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Manufacturing, Mechanical and Morphological Characterization of new Natural Hybrid Biocomposite Materials of Fique – Mulberry

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Currently, the industry requires the use of new materials that have better technical characteristics and that have a minimal environmental impact. A viable option to meet these requirements are biocomposite materials reinforced with natural fibers. Recent research in this field of composite materials has sought to develop better properties in the materials, giving rise to hybrid composites, which are composed of more than one fiber as reinforcement together with the matrix; however, these studies are mainly focused on synthetic fibers, leaving a large area of research regarding natural fibers. This article describes the manufacture, mechanical, and morphological characterization of new natural hybrid biocomposite materials of fique - mulberry. Five biocomposites were elaborated under the hand lay up manufacturing technique using various layers of fique and mulberry and using polyester resin as a matrix. The mechanical characterization was carried out by means of the tensile and bending test, obtaining the best mechanical properties in the composite with the greatest amount of natural fiber, which presented a tensile stress of 30.27 MPa and a bending stress of 18.97 MPa. The morphological characterization was carried out using scanning electron microscopy, where a medium resin-fiber adhesion and a decomposition of the matrix were observed.

Keywords: *Fique, Mulberry, Biocomposite, Mechanical Characterization.*

1. Introduction

Today's industry requires that the materials from which products are manufactured have better properties with low environmental impact, in order to improve the reliability and efficiency of systems. Natural fiber reinforced composites are a viable option to meet these requirements[1].

A composite material is understood as the mixture of two or more materials in order to obtain a new material with better characteristics than the initial components. Usually they are made up of a reinforcement, in the form of fiber, and a matrix. Within the composite materials are the hybrid composite materials, which have characteristics that allow meeting design requirements that traditional composites cannot achieve[2].

A hybrid composite material is defined as a composite that has two or more reinforcing fibers along with the matrix[3]. Various hybrid composite materials have been developed and used in industrial applications such as brakes, automotive clutches and cams, bearing components, and parts used in mining and agricultural machinery[4].

The most common fibers for the development of hybrid composites are the traditional synthetic fibers (carbon, glass and Kevlar) in different configurations such as carbon-glass[5,6], Kevlar-glass fibers[7,8], carbon-basalt fibers[9] and Kevlar - carbon-glass fiber[10].

Recently, hybrid composites reinforced with natural fibers are taking great importance due to the need for environmental conservation and the development of sustainable products, however studies on these products are scarce[11].

Natural fibers bring advantages to composite materials due to their relatively low weight, low cost, less impact on processing equipment, good mechanical properties. Besides being an abundant, biodegradable and renewable raw-material which if flexible during processing without risk to health and contamination[12]. Within these natural fibers are the fiber of mulberry and fique.

Mulberry fiber is obtained from the bark of the mulberry tree (Broussonetia kazinoki Siebold). This plant is mainly cultivated in Asian countries, the most representative being Thailand, China, India, Laos and Myanmar[13]. Its use is mainly focused on paper manufacturing, the food industry, the pharmaceutical industry, and the textile industry[14]. Mulberry fiber has high amounts of holocellulose which leads to high mechanical and chemical resistance[15].

Fique is a fiber produced in South America, in countries such as Colombia, Brazil, Ecuador and Venezuela. It is mainly used in the manufacture of coffee sacks, ropes and handicraft products[16]. Fique has good mechanical properties, which is why it has been used in the manufacture of various composite materials[17-22].

This article presents the manufacture and mechanical characterization (tensile and bending test), and morphological

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characterization (Scanning electron microscopy) of new fique-mulberry hybrid composites with a polyester matrix.

2. Methods

2.1. Materials

As reinforcement material, fique fiber and mulberry fiber were used in random configuration (Mat). The fique fiber was supplied by the foundation of Barichara, Santander Colombia and the mulberry fiber by the Hanspaper industry company in Jiangsu, China. Fique fibers have an average content of 52.3% cellulose, 23.8% hemicellulose and 23.9% lignin[23] while mulberry fibers have 42.8% cellulose, 29.5% hemicellulose and 21.3% lignin[24].

