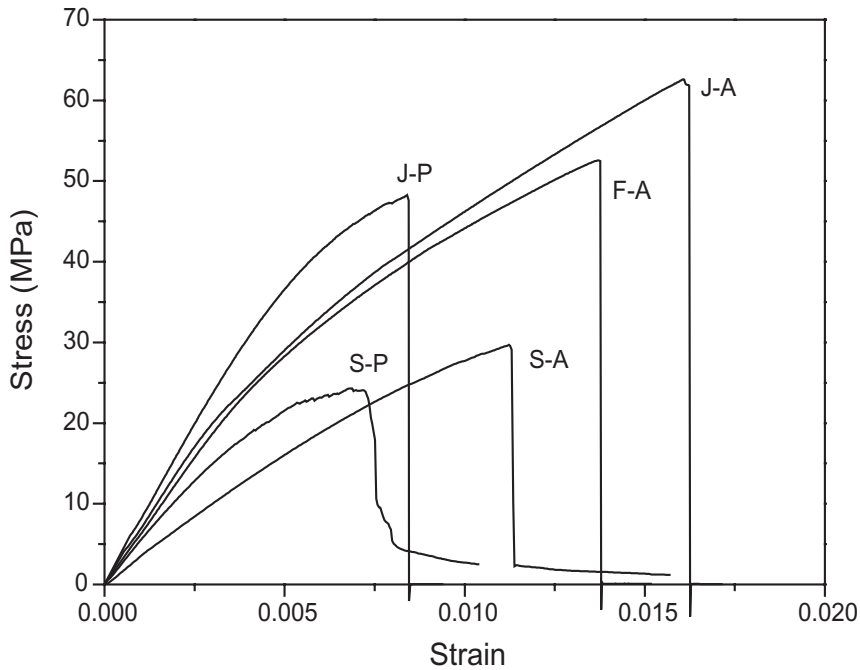


Figure 5. *Stress–strain curves in tensile test for different natural fibers composites tested: G-A (glass + acrylic); G-P (glass + polyester); J-P (jute + polyester); J-A (jute + acrylic); F-A (flax + acrylic); S-P (sisal + polyester); and S-A (sisal + acrylic).*

jute fibers, and this is a consequence of the different used mats (non-woven for flax fibers and woven for jute fibers).

Figure 5 shows the tensile experiment for each specimen, and the results are shown in Table 5. The tendency in the tensile experiments is similar to flexural results: jute $\approx$ flax > sisal. The comparison between polyester and acrylic resins result in a higher modulus for the case of composite based on polyester. SEM of fractured surfaces of glass fiber reinforced polyester and acrylic resins showed that for the latter, cleaner fibers were observed indicating a lower matrix–resin adhesion in this composite (Figure 6). In Figure 5, a higher area was observed under the curve for the composite based on acrylic resin in

Table 5. Results of tensile tests for each composite.

Composite	Flexural modulus (GPa)	Flexural strength (MPa)	Modulus/δ_{sp} (GPa)	Strength/δ_{sp} (MPa)
Jute + Acrylic	6.9 ± 0.1	62 ± 5	5.50	49.0
Jute + Polyester	8.0 ± 0.2	50 ± 5	6.30	39.0
Sisal + Acrylic	3.4 ± 0.2	31 ± 4	2.84	26.0
Sisal + Polyester	5.3 ± 0.2	24 ± 2	4.45	20.0
Flax + Acrylic	6.3 ± 0.2	52 ± 2	5.18	42.7
Flax + Polyester	6.3 ± 0.1	61 ± 1	5.19	50.3
Glass + Acrylic	13.3 ± 0.6	201 ± 19	7.78	117.5
Glass + Polyester	14.9 ± 0.5	190 ± 14	9.10	116.0

