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Recent Advancement via Experimental Investigation of the Mechanical Characteristics of Sisal and Juncus Fibre-Reinforced Bio-Composites

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In this work, the mechanical characteristics of unidirectional bio-composite materials reinforced by two types of natural fibres (sisal and juncus) were studied in order to develop new materials. The effect of the fibres' extraction methods and their new assembly techniques on the mechanical properties of the elaborated composites was investigated. This is based on three methods of extracting natural fibres: the first uses water treatment alone over a long period, while the second uses alkaline chemical treatment with a sodium hydroxide solution. The last method uses the burial of plant leaves in moist soil. The obtained fibres are assembled according to techniques, such as monolinear fibres, twisting fibres into rope and braiding fibres into rope. The composite materials are produced manually using a pressure-contact moulding process. The outcomes demonstrated that the resulting compounds' mechanical properties are significantly impacted by the chemical treatment. The sisal/polyester composites exhibit better mechanical tensile test behaviour than those made with juncus fibres. Moreover, contrary to the results of some other studies, the recently developed techniques of assembling with a chemical treatment process enabled the reduction of the bio-composite's thickness as well as the cost of its preparation.

Keywords: natural fibre, sisal, juncus, mechanical properties, bio-composite

Highlights

- *Three procedures are used to extract natural fibres: the first involves prolonged water treatment alone; the second involves alkaline chemical treatment using sodium hydroxide solution; the final technique uses burying plant leaves in moist soil.*
- *The sisal/polyester composites exhibit better mechanical tensile test behaviour than those made with juncus fibres.*
- *Young's modulus of the composite reinforced with sisal fibres is twice as high as that reinforced with juncus fibres.*
- *The twisted rope assembly technique exhibits the highest values of Young's modulus compared to the other forms of assembly.*
- *The recently developed technique of assembling fibres into a rope with a chemical treatment process has contributed to the best reinforcement of the bio-composite materials.*

0 INTRODUCTION

The increasing interest in plant fibres is evident due to the proliferation of documents dealing with the use of these natural or modified fibres in composite materials. These plant fibres are already occupying an important place in the composites industry thanks to their best mechanical and physicochemical properties [1]. They are used in various fields of application, such as transport, construction, medical and leisure [2] and [3]. Among the main advantages of these natural fibres are their availability in several countries, regeneration, bio-degradability, and the possibility of extraction by various methods without damaging the fibres. They can also play a remarkable role in the development of new bio-degradable green materials with desirable characteristics. This can be used to solve some of the current ecological and environmental problems.

Natural fibres have become a viable, eco-friendly, and plentiful alternative to expensive, non-renewable synthetic fibres [1]. They present a promising reinforcement for composites for some applications due to their low cost, low density, and relatively good mechanical properties [4] and [5]. In addition, they are also characterized by the absence of health risks, easy handling, high flexibility, and sound insulation [6]. Natural fibres such as sisal, linen, kenaf, Alfa and jute have been used as reinforcement in bio-composites [7] to [9].

Our interest in this work is oriented towards the search for plant fibres that are generally the most abundant during the year. Furthermore, research is moving towards the development of bio-composites with the best possible mechanical properties at a lower cost.

Generally, the mechanical properties of bio-composites are often influenced by several factors, and they heavily depend on the fibre content and, therefore, on the nature and quality of the implementation [10]. These factors can be grouped into two types based on their origin: constitutional and structural. Among the elements of the constitutional

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type is the nature of the plant (kind of fibre), the nature of the resin, the physicochemical characteristics of these components (fibre/resin), the geometry of the fibre cells and their porosities, etc. The main elements of the structural type are the fibre-resin structure, the fibres' orientation, the size of the fibres, the mass fraction of the fibres, the fibre/matrix interface quality, the extraction methods and the manufacturing process of the composite (contact moulding, vacuum pressure moulding, etc.) [10].