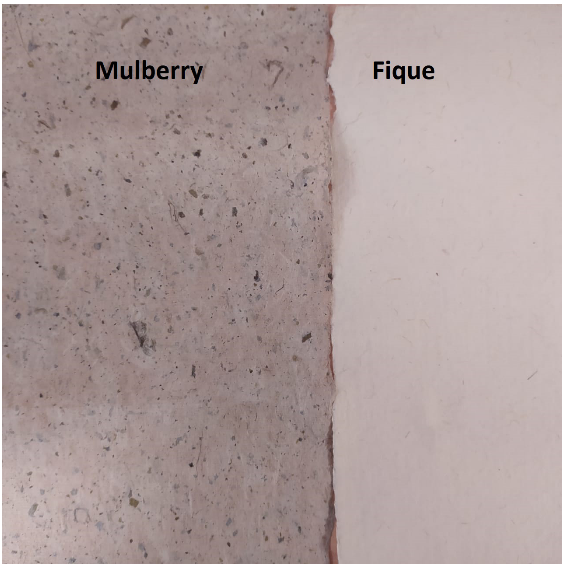

Figure 1. Mulberry and fique fibers used.

The randomization of mulberry and fique fiber was obtained manually. Got et al.[25] describe the process for obtaining this configuration in mulberry, and Gómez Suárez et al.[26] do it for fique. The two processes present very similar characteristics since they follow the methodology for the manufacture of handmade paper (also known as Hanji) where the fibers initially receive a thermal treatment with the objective of eliminating humidity, then they are macerated to obtain short fibers, subsequently they are classified using a sieve and, finally, the random arrangement is pressed to obtain the sheet (random configuration). Figure 1 shows the sheet in random configuration of each of the fibers.

The mulberry fibers presented a diameter of 14.156 ± 2.81 μm while the fique fibers presented a diameter of 17.168 ± 2.299 μm. Figure 2 shows the images obtained by scanning electron microscopy of the mulberry and fique fibers together with their measurements.

The density of the sheet (random configuration) of the mulberry fiber used was 0.254 g/cm^3 and of the fique was 0.782 g/cm^3. Additionally, the thickness of the fique sheet was 340 ± 12 μm while the mulberry sheet was 105 ± 8 μm.

The matrix used in the manufacture of the material was polyester, thermosetting polymeric resin, reference 102, acquired from the company Ingequimicas of Bucaramanga, Santander, Colombia. The lowest resin viscosity (Approximately 150 cP) available in the local market was used to facilitate its application. The percentage of resin-catalyst in weight was 100:2 respectively. The catalyst used was methyl ethyl ketone peroxide (MEK peroxide).

2.2. Manufacture of test specimens

The hybrid composite materials were manufactured following the manual hand lay up manufacturing technique. Initially, five laminates were manufactured with different