a) Glass-Acrylic b) Glass-Unsaturated Polyester

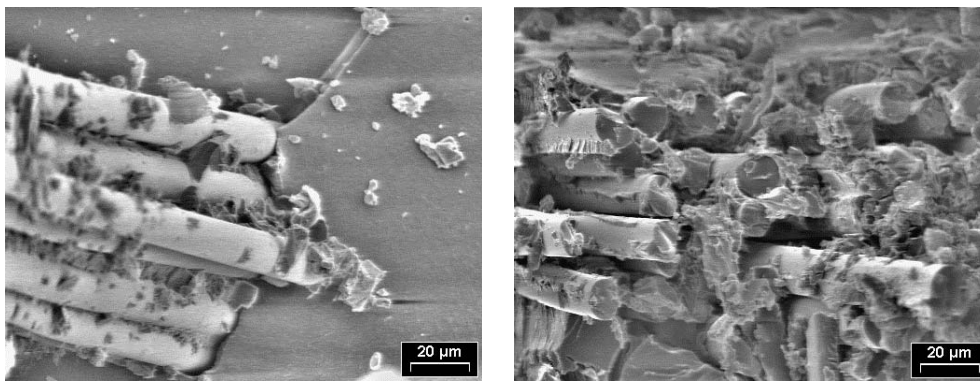


Figure 6. *Scanning electron micrograph of the surface of the composite based on glass fibers and: (a) acrylic resin and (b) polyester resin.*

comparison with the others due to their higher elongation before breaking (higher toughness material).

Scanning electron micrographs of the fractured surfaces obtained after impact test are shown in Figure 7. The micrographs show higher pull-out in the case of sisal composites than jute composites. The sisal fibers have a wider diameter than other natural fibers, and as a consequence these fibers have low interface than the others for the same volume content. Elementary flax fibers showed higher axial splitting and this mechanism has produced higher toughening of the fracture flax composites.

Figure 8 shows the results of impact tests for sisal and jute composites. Sisal fibers showed higher energy of propagation than jute fibers composites, and it indicates good compatibility of jute fibers with the thermoset resins and less pull-out of fibers from the matrix. Optical microscopy of the fractured specimens shows the different profiles for each composite (Figures 9 and 10).

The impact result for each composite is shown in Table 6. The composite based on glass fibers shows values ten times higher than the natural fiber composites. The highest energy value was obtained with flax fibers and acrylic resin, in agreement with the profile obtained in Figures 9 and 10. As can be seen in Figure 9(a), axial splitting is an important mechanism of fracture for the flax composites because the fibers are more