Improving the properties of a bio-composite or its performance comes down to improving the various elements mentioned above. Therefore, among the methods of extracting plant fibres, the method of chemical treatment with sodium hydroxide (NaOH), which, according to the literature [11] to [13], facilitates the separation of fibres from the leaf matrix of their plant, by reducing impurities such as pectin, wax, and lignin around the outer surface of the fibres. It also improves the bond between the fibres/matrix of the composite to give the best mechanical results [14].

Belaadi et al. [15] indicated that the mechanical properties of polymer composites reinforced with sisal fibres are largely influenced by the mechanical properties of these fibres. Through their studies, they analysed the mechanical behaviour of sisal fibres and compared them with other bio-composites made of jute and juncus fibres; they observed the existence of the same influence of the fibres on the overall behaviour of the composites.

Joseph et al. [14] analysed the effect of water absorption on the tensile strength of sisal/polypropylene (PP) composites at different volume fractions. They found that the maximum tensile stress decreased as a function of the immersion duration. In addition, they found that sisal/PP composites with treated fibres exhibit higher strength than those of composites without treated fibres. Also, the mechanical properties of the polymer laminates reinforced with sisal fibres were studied, and they showed that the fracture stress increased with the immersion time in water during the extraction phase of these fibres [16]. In addition, the mechanical properties of the composites (epoxy type) reinforced with sisal fibres were examined. The mechanical decortication method was used to extract these fibres, which were subjected to chemical treatment with alkalis and binding agents. These pre-treated fibres showed improved mechanical and hydrophilic tendencies compared to untreated fibres. Thus, they resulted in efficient bonding at the fibre/polymer matrix interfaces [17]. The researchers also showed that the mechanical properties of the developed composites depend on various parameters, such as fibre length, fibre orientation, and fibre volume fraction [17].

Uppal et al. [18] observed that composites containing short shredded sisal fibres have high mechanical properties compared to those formed from sisal fabric. The ageing of these fibres also changes the mechanical properties of the bio-composites compared to those having newly chopped sisal fibres.

Maurya et al. [19] studied the mechanical properties of the epoxy composite reinforced with sisal fibres by varying the length of the fibre and keeping a constant weight percentage of the fibres. It was concluded that the tensile strength of the composite was not improved by reinforcing the length of the sisal fibres.

In this work, the influence of three extraction methods (with various physical aspects) of sisal and juncus fibres, as well as the new techniques for assembling these fibres, on the mechanical tensile properties of composites will be studied in order to deduce the best-elaborated bio-composite and propose it to the industry. Although they can be found on other continents as well, the sisal and juncus fibres used in this study were taken from North African plants.

1 EXPERIMENTAL

1.1 Choice of Plants and Natural Fibre Extraction Methods

The natural fibres studied were extracted from sisal and juncus plants, as illustrated by Fig.1. The choice of plants is dictated by the availability in our region of North Africa and by the rapid renewability factor during the year. There are several methods for extracting plant fibres [20], mainly according to mechanical, chemical, and biological processes.

The first fibre extraction method is mechanical, under water alone (water treatment method T_{Wt}). It consists of cutting the juncus stalks longitudinally into two slices lengthwise and in the same way for the sisal leaves; then, these sisal leaves and the cut juncus stalks are immersed in a large container of water. They will be kept for 4 days to 5 days at room temperature until fully saturated with water. The recovered sheets are then washed and dried, then mechanically tapped and manually brushed to extract the fibres. The obtained fibres from plants are shown in Fig. 2 (bundles no. 1 and 4).

The second method is a chemical method (the alkaline treatment method, T_{Alk}). After immersing the stems or the sliced leaves of the plants in water for 24 hours, a solution of sodium hydroxide (NaOH) at a 7 % concentration is added for 48 hours at ambient temperature. Next, they are well rinsed, and the traces of NaOH are neutralized in these fibres with a 2 % distilled water solution of sulphuric acid (H_2SO_4) immersed for 30 minutes [14]. Afterwards, they are immersed in distilled water for one hour to have a neutral potential of hydrogen (PH). The leaves obtained are placed on soft ground, and with a wooden stick, these leaves and stems are struck and shaken until the fibres are separated. Finally, they are dried in a room at room temperature for 24 hours, Fig. 2, (bundles no. 2 and 3).