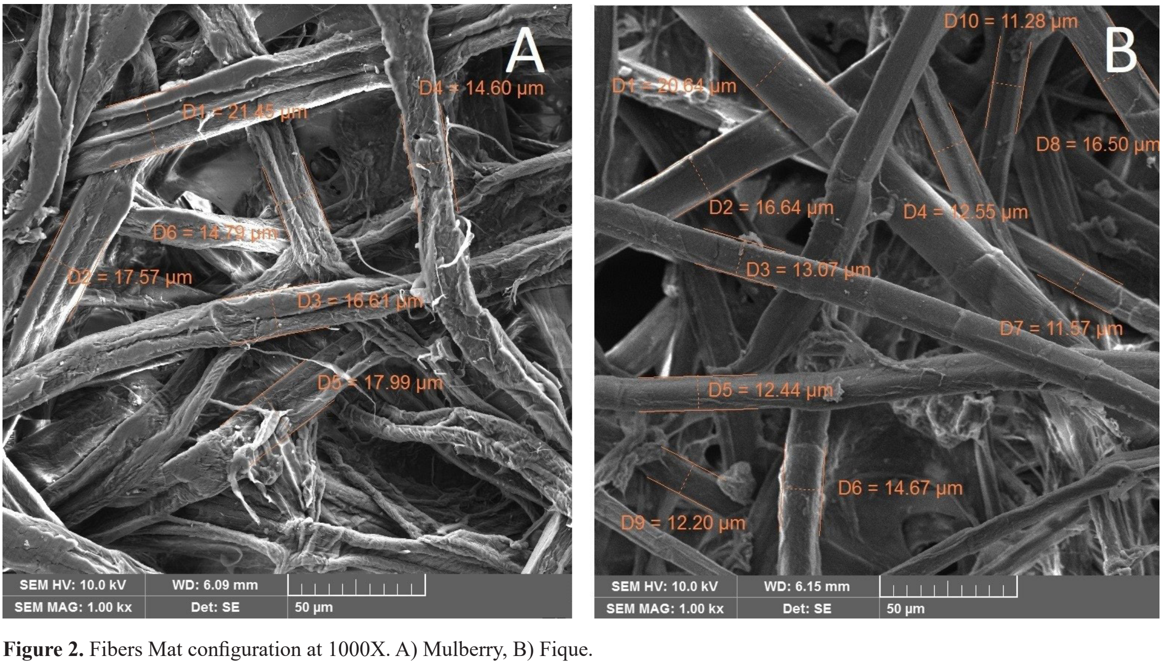

Figure 2. Fibers Mat configuration at 1000X. A) Mulberry, B) Fique.

amounts of fique and mulberry fiber content with a geometry of 20 cm wide, 35 cm long and 2.5 mm thick were made by stacking layer after layer of fique and/or mulberry, impregnating them with the polyester resin using a brush on a flat mold previously moistened with release agent; each layer was pressed with a metallic roller to improve the adhesion between the fiber and the matrix. Curing was performed at room temperature at a temperature of 24.2 +/- 1.2 °C and a maximum humidity of 76.2%. No post-curing was performed on the materials. The manufacturing process and the manufactured specimens are shown in Figure 3.

The composition of each of the biocomposite materials manufactured, together with the amount of mulberry and fique fiber used, is shown in Table 1.

The number of fique and mulberry fiber layers in biocomposites 1 and 2, respectively, were selected as the maximum amount so that when manufacturing the biomaterials their thickness would not exceed 2.5 mm, since this is a parameter established by the standards used in the mechanical characterizations. The fiber percentage of these bicomposites was not the same because the granulometry and density of the fique and mulberry fiber sheets are different.

Composites 3, 4 and 5 were manufactured by increasing the number of layers of mulberry fiber and decreasing the number of layers of fique fiber. It was ensured that the thickness of the biocomposites did not exceed 2.5 mm in thickness.

The biocomposite laminates were cut according to the geometry established in each characterization test, using a laser cutting machine. Five test specimens of the manufactured materials were obtained for each of the tests. The five specimens were taken from the same laminate, previously manufactured, to ensure the same amount of fiber in each sample.

2.3. Tensile test

The tensile test was performed according to ASTM D3039/D3039M[27], applied to 5 test specimens of each material with a geometry of 25 cm long by 2.5 cm wide by 2.5 mm thick as established in the standard. The results presented are the average of those obtained in the five test specimens.

To obtain the maximum tensile stress and the modulus of elasticity of the materials, a 10 kN universal machine, MTS model C43.104, was used. The test speed was defined at 2 mm/min with an ambient temperature of 23.2 +/- 3.4°C.

Figure 4 shows the mechanical characterization in tensile of the biocomposite 3 hybrid material of fique-mulberry.

2.4. Bending test

The bending test was performed at three points following ASTM D7264/D7264M-21[28]. The geometry of the five test specimens for each of the materials was 100 mm long, 2.5 mm

Figure 3. Manufacturing. A) Manufacturing process, B) Manufactured specimens.

Table 1. Manufactured biocomposites.