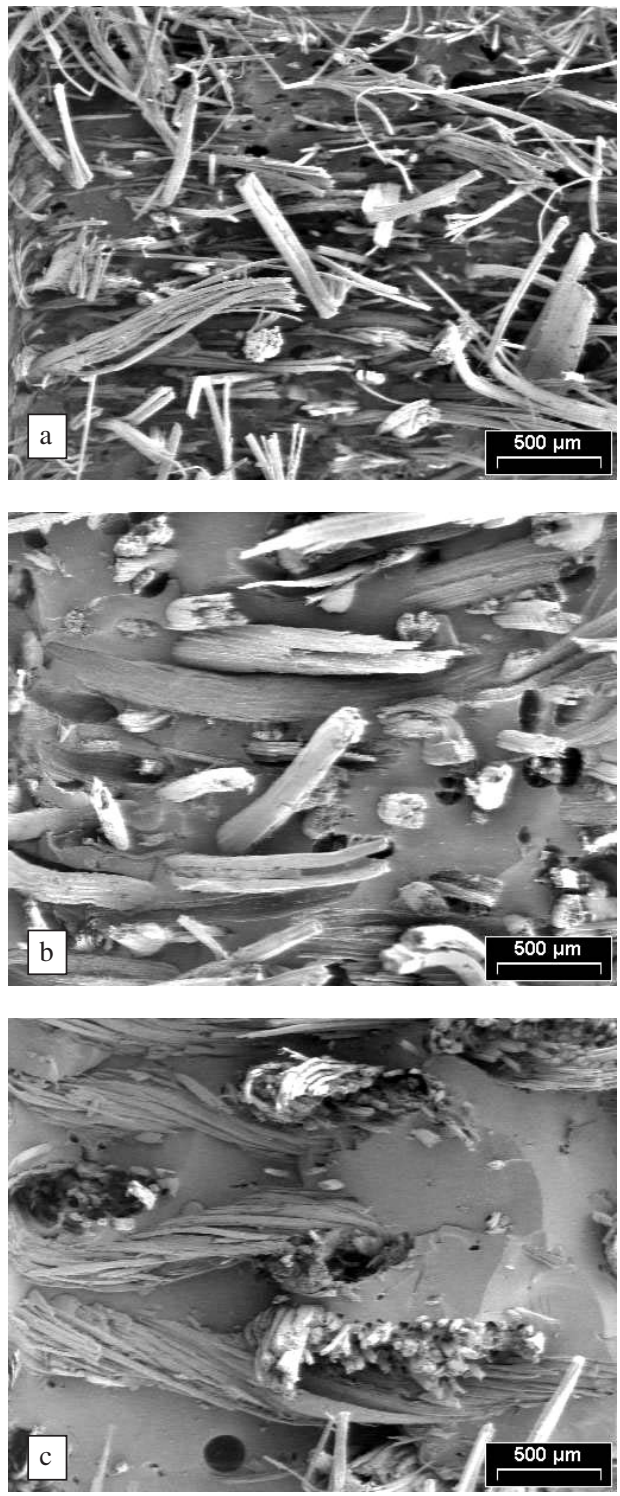


Figure 7. *Scanning electron micrograph of the fractured impact surface of the composites with acrylic resin and different reinforcements: (a) flax; (b) sisal; and (c) jute.*

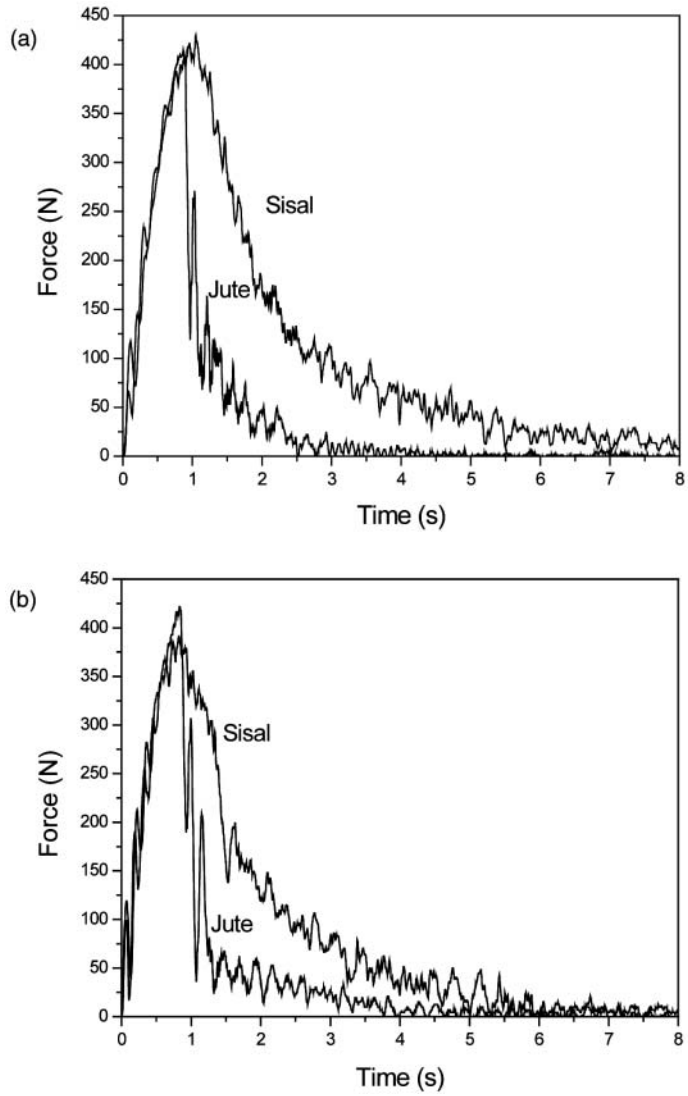


Figure 8. *Comparative force–time graphs for jute and sisal composites with two different matrices: (a) acrylic and (b) polyester.*

