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Influence of Alkaline and Enzymatic Treatments on the Properties of Doum Palm Fibres and Composite

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Abstract: In the presents study, an analysis of the influence of alkaline treatment for different concentrations as well as that of the enzymatic treatment on the morphology and on the mechanical properties of the leafstalk doum palm fibres is detailed to improve the hydrophobicity, the compatibility fibre/matrix and the composite mechanical resistance. The fibre surface topography has been characterized by SEM. And the obtained images show that the alkaline treatment gives porous fibres and the biological treatment gives smooth surfaces. The results confirm that both treatments eliminate the fibre residual impurities and influence their properties. A tensile test series were carried out to study the effect of the treatments on the mechanical features of the leafstalk doum palm fibres. The chemical treatment for a concentration of 1.5 N has improved considerably the mechanical properties. The composites used were made of doum palm fibres as reinforcement for epoxy resins. The composite mechanical properties have been studied from flexural tests. This study presents the results of an experimental investigation in order to select the best mechanical characteristics of epoxy resin/doum palm fibres.

Key words: Natural fibres, alkaline treatment, enzymatic treatment, mechanical properties, composite

INTRODUCTION

The choice of fibres depends on their sensitivities and their mechanical, thermals and physical properties. Among the used fibres let's mention: Fibres of ananas, abaca, sisal, esparto, kenaf, the textile palm.

The literature has show that the fibre extraction influences their length distribution, their mechanical properties and their components (Sreekala *et al*., 1998; Malainine *et al*., 2003). The lignocellulosic fibres are constituted by micro fibrils and an amorphous matrix made up of lignin and hemicellulose (Murali and Mohana, 2007).

Surface modifications is usually applied to impart bonding and adhesion affinity to matrices, such as the thermal treatment (Bledzki and Gassan, 1999), alkaline treatment (Cao *et al*., 2006) and enzymatic treatment (Akin *et al*., 2001).

The alkaline treatment can clean the fibre surfaces, increase its roughness modify its composition and stop the process of the moisture absorption as shown by Mohd *et al*. (2007). This treatment requires a great quantity of energy, water and chemical product. It improves interfacial adhesion (Zafeiropoulos *et al*., 2007), but it can be expensive. The use of the enzymes and more precisely the pectinase can contribute to reduce the environmental impact and the expenses of development (Calafell and Garriga, 2004). It is a biological catalyst and an enzymatic preparation with several strongly effective elements for the depolymerization of vegetable pectins.

The researchers used enzymes for extracting fibre hemp, flax and cotton (Akin *et al*., 2001; Calafell and Garriga, 2004; Evans *et al*., 2002). After cleaning with the acid pectinase, the cotton fibres have an almost intact cellulose structure. Compared to alkali process they note a less significant loss of weight and a better resistance (Rosenbohm *et al*., 2003).

Early research studies on natural fibres reinforced composites. Herrera-Franco and Valadez-Gonzalez (2005) studied the mechanical behaviour of short natural fibre reinforced HDPE. It resorts that the fibre-matrix interaction depends on the surface properties of the fibre, witch increases the area of contact, exposes further the cellulose microfibrils and improves fibre wettability and impregnation.

This study is composed of three phases, the first interests the understanding of fibre mechanical properties, the second proposes a preliminary study of their interfacial features and the third deals with the effect of the treatment on the behaviour of the fibre and epoxy resin/doum palm fibre composite.

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MATERIALS AND METHODS

The tested fibres have been extracted from the doum palm tree. That tree belongs to the family of the monocotyledons, like the graminy and musaces. The leaf is composed of folioles on which grow the leafstalks. The obtained fibre length varies according to the tree type, its form, age.

Mechanical extraction: The mechanical extraction of natural fibres has been made with raspadols which, by a combined action of scratching and hyping, scratches the pulp and frees the fibre (Ghali *et al*., 2006). Once extracted and washed, the fibres undergo an operation of drying and combing. The first step can be accomplished naturally by exposing the product to sunlight or artificially using an oven. Therefore the obtained fibres are stuck against each other and a combing operation is required.