The third method is biological under wet ground (T_{Gr}). It consists of burying the leaves and stems of sisal or juncus in the wet ground for 55 days to 57 days [21]. These sheets must be cut longitudinally beforehand into two or three parts; this promotes the biodegradation of these leaves, thus making the extraction of the fibres easier. Then, the obtained fibres are washed with water and then dried at room temperature, Fig. 2 (bundle no. 5).

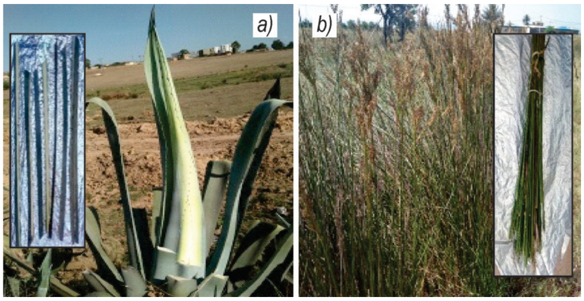


Fig. 1. *Studied plants in their natural habitat; a) sisal and b) juncus*

Fig. 2. *Some natural fibres obtained by different methods of extraction; no. 1, 2, 5 sisal's fibres, and no. 3 and 4 juncus' fibres*

1.2 Forming the New Assembly of Plant Fibres

The assembly of both studied fibres in the structure of composite materials is manually placed into two new rope shapes, with the addition of the usual form of linear assembly. The first one forms ropes by braiding or torsion, the second one forms ropes by twisting, and finally, it forms unidirectional straight fibres without overlapping, so we named this last assembly "mono-linear". The strings contain six fibres each.

1.2.1 Assembly in Ropes by Braiding

A braid is an assembly of bundles of fibres with a total of six threads. The different wicks of fibres pass alternately between them (Fig. 3a), such that the left strand is passed over the neighbouring strand and below the following one. It proceeds in this way until the last strand. The crossing of the wicks is done at a right angle and offers an oblique checkerboard pattern (Fig. 3a).

1.2.2 Assembly in Ropes by Torsion

In this mode of assembly, the six fibre threads are grouped in parallel and twisted in pairs (2×2×2 threads), then twisted together so as to form the rope by torsion (Fig. 3b). In this second case, the strands of twisted fibres are placed in a spiral whose centre is that of the rope.

1.2.3 Straight Unidirectional Assembly of the Fibres (Mono Linear)

In this last and usual mode of assembly, the fibres are grouped in parallel, quasi-straight and unidirectional lines without overlapping (Fig. 3c).

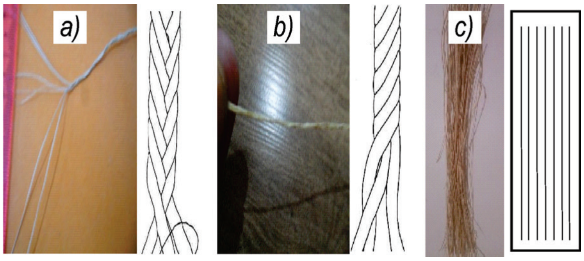


Fig. 3. *Assembly of fibres into a) ropes by braiding, b) ropes by twisting, and c) monolinear*

1.3 Fabrication of Laminated Composite Plates and Single Resin Plates

In order to fabricate our plates of bio-composite materials, we first prepared the plant fibres and then assembled them. Secondly, a polyester-type resin based on three components was also prepared. This type of polyester was chosen because it is the most used and the cheapest. The polyester matrix is therefore obtained by mixing a primary resin with a hardener (7 %) and an accelerator (3 %) by volume. Polyester-only (resin) tensile specimens are obtained from manually prepared plates. These specimens are used as a reference for the bio-composite specimens.