fibrillated than in the other composites. The lowest value for natural fibers composites was obtained with jute fibers. The index of ductility, I.D., was defined by Beaumont et al. [19] as the relation between the process propagation area divided by the total area under the curve of force versus time, and it is a value related to the toughening of the material. The high value of I.D. was found for the composite based on sisal fibers and glass fibers.

The water absorption results are shown in Table 7. Dispersion in the results may be due to the different thickness of specimens obtained after demolding. The results are lower than the previously published values of Dash et al. [20]. It could be due to the higher thickness of the specimens used and the short time for which they were immersed in

Figure 9. *Optical micrograph profiles of the fractured surface from the following composite based on unsaturated polyester and: (a) jute; (b) flax; and (c) sisal.*

Figure 10. *Optical micrograph profiles of the fractured surface from the following composite based on acrylic and: (a) jute and (b) sisal.*

Table 6. Impact tests results.

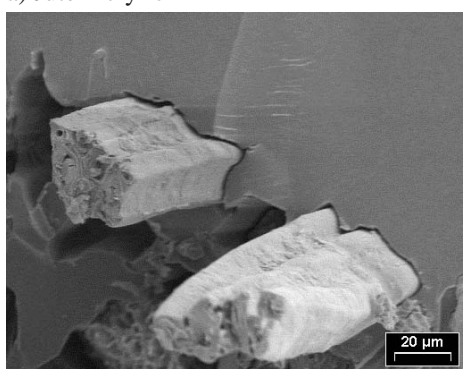
Composite	Energy (kJ/m^2)	I.D.	Energy/δ_{sp} (kJ/m^2)
Jute + Acrylic	8.8 ± 1.0	0.37	7.02
Jute + Polyester	10.6 ± 1.0	0.43	8.36
Sisal + Acrylic	12.7 ± 1.4	0.71	10.61
Sisal + Polyester	12.2 ± 1.7	0.70	10.23
Flax + Acrylic	15.0 ± 0.9	0.68	12.35
Flax+ Polyester	13.2 ± 0.9	0.69	10.88
Glass + Acrylic	98.7 ± 8.0	0.72	57.72
Glass + Polyester	106.5 ± 4.2	0.75	65.06

water (24 h). The specimens did not obtain the equilibrium value of absorption, and the center of the specimens was dry and the dried thickness depends on the thickness of the sample. As a consequence, the results can only be taken as comparative values. The results obtained in this experiment is a consequence of the different effects which can influence the

Table 7. Water absorption of composites after immersion in distilled water during 24 h at room temperature.

Composite	Increase in weight (%)	Void content (%)	Average pore diameter (mm)
Jute + Acrylic	3.00 ± 0.20	2.3 ± 1.3	0.34
Jute + Polyester	2.70 ± 0.40	2.8 ± 0.8	0.34
Sisal + Acrylic	3.20 ± 0.50	1.5 ± 1	0.68
Sisal + Polyester	2.70 ± 0.30	8.1 ± 3	0.43
Flax + Acrylic	2.80 ± 0.20	2.4 ± 1	0.19
Glass + Acrylic	0.36 ± 0.04	–	–
Glass + Polyester	0.40 ± 0.01	–	–