The mechanical extraction needs much water and causes a change in the fibre characteristics because of mechanical stress which it undergoes.

Enzymatic treatment: In this study, fibres have been extracted from leafstalk of the doum palms, by using the pectinase polygalacturonase as enzyme. The enzyme activity is 3 to 9 units mg^{-1} at 25°C, witch can release a micro mole of galacturonic acid per minute (Akin *et al*., 2001). Some fibres have been weighted using an accurate balance then immersed in a pectinase bath the concentration of which is 10 ml L^{-1} during 48 h under the ambient temperature. Once this is done fibres have been rinsed with tepid distilled water. Then brushed to separate them and left in the air to dry before pulling the whole set in an oven during 3 h at 90°C. Finally the product has been stored in a bag.

Chemical treatment: This treatment consists on dissolving lignin and hemicelluloses using aqueous solution in order to recover cellulose fibres. The process is less harmful and does not attack the fibres mechanically. It is based on the basic hydrolysis of the leaf components.

In order to quantify the effect of the concentration, palms fibres have been plunged in a NaOH solution of variable concentration from 1 N to 3 N. The mixture is put in a drying oven during 2 h at 90°C (Cao *et al*., 2006).

To eliminate the residual aqueous solution traces fibres have been washed and rinsed several times then bleached with chloride at the ambient temperature and finally rinsed to eliminate the chlorine residual traces.

Table 1: Nomenclature used for the final composites

Description	Abbreviations
Epoxy resin	ER
Epoxy resin/untreated doum Palm fibre	ER/PF
Epoxy resin/treated doum Palm fibre with 1.5 N	ER/1.5PF
Epoxy resin/treated doum Palm fibre with 3 N	ER/3PF

Composite preparation: Manufacturing has been done by using compression molding techniques. Doum palm fibres are dried in an air-circulating oven at 80°C during 24 h. Then the fibres are cut to the desired length, 20% of the fibre volume has been chosen, the epoxy resin has been mixed with 1 wt.% of accelerator. The whole mixture is put in glass mold, the fibres are added to the mixture by keeping the same fibre direction. The mold is closed at a 0.5 kg cm^{-2} pressure during 10 h from each flexural test, five specimens were prepared by cutting rectangular samples with 80×15×4 mm^3. Table 1 presents the abbreviations of the final composite.

Mechanical properties: The tensile tests on the doum palm fibres are carried out under standard conditions of relative humidity and temperature according to the French norm NF G 07-002. For each test the fibres characterization is made on 10 samples, chosen according to their titles (linear densities). The speed of adopted test is 5 mm min^{-1}. The length between grips is 50 mim^{-1}. For reasons of simplification, the fibres are assumed to be of circular form where the diameter is given by:

$$De(\mu m) = 1000 \times \sqrt{\frac{4 \times \text{linear density(mtex)}}{\pi \times \text{density}(g\ cm^{-3})}}$$

The tensile modulus E is given by the tangent at origin of the stress-strain curve. Flexural tests (three point bending) of composite were realized using the same machine under the same conditions. Five samples were tested in each experiment and the average value is reported. The evolution of the stress (MPa) with respect to the strain (%) is plotted.

RESULTS AND DISCUSSION

Fibre morphology: The effect of modification upon the fibre was examined using a SEM Microscope (PHILIPS XL-30). Prior to the analysis, the fibres were immersed in gold, in order to ensure their conductivity. The examination of the treated and untreated fibre surfaces revealed a dimensional variation of the doum palm fibre. The diameter of untreated leafstalk fibres is of the order of 280 μm. One notes that the diameter of microfibrils of cellulose is about 7.64 μm on the sun side and 8 μm in inside of the leafstalk. The structure of the doum palm