Material	Description	Density (g/m^3)
Biocomposite 1	6 layers of fique fiber corresponding to 31.25% by weight of the biocomposite	1.29
Biocomposite 2	10 layers of mulberry fiber corresponding to 15.32% by weight of the biocomposite	1.19
Biocomposite 3	3 layers of fique fiber corresponding to 17.67% by weight of the biocomposite 5 layers of mulberry fiber corresponding to 5.27% by weight of the biocomposite	1.23
Biocomposite 4	2 layers of fique fiber corresponding to 13.24% by weight of the biocomposite 6 layers of mulberry fiber corresponding to 7.328% by weight of biocomposite	1.14
Biocomposite 5	1 layer of fique fiber corresponding to 7.215% by weight of the biocomposite 8 layers of mulberry fiber corresponding to 10.719% by weight of the biocomposite.	0.99

thick and 13 mm wide, maintaining a minimum thickness-to-span ratio of 32:1 (Span) in accordance with the standard.

The test was carried out in a universal machine MTS model C43.104 of 10 kN at a speed of 1 mm/min and a temperature of 24.3 ± 3.2°C.

2.5. Scanning Electron Microscopy Test

Specimens of 1 cm x 1 cm were evaluated to assess the adhesion between the different layers of fique and mulberry with the resin in the materials. The samples were obtained from the laminates previously manufactured using the laser cutting machine. The analysis was performed on the fractured surfaces of the composites previously obtained in the tensile test.

The specimens were covered with a small layer of gold to improve their conductivity and to be able to carry out the test with scanning electron microscopy.

Scanning electron microscope Tescan model MIRA 3 FEG-SEM with secondary electron detector model A65c SED was used. Images were captured at different focal planes for analysis at 100X magnifications.

2.6. Statistical analysis

For the estimation of the mechanical properties, the mean and standard deviation were used. In order to define if the differences obtained between the results of the different biomaterials are statistically significant, the ANOVA or analysis of variance was used, in which if the P value is less than the level of significance (defined as 0.05), it is concluded that at least one mean of the mechanical properties is different. Additionally, Scheffé post hoc tests were performed to perform multiple comparisons of the means and recognize which of them was different.

Figure 4. Tensile test.

3. Results

3.1. Tensile test

Figure 5 shows the tensile stress vs. strain curves of the different biocomposites. According to the results obtained, a linear elastic behavior is observed for all configurations. Additionally, the materials present a brittle behavior, typical of the nature of composites with a thermosetting matrix.

Table 2 shows an increase in tensile stress with a greater amount of natural fiber (the sum of fique and mulberry) in the composite. This is due to the fact that there is a greater amount of reinforcement that provides greater strength and stiffness to support the loads. Hidalgo et al.[29] obtained a similar behavior in composite with polyethylene and aluminum matrix with fique fiber reinforcement to the greater amount of natural fiber that composed the composite.

The best stress behavior was obtained in biocomposite 1, which only has fique fiber, being 53.02% superior to biocomposite 2, which only has mulberry fiber, being the latter the one that presented the worst mechanical behavior of all the materials.

Table 3 shows the ANOVA tests where the significant differences between the means of the different mechanical properties of the manufactured biocomposites are evaluated. As evidenced in the tensile test, a P value of less than 0.05 was obtained, indicating that at least one of the materials has a different mean than the others. When performing the post hoc tests, Scheffé values greater than 0.05 were obtained only between biocomposites 4 and 5, indicating that these two biomaterials do not present statistically significant differences between their tensile stresses.

Regarding the values of the modulus of elasticity, the results are in the same order in the composites containing

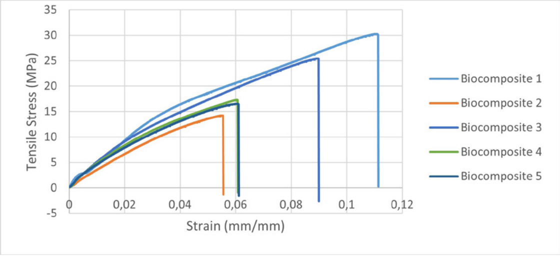

Figure 5. Stress-strain curve.

Table 2. Tensile mechanical properties.