a) Jute-Acrylic



b) Jute-Unsaturated Polyester

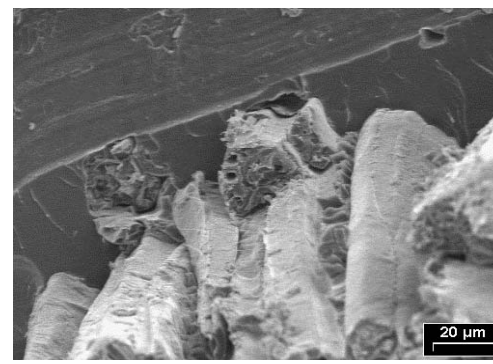


Figure 11. *SEM micrograph of the surface of the composite based on jute fibers and: (a) acrylic resin and (b) polyester resin.*

water-uptake: the (i) hydrophilic character of the resin and fibers, (ii) interfacial debonding due to the lack of adhesion between the fiber and matrix [21], (iii) void and size content in the material [22].

Glass fibers composites absorbed ten times less than the others showing that the absorption of water is a problem in natural hydrophilic fibers composites. In the case of natural fibers composites, the water-uptake values are similar, showing a slight increase with acrylic resin, which may be due to their high hydrophilic character and low fiber–matrix adhesion. Figure 11 shows the photomicrographs of the jute–acrylic and jute–polyester composites. At the interphase, the jute–acrylic composite shows higher debonding (or lower interfacial adhesion) than jute–polyester composite, in agreement with the water-uptake results.

Figure 12 shows the optical micrographs of the transverse area of the specimens. Table 7 shows the void content and average size of the void in each composite. For sisal composites, a relationship between the size and the void content appears to exist. The coalescence of the initial voids produces voids with higher diameter, hence, leading to a reduction of the void content. The composite based on sisal and polyester resin has a high void content. The average size of the void has the following order: sisal > jute > flax. The size of the voids appears to be the more important factor, because the composites with sisal and acrylic resin showed higher water-uptake and higher void size.

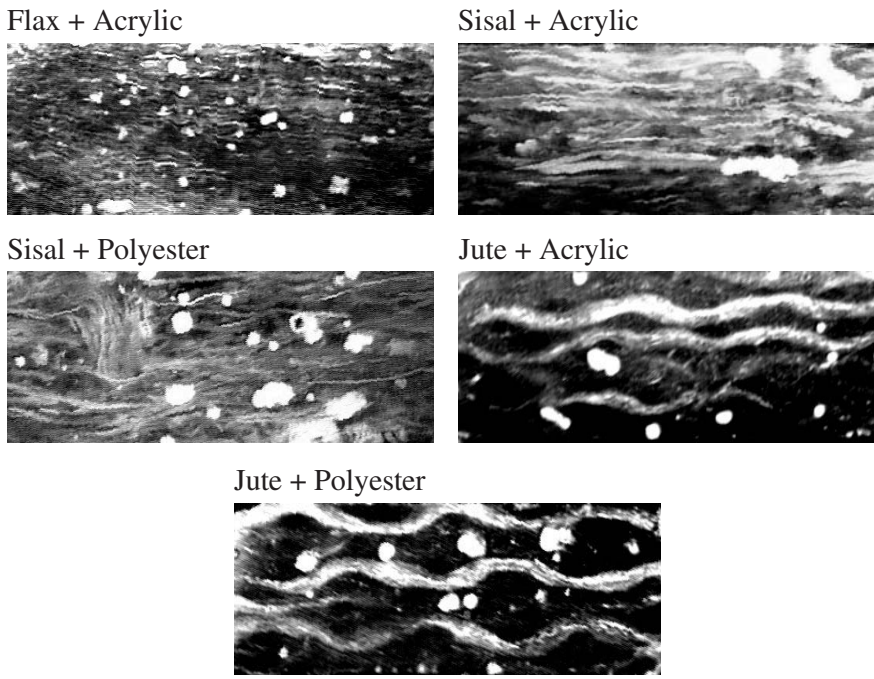


Figure 12. *Optical micrograph of the different cross-sectional areas is of the composites: (a) flax/acrylic; (b) sisal/acrylic; (c) sisal/polyester; (d) jute/acrylic and (e) jute/polyester.*