Unidirectional sisal/polyester and juncus/ polyester laminated plates (bio-composite) are made in mono-layers, such that the mass of fibres in a plate is approximately 30±0.5 g. The ends of the fibres are attached by double-sided adhesive tape to the edges of the mould during their deposits. This is done to ensure that the directions of the fibres are parallel and straight when casting the resin manually.

The composite plates were fabricated using the contact moulding method. The thicknesses of the plates are checked using metal wedges (2±0.1mm). The bio-composites are impregnated at room temperature (25±1 °C). The polyester resin obtained is catalysed and hardened in proportions of between 2 % and 3 % by mass of the primary resin.

Once the plates are reticulated, they all undergo a 24-hour polymerization cycle at room temperature before demoulding. In order to have a total polymerization of the polyester resin, the laminated plates are left in the open air for 3 days before being cold cut into test specimens. The bio-composite plates produced in dimensions of 180 mm×170 mm are cut into specimens according to the ASTM 3039 standards, as cited by Hassan and Abdullah **[22]**. In order to estimate the fibres' mass rate, each plate was weighed with a precision electronic balance (0.01 g). The tensile specimens (Fig. 4a) are in accordance with the ASTM 3039 standard **[23]** and have the following dimensions: Total length L=175 mm, thickness h=2 mm and width b=25 mm.

Fig. 4b shows some tensile specimens of bio-composites with wedges in their ends.

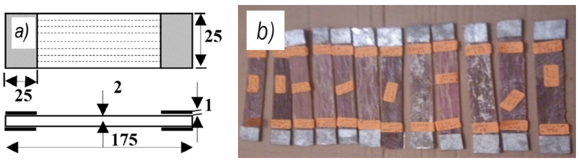


Fig. 4. *a) Schematic specimen with its dimensions; and b) some tensile specimens of bio-composite with wedges in their ends*

1.4 Mechanical and Structural Characterization of Bio-composites

To characterize the laminated plates produced, the mechanical properties and microstructure of all bio-composites elaborate are studied. The determination of the main mechanical properties is carried out via monotonic tensile tests under a universal machine of the Zwick-GmbH type (Fig. 5a). The tests are carried out with a pre-load of 1N until failure. To ensure good reproducibility of the results, at least three specimens for each category were tested at the same speed, which was quasi-static on the order of 1 mm/min. The direction of the tensile forces is the same as the orientation of the fibres. The length of the metal standards (wedge) placed at the end of the test specimens is equal to 25 mm. The latter serves to prevent crushing under the jaws of the universal testing machine (UTM). The longitudinal direction of the specimens is chosen to be the same direction as that of the fibres. Fig. 5b shows a bio-composite specimen under axial tensile loading with a zoom lens showing crack initiation.

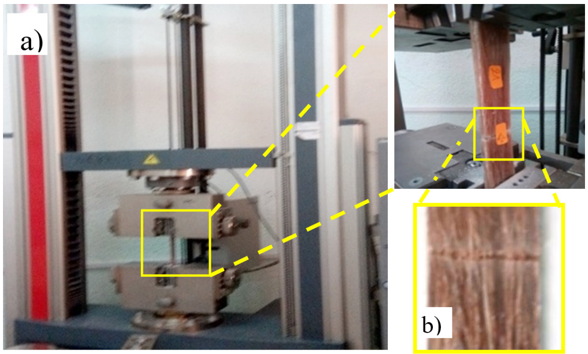


Fig. 5. *a) Tensile testing machine with zoom under the test, and b) zoom after tensile test*

Observation analyses of sisal and juncus fibres without resin and others into resin were carried out using optical microscopy (OM) and scanning electron microscopy (SEM).