Biocomposite	Tensile stress (MPa)	Elasticity module (GPa)	Strain (%)
Biocomposite 1	30.27 ± 1.53	0.50 ± 0.0134	10.92 ± 0.250
Biocomposite 2	14.22 ± 0.332	0.37 ± 0.0195	5.52 ± 0.0976
Biocomposite 3	25.44 ± 0.824	0.50 ± 0.0336	8.97 ± 0.198
Biocomposite 4	17.28 ± 0.653	0.54 ± 0.0158	6.03 ± 0.448
Biocomposite 5	16.50 ± 0.753	0.52 ± 0.0305	6.06 ± 0.667

fique fiber; however, the lowest stiffness value is obtained in the mulberry fiber only composite, it should be noted that biocomposite 2 is the one with the least amount of fiber, which directly affects the mechanical properties. In the statistical analysis of the modulus of elasticity shown in Table 3, a P value of less than 0.05 is obtained, which indicates that at least one material presents a stress modulus statistically different from the others. When the post hoc tests were performed, the only biomaterial that presented a Scheffé value lower than 0.05 was biomaterial 2, which indicates that this material presents significant differences between its modulus of elasticity and the others.

In the percentage of deformation, it is evident that there is greater elongation the greater the amount of fique fiber in the biocomposite material. The lowest deformation is presented in the mulberry biocomposite material. This can be explained because the mulberry fiber has an ultimate elongation percentage of 3.5% to 5.5%[30] while the fique fiber is 9.8%[31], which allows composites with higher fiber fique present greater ductility. As a result of the ANOVA, a value of P less than 0.05 was obtained, which indicates that at least one material presents a percentage of deformation statistically different from the others. With the Scheffé post hoc tests, it was shown that the difference in the percentage of deformation of biocomposites 2, 4 and 5 is not statistically significant.

3.2. Bending test

Figure 6 shows the flexural behavior of the different biocomposites. According to these results, there is an improvement in bending strength as the amount of fiber (the sum of fique and mulberry) increases.

Biocomposite 1 presents the maximum bending stress of 18.97 MPa, this being 21.79% higher than biocomposite 3, which reports a stress of 15.58 MPa and 31.87% better than biocomposite 4 with a value of 14.39MPa The lowest value obtained was for biocomposite 2, composed of only mulberry fiber, being 12.21 MPa, followed by biocomposite 5 with a value of 13.25 MPa. The bending stress tests shown by Hidalgo et al.[32] presented a behavior similar to that obtained in the present study, in fique fiber-reinforced biocomposites with a greater amount of fiber within the composite.

Table 4 shows in the ANOVA analysis where a P value below 0.05 was obtained, which indicates that at least one biomaterial presents a bending stress statistically different from the others. When performing the Scheffé Post Hoc analysis, it was shown that between biomaterial 2 and 5 there are no significant differences, just as between biomaterial 3 and 4, this can be explained because they have similar percentages of natural fiber.

3.3. Scanning electron microscopy

In relation to the adhesion of the five biocomposites, a medium interaction between the fiber and the matrix with defects and gaps between the fibers and the resin is evidenced (as shown in Figure 7). This is due to the manual manufacturing system employed, and to the hydrophilic characteristics of the fibers and the hydrophobic nature of the matrix[33].

Additionally, the presence of cracks at the interface of the biomaterials is denoted due to the use of laser cutting machine to obtain the specimens. This phenomenon occurs because the thermal conductivity of the matrix is lower than that of the fibers, so the energy concentration is higher in the polymeric material, affecting its surface as mentioned by Riveiro et al.[34].

The low adhesion between the fiber and the matrix negatively affected the mechanical properties of the manufactured composite biomaterials, due to the fact that there is a low load transmission from the resin to the fiber, additionally the

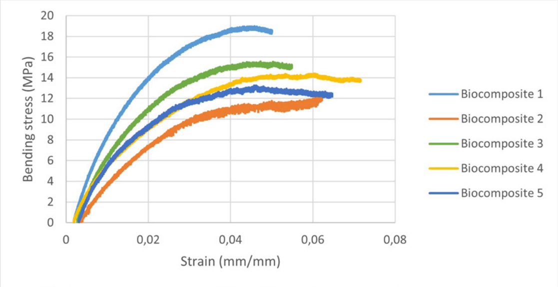

Figure 6. Bending stress.

Table 3. ANOVA tensile test.