Thermogravimetric analysis was also carried out on the fibers, matrix, and composites. These experiments are important in order to determine the lowest temperature so the material can be processed without weight loss. Jute, sisal, and flax are compared in Figure 13(a). Flax fibers showed higher thermal resistance than jute and sisal fibers. This may be due to the low lignin content (Table 2). Figure 13(b) shows the result of the matrices and it can be seen that the acrylic resin showed higher thermal resistance compared to polyester. Figure 14 shows the different composites. Glass fiber composites present higher thermal resistance. Flax-based composites showed slightly higher thermal resistance compared to the other natural fiber based composites.

Composite samples were also tested under a flame**.** Specimens were put in the vertical position and exposed to a small flame for some time. Burning time was obtained for each sample. Classification of each sample depends on the burning time, the material consumption, and the production of material drops after the flame of the equipment was separated from the specimen. The time-index was defined as the time when the material was not burning (IRI), and the results are shown in Table 8. The index IRI = 0B in 4 mm means that the specimen of thickness 4 mm was exposed to a direct flame for 5 s and it burned for more than 30 s after the flame were separated from the specimen. Comparing composites with similar thickness, the sample with flax fibers and acrylic resin showed the highest burning time. The sample with higher dimensional stability after burning was the composite with jute fibers, because of the bidirectionality of the woven mat.

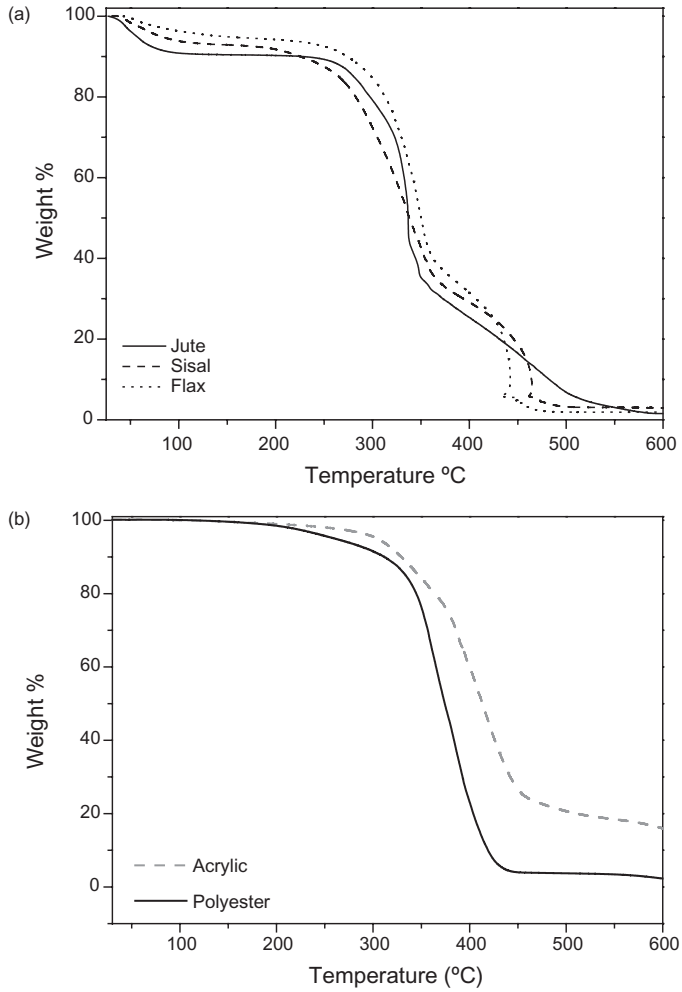


Figure 13. *Thermogravimetric analysis of: (a) different fibers and (b) matrices.*

CONCLUSIONS

Natural fibers composites with 30 vol.% of fibers content have lower mechanical properties than glass fibers composites. If specific modulus is taken into account, the difference is small. Sisal showed the lower mechanical, flame resistance behavior, and water resistance. Jute composites showed lower impact results as a consequence of the higher interface adhesion. The higher interface adhesion between the matrix and the fibers produce lack of absorption mechanism of energy in the impact test. Jute composites showed good mechanical properties compared to other natural fibers, because of higher wettability of the fibers by the low initial viscosity thermoset resin. Woven jute and nonwoven flax have similar tensile and flexural properties, and also water absorption rate. Acrylic resin showed the highest temperature resistance behavior. All the materials need flame retardants, and the flax–acrylic resin showed the highest burning time.