2 RESULTS AND DISCUSSIONS

2.1 Mechanical Properties of Polyester Resin

Fig. 6 shows the curves of tensile testing of specimens in polyester-alone resin, and an example of a broken tensile specimen is illustrated. Each time, three specimens are tested. It is noted that the shapes of these curves are very close to each other except for a slight shift in elongation during the rupture phase. The overall mechanical behaviour of the resin specimens shows a bilinear stress-strain relationship, followed by sudden rupture. The elastic limit is approximately 5±0.5 MPa (Fig. 6). Young's modulus, the tensile strength, and the deformation of each specimen are grouped together in Table 1, associated with mean values.

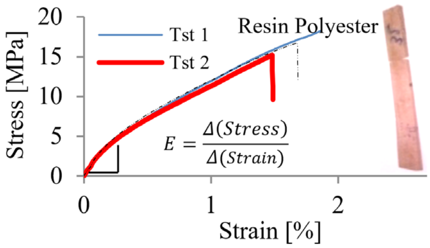


Fig. 6. *Curves of tensile tests of polyester specimens alone and an example of the specimen after the test*

Table 1. *Summary of the mechanical properties of the polyester resin specimens*

Speci-mens	Young's modulus E [GPa]	Plastic zone modulus $E2$ [GPa]	Maximum tensile strength Rm [MPa]	Rate of deformation [%]
Tst1	1.701	0.871	18.25	1.80
Tst2	1.533	0.805	15.12	1.52
Tst3	1.811	0.805	16.41	1.67
Average values	1.681 ±0.148	0.827 ±0.044	17.33 ±2.21	1.66 ±0.16

Table 1 shows the average maximum stress reaching a value of 17.33 MPa before the specimen breaks. This value is comparable to those given in references [24] and [25]. Young's modulus (elastic zone) has an average value of 1.681 GPa, and that of the plastic zone is around 0.827 GPa, with an error of ±0.15 GPa. However, the rate of deformation is of the order of 1.66±0.16 %. These results will serve as a reference for the composites to be developed.

2.2 Young's Modulus of Developed Bio-composites

The results of Young's modulus of the specimens of the different cases developed with the sisal fibres (Fig. 7a) and the juncus fibres (Fig. 7b) are presented in histograms. The different cases studied concern the three methods of extraction (chemical treatment T_{Alk}, water treatment T_{Wt}, and stripping in the wet ground T_{Gr}) and the three forms of fibre assembly (assembly in cords by twisting "#1", in cords by braiding "#2", and fibres in straight lines noted mono-linear "#3").

Overall, it can be seen that Young's modulus E of the composites is higher than that of the pure resin, and its value has been amplified several times (from 2 to 7 times that of the resin, depending on the nature of the fibres; Fig. 7) by the addition of fibres. From this Fig. 7, the effect of the extraction method on Young's modulus of the composites can therefore be classified according to their values as elasticity modulus E according to the extraction method, is classified by Eq. (1):

$$E_{Res\,Ref} < E_{Twt} < E_{TGr} < E_{TAlk}. \tag{1}$$

This result is valid for both cases of plants regardless of the type of assembly form. From Fig. 7a, Young's modulus of composites reinforced by sisal fibres and obtained by chemical treatment ($S\!-\!1\_T_{Alk}$) is compared to the modulus of the same composite obtained with water treatment ($S\!-\!1\_T_{Wt}$); it shows an increase of approximately 70.01 %. However, the comparison of Young's modulus of ($S\!-\!1\_T_{Gr}$) with that of ($S\!-\!1\_T_{Wt}$) gave an increase in value of about 13.60 %. The same observation of an increase in modulus is observed for the other cases of $S\!-\!2$ and $S\!-\!3$, but with low percentage values. It is concluded that the extraction method by treatment with NaOH has increased Young's modulus value of the composite compared to the other extraction methods. This observation is in accordance with some of the literature [26] and [27].

Young's modulus of composites reinforced with juncus fibres (Fig. 7b) evolves in the same way as that of sisal, according to the different extraction methods, but with a difference in value. This reveals the importance and interest in using chemical treatment of fibres. However, there is no case for under wet ground (T_{Gr}) extraction for this juncus plant.