Property	Source	Sum of squares	Degress of freedom	Mean square	FO	p
Tensile stress	Biocomposites	926.8	4	231.7	282	<0.001
	Residuals	16,4	20	0.822		
Elasticity module	Biocomposites	0.097	4	0.0242	42.2	<0.001
	Residuals	0.0115	20	0.0006		
Strain	Biocomposites	107.12	4	26.779	177	<0.001
	Residuals	3,03	20	0.151		

Table 4. ANOVA bending test.

Source	Sum of squares	Degress of freedom	Mean square	FO	p
Biocomposites	136,2	4	34,06	37,9	<0.001
Residuals	18	20	0,899		

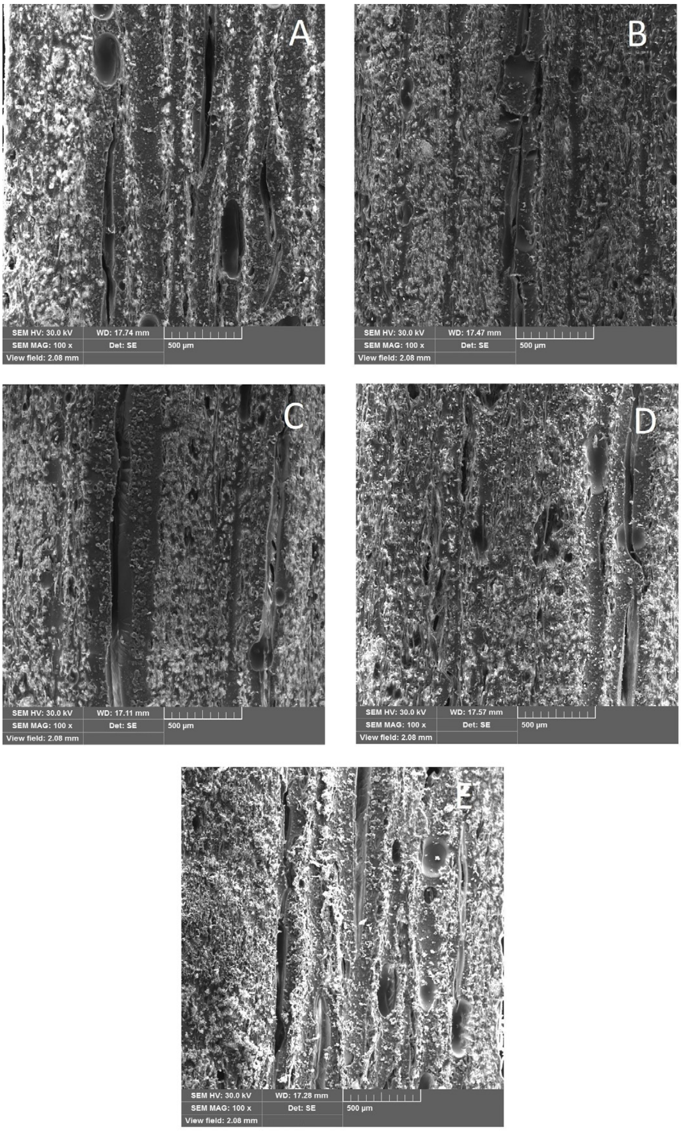

Figure 7. Electron microscopy. A) Biocomposite 1, B) Biocomposite 2, C) Biocomposite 3, D) Biocomposite 4, E) Biocomposite 5.

defects in the interface act as stress concentrators, according to as indicated by Gómez et al.[35].

It is recommended to use the water jet cutting technique in future works, since the affectation to the matrix is lower compared to laser cutting as mentioned by Vinayagamoorthy and Rajmohan[36].

4. Conclusions

Five materials were manufactured following the manual Hand lay up manufacturing technique using fique and mulberry fibers. Biocomposites with a single type of fiber and hybrids were made by mixing various percentages of both types of fibers (Fique - Mulberry).

An increase in the mechanical properties was evidenced by means of the tensile and flexural test in the different biocomposites with a higher percentage of fiber used in their manufacture.

The morphological analysis with scanning electron microscopy allowed observing that there was a medium adhesion between the matrix and the fiber due to the hydrophobic nature of the matrix and the hydrophilic characteristics of the fibers; additionally, because laser cutting was used to obtain the test specimens, delamination of the matrix occurred.

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