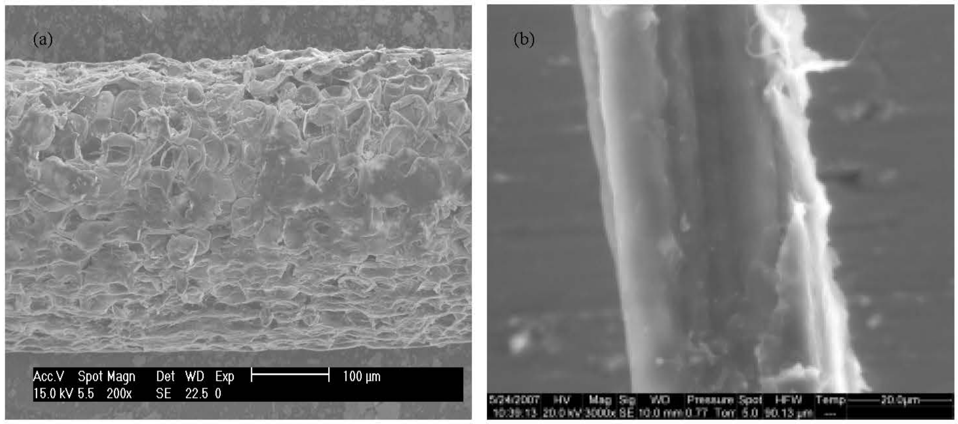

Fig. 1: Influence of the biological treatment on the leafstalk fibres morphology

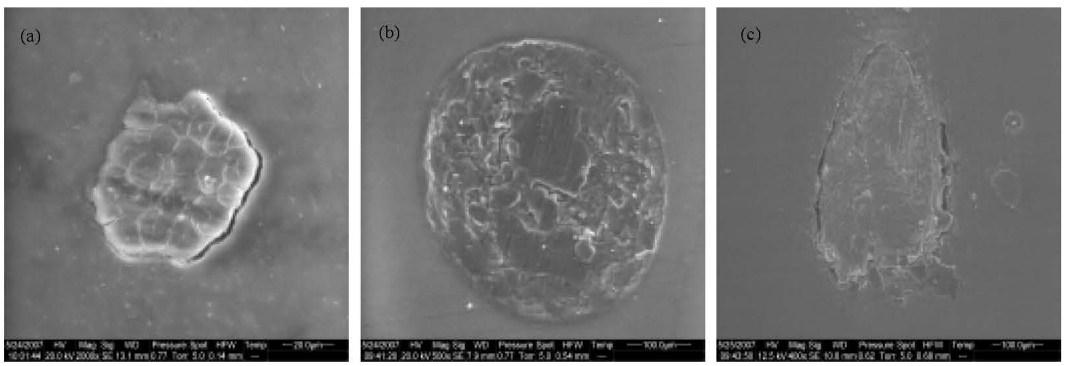

Fig. 2: (a) Biological treatment of the foliole fibres, (b) Biological treatment of the leafstalk fibres and (c) Alkaline treatment of the leafstalk fibres

fibre is similar to that of the natural fibres (sisal, esparto) (Bledzki and Gassan, 1999). It represents a natural composite where, the ultimate fibres of cellulose form the reinforcement and the other substances form the matrix. Figure 1a shows the untreated leafstalk fibre with an external depot of lignin on the surface. Figure 1b shows that the biological treatment has reduced the extent of lignin and improved the surface of the leafstalk fibre. It appears clearly that all the alkaline treatment decrease the section of leaf stalks doum palm fibres. For concentrations 1 and 3 N the diameter of the leafstalk fibres are 280 μm and 209 μm, respectively; however the biological treatment has given fibres which contain less gummy substances then the alkaline treatment.

The alkaline treatment has eliminated impurities of lignin and of hemicelluloses in fibres without attacking cellulose microfibrils. This is conform to studies already made on kenaf fibres (Mohd *et al*., 2007). The biological treatment has improved the surface as it gives smooth one.

From Fig. 2, it can be seen a variation of fibre shapes due to different treatments. Leafstalks and folioles fibres treated by enzymes have a circular section as fibres of the sisal, palm, banana and bamboo (Murali and Mohana, 2007), but alkaline treatment yield ellipsoidal fibres.

Mechanical properties of fibres: In Fig. 3a-c, typical stress-strain curves for treated and untreated leafstalks doum palm fibres are plotted. For each treatment, we present some tests as well as then middle curves.