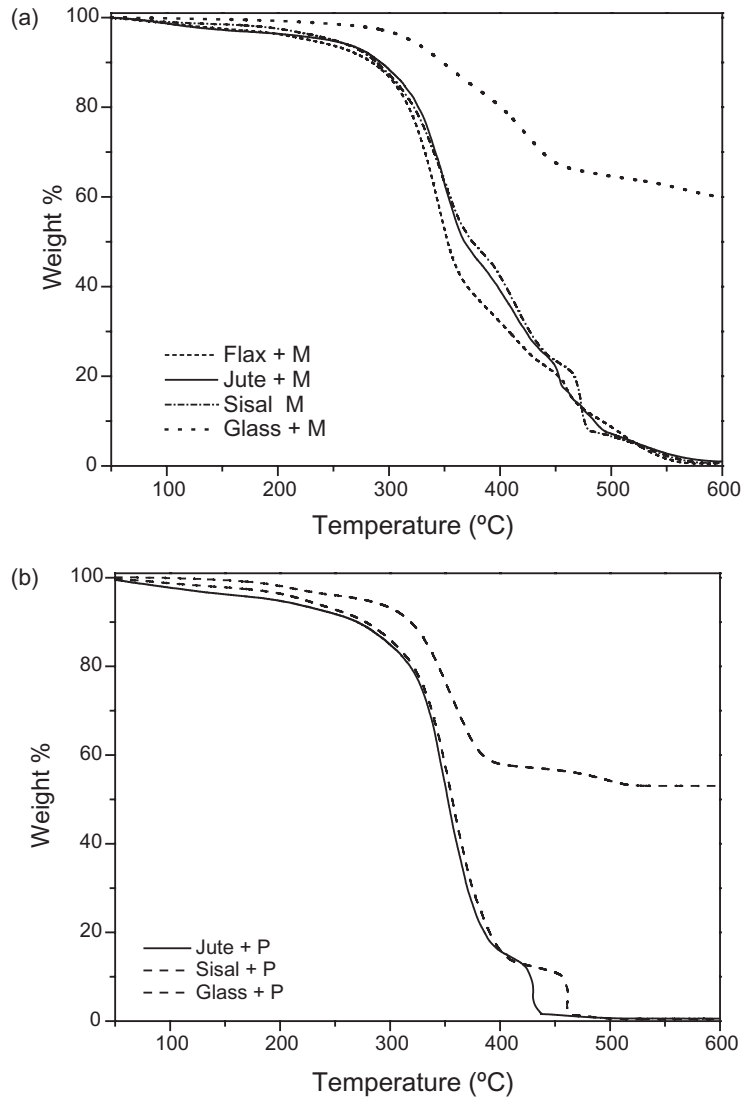


Figure 14. *Thermogravimetric analysis of: (a) composites based on acrylic resin and (b) composites based on polyester resin.*

Table 8. Response of composites to ignition by a flame.

Material	IRI	Burning time (s)
Jute + Polyester	0B in 4 mm	205
Jute + Acrylic	0B in 4 mm	220
Sisal + Polyester	0B in 4 mm	211
Sisal + Acrylic	0B in 4 mm	230
Flax + Acrylic	0B in 4 mm	270
Glass + Polyester	0B in 3 mm	193
Glass + Acrylic	0B in 3 mm	121

Natural fibers can compete with glass fibers in modulus and cost, but not in impact and strength as well as water absorption with glass fibers.

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