Note: this last method of extraction by the under wet ground technique on juncus fibres was not successful during the tests, so its effect was not studied. This is due to the very fast degradation and deterioration of these fibres during the execution of this method because of the low amount of pectin in this plant.

It can be summarized that for the same nature of fibre and for a given extraction method, Young's modulus E of the composite evolves according to the three forms of assembly as indicated in Eq. (2). It is noticed that the case of twisting assembly (#1) presents the highest values of the modulus compared to the other forms of assembly (#2, #3). They can therefore be classified by Eq.(2):

$$E^{\#3} < E^{\#2} < E^{\#1}. \tag{2}$$

It is observed that Young's modulus of the sisal-reinforced composite is twice as high as that of the juncus-reinforced composite for the same characteristics. Therefore, these results have helped to highlight these new fibre assembly techniques in the field of bio-composites. The main mechanical properties of the elaborated biocomposite materials (sisal/polyester and juncus/polyester) are summarized in Table 2 (S = sisal; J = juncus). Thus, it can be deduced from the values of the ultimate tensile strength that the latter has almost the same evolutionary trend as that of Young's modulus in the different cases (Eq. (1)). Also, it was shown that the mechanical properties of the composites are improved compared to the resin-only case several times. In fact, the *Rm* of the composites increases by 4 to 5 times with the use of sisal fibres and by 2 times to 3 times with juncus fibres.

2.3 Influence of the Fibre Assembly Form on Mechanical Properties of Bio-composites

2.3.1 Case of Rope Assembly by Twisting #1

Fig. 8 shows the evolution of the tensile stress as a function of strain for different composites according to the three extraction methods (T_{Alk}, T_{Wt} and T_{Gr}), for which a pure resin curve is introduced as a reference (Res−Ref). The fibres of composites (sisal and juncus) are assembled in rope by twisting.

The figure illustrates a large shift between the curves of reinforced composites with sisal, juncus and

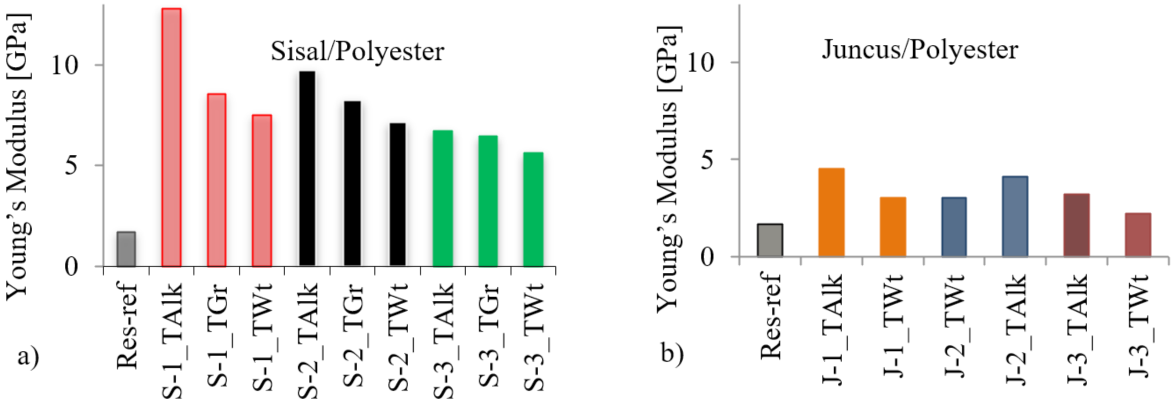


Fig. 7. *Young's modulus evolution of composites; a) sisal/polyester, and b) juncus/polyester*

Table 2. *Tensile mechanical proprieties of the composites produced (sisal and juncus/polyester)*