As can be seen from Fig. 3a-c all grades of doum palm fibres exhibited essentially two zones: The first is linear where the stress is proportional to the strain and the second is non linear and presents a non elastic behavior. The fibre section of the doum palm is a parameter which influences the mechanical properties. The alkaline and the enzymatic treatments have reduced the fibre diameter. The near observation of the stress evolution shows a small drop followed by resumption for treated leafstalks fibres

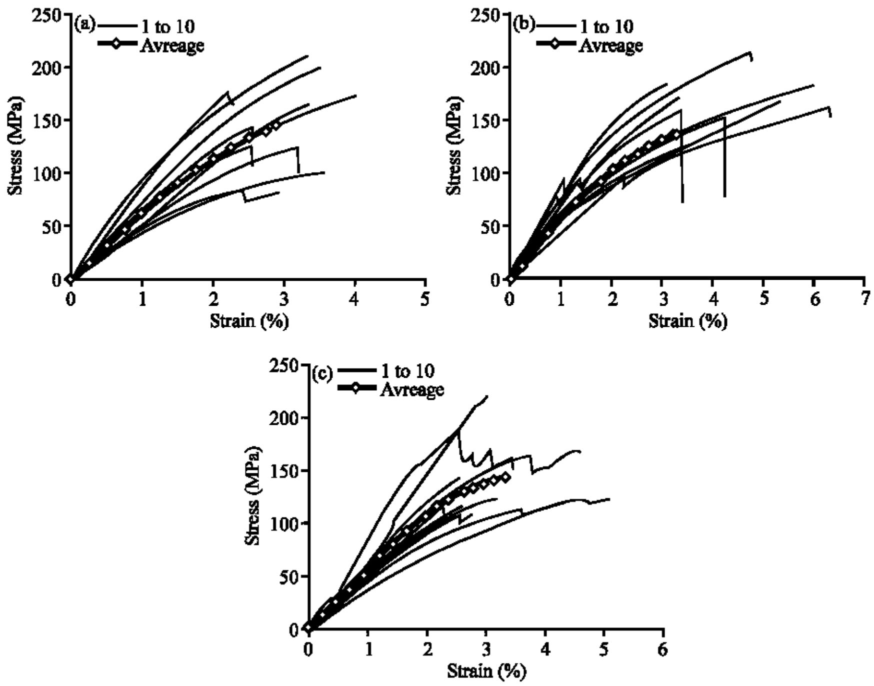

Fig. 3: Typical stress-strain curves for treated and untreated leafstalks doum palm fibres. (a) Untreated, (b) Alcaline treatment 1 N and (c) Enzymatic treatment

Table 2: Tensile properties for natural fibres

Fibers	Average tensile Modulus E (GPa)	Average tensile strength σ_r (MPa)	Average tensile strain A (%)	Specific tensile strength σ_r /d (MPa/ (g cm^{-3}))	Specific tensile modulus E/d (GPa/ (g cm^{-3}))
Leafstalks					
Untreated	5.76±1.35	149.72±34.25	3.19±0.38	118.82	4.57
Treated 1 N	6.12±1.23	168.04±16.89	4.46±0.94	130.26	4.74
Treated 1.5 N	7.49±1.24	212.82±33.23	4.81±0.55	164.97	5.80
Treated 2.5 N	5.65±1.70	183.16±31.87	3.89±0.85	141.98	4.31
Treated 3 N	5.75±2.52	100.55±19.94	2.52±0.39	76.17	4.35
Enzymatic treatment	5.36±0.79	142.96±31.05	3.30±0.79	216.60	8.12
Esparto	11.18	264.79	4.08	196.14	8.28
Palm	2.75	377.00	13.71	366.01	2.66
Sisal	10.40	567.00	5.47	391.03	7.17

in the zone between 50 and 120 MPa. This for treated leafstalks fibres with an aqueous concentration of 1, 1.5 N and also those that incurred an enzymatic treatment.

This phenomenon results from the microfibrils break that generates a decrease of the stress until a final breaking of the fibre. For untreated fibres, gummy substances in the fibre ensure the cohesion between microfibrils of cellulose. Mechanical properties of the doum palm fibres well as that of esparto and of sisal are shown in Table 2 (Ghali *et al*., 2006; Murali and Mohana, 2007). The treatments influence the mechanical properties such as the tensile modulus, the tensile strain and the tensile strength.

Even if the treatment has reduced the fibre diameter of the tensile modulus, the tensile strain and the tensile strength have been improved for a concentration of aqueous solution equal to 1.5 N. The enzymatic treatment has eliminated supplemental impurities of the fibre, but has not significantly improved the mechanical properties. If the concentration of aqueous solution exceeds 3 N, properties of leafstalks fibres will start to degrade.

The comparison between the mechanical properties only is not sufficient as fibres present neither the same densities nor the same linear densities.

Then, it is worth better, to well characterize fibres, to compare specific ratios of specific tensile strength and specific tensile modulus for different natural fibres. The best ratio is the best the fibres are as they have better resistance for low densities. The specific tensile strength and modulus of various fibres are also listed in Table 2. The enzymatically treated fibres have specific tensile strength ratios greater than that of untreated doum palm fibres or alkali treated. These ratios near of those of sisal and the palm (Murali and Mohana, 2007) and superior to those of the esparto (Ghali *et al*., 2006) show a good compromise between the resistance and the density.

Table 3: Flexural properties of composites

	Flexural modulus (GPa)	Flexural strength (MPa)	Strain to break (%)
ER	0.887±0.07	34.00±0.56	12.96±0.25
ER/PF	1.230±0.12	42.49±6.23	6.66±0.71
ER/1.5PF	1.935±0.14	62.92±4.25	5.61±0.398
ER/3PF	1.752±0.14	57.38±6.19	4.76±0.57
Resole-epoxy resin hemp fibre*	1.750±0.150	20.00±4.0	3.50±0.5
Resole-epoxy resin ramie fibre*	1.790±0.115	22.00±1.5	1.50±0.15
Resole-epoxy resin flax fibre*	0.420±0.025	5.00±0.9	>3.15±0.5

*: (Maffezzolia *et al*., 2004)

Mechanical properties of composite: Flexural properties of the composite achieved as well as others presented at the literature are shown in Table 3. One can observe that flexural strength and flexural modulus are improved after the treatment.

The treated composite results in better properties. Alkaline treatments results in freeing the hydrogen bonds making them more reactive and giving some porous fibres. It generates the increase of the void content in fibres, the improvement the wettability and the fibre/matrix contact. All these factors provide better mechanical properties compared to the ER/PF. The proprieties of the doum palm fibre/epoxy resin are comparable to resole-epoxy resin hemp and ramie fibre but superior to resole-epoxy resin flax fibre (Maffezzolia *et al*., 2004; Van-de *et al*., 2003) show that flax fibre reinforced can be a good reinforcement for epoxy composites. Nevertheless, strength properties of the composite remain low if no treatment is performed to enhance the adhesion (Van-De *et al*., 2003). The ER/3PF has some mechanical properties lower than those of ER/1.5PF. It is justified by tensile tests realized on the fibres alone because these are degraded at a concentration equal to 3 N, but ER/3PF remains always better that ER/PF.

CONCLUSION

The fibres extracted from the leaves of the doum palm were underwent an alkali and an enzymatic treatment. The morphological study was carried out starting from the images obtained by SEM witch show the effect of the type of the treatment on the fibre diameter and surface quality. The biological treatment eliminates the residual impurities, gives smooth and circular fibres. The alkaline treatment yields less clean, more porous and oval surfaces. The tensile tests on doum palm fibres reveal that fibre mechanical properties reach their optima for a concentration of 1.5 N beyond of which they degrade gradually till their minima. The biological treatment of the leafstalks fibres does not improve the mechanical properties, but it gives the best specific tensile strength ratio. A better resistant fibre with low densities is provided. This is an attractive factor for the manufacturing of light weight materials. The present study showed the usefulness of doum palm fibre as a good reinforcing agent for composite fabrication. Flexural properties depend on the concentration of aqueous solution. It is seen that the alkali treated composites showed superior flexural properties than untreated composites. The ER/1.5PF has the best flexural strength and flexural modulus.

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