Fibres/ matrix	Composite types	Symbol	Young's Modulus E [GPa]	Modulus of plastic zone $E2$ [GPa]	Ultimate tensile strength Rm [MPa]	Strain ε [%]
Polyester	Resin-Ref N°1	Res-ref	1.701	0.871	18.25	1.80
Sisal (S)	S- in rope by twisting with NaOH treatment (Alkaline)	$S-1\_T_{Alk}$	12.77	4.693	95.24	2.13
	S- in rope by braiding with NaOH treatment (Alkaline)	$S-2\_T_{Alk}$	9.724	4.057	88.02	2.30
	S- monolinear with NaOH treatment	$S-3\_T_{Alk}$	6.704	3.176	74.80	2.88
	S- twisting rope with an underground processing T_{Gr}	$S-1\_T_{Gr}$	8.524	3.728	82.30	2.35
	S- braiding rope with T_{Gr}	$S-2\_T_{Gr}$	8.229	3.828	74.76	2.50
	S- monolinear with T_{Gr}	$S-3\_T_{Gr}$	6.436	2.843	74.80	3.04
	S- twisting rope with water treatment T_{Wt}	$S-1\_T_{Wt}$	7.501	3.930	73.89	2.59
	S- braiding rope withT_{Wt}	$S-2\_T_{Wt}$	7.146	3.396	72.51	2.91
	S- monolinear with T_{Wt}	$S-3\_T_{Wt}$	5.635	2.810	72.17	3.21
Juncus (J)	J- twisting rope with NaOH treatment	$J-1\_T_{Alk}$	4.529	2.346	55.40	2.78
	J- braiding rope with NaOH treatment	$J-2\_T_{Alk}$	4.103	2.074	53.49	3.14
	J- monolinear with NaOH treatment	$J-3\_T_{Alk}$	3.187	1.335	43.27	3.92
	J- twisting rope with T_{Wt}	$J-1\_T_{Wt}$	3.045	1.659	47.70	3.23
	J- braiding rope with T_{Wt}	$J-2\_T_{Wt}$	3.041	1.455	46.61	3.46
	J- monolinear with T_{Wt}	$J-3\_T_{Wt}$	2.230	1.193	45.29	4.60

pure resin. The spacing between curves (the slopes and the maximum values (peak)) is clearly different. This is due to the intrinsic nature of each substance, which is quite different. The shape of these curves represents two quasi-linear phases followed by an abrupt rupture (brittleness). It is a very short elastic zone followed by a wider one: the plastic zone (from approximately 0.2 % of the deformation). Thus, it is bilinear behaviour for the three materials (Fig. 8).

This behaviour is due to the natural response of the fibre reinforcements and due to the specificity of the fibre/resin interface of each composite.

It can be seen from the various curves that the maximum values (peaks) of the breaking stress are between 95.24 MPa of ($S-1\_T_{Alk}$) and 45.29 MPa of ($J-3\_T_{Wt}$) and are always followed after these maximums by a sudden break. This is obtained with an elongation between 2.13 % and 4.60 % (Table 2).

It is concluded that the highest *Rm* of the maximum stress at failure corresponds to the composite reinforced with sisal fibres and elaborated by the NaOH treatment.

While the maximum stress at break corresponding to the juncus-reinforced composite (Fig. 8) is about 55.4 MPa ($J-1\_T_{Alk}$) with an elongation of 2.78 %, this plant gives a composite that is lower in maximum stress but has more elongation and is, therefore, more ductile than sisal. This is also due to its weak nature (low cell pectin density), resulting from the fact that the diameters of their fibres are narrower than those of sisal.

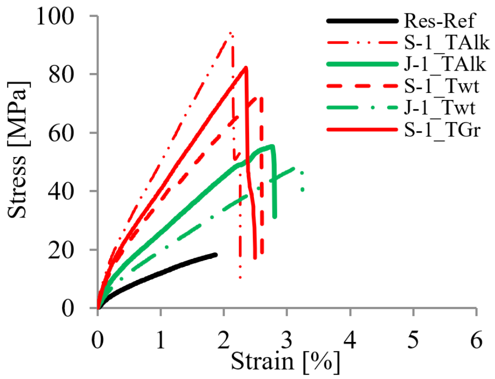


Fig. 8. *Tensile curves of resin and laminates assembled in rope by twisting (according to extraction methods)*

2.3.2 Case of Rope Assembly by Braiding #2

Fig. 9 shows the evolution of the tensile stress as a function of the deformation of the composites, where the fibres (sisal and juncus) are assembled in rope by braiding according to the three extraction methods.

It is found that the mechanical properties of bio-composite materials reinforced with sisal fibres are better than those reinforced with juncus fibres, with an increase in *Rm* of about 64 % and 55 % compared to juncus fibres (Fig. 9). This leads us to recommend the use of sisal fibres in bio-composites over juncus fibres.

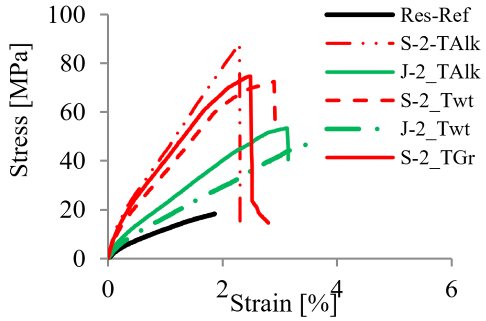


Fig. 9. *Tensile curves of resin and laminates assembled in rope by braiding (according to extraction methods)*

These results are similar to the previous case (fibres assembled in rope by twisting) and have a maximum increase of $Rm \approx 72$ % (Fig. 8). It can also be deduced that the twisting assembly is better than the braiding assembly.

2.3.3 The Case of Mono-Linear Assembly of the Fibres #3

Fig.10 shows the evolution of the stress to failure as a function of the deformation of the same laminates as above, but the fibres are assembled in a mono-linear fashion. As before, a large gap between the three curves (sisal, juncus, and pure resin) can be observed. This is due to the same reasons as before (rope assembly by twisting and braiding). It can be seen that there are also two quasi-linear phases in their overall behaviour. It can be seen that the maximum value of the stresses at failure (≈70 MPa) is lowered by about ≈25 % compared to the first case (by twisting), while their deformations were enlarged in the majority for this assembling case. This can be justified by the installation of the fibres in the resin in an evenly spaced manner without overlapping. It reduces the maximum stress by their dispersion but facilitates the elongation of the bio-composite. It can be seen that sisal has always kept the best mechanical behaviour compared to juncus. The latter keeps the large elongations.

Finally, it can deduce that the composites having an assembly in rope by twisting have better mechanical behaviour than those obtained by the fibres assembled in a monolinear fashion for the same type of plant.

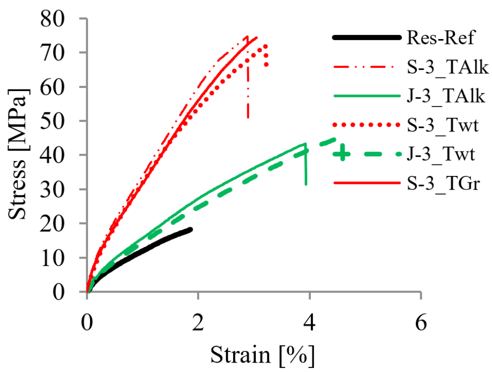


Fig. 10. *Tensile curves of resin and elaborated laminates by mono linear assembling (according to extraction methods)*

2.4 Influence of the Fibre Extraction Method on Mechanical Properties of Bio-composites

Fig. 11 shows the stress versus strain curves after the tensile test of the same composites for the three fibre extraction methods. The results in Fig.11a show the behaviour of the different composites obtained via the chemical treatment method (T_{Alk}). The shapes of the curves show a difference from one composite to another with a ranking discussed previously and prove that the behaviour of composites with sisal fibres is better than those with juncus fibres. Fig. 11b shows the effect of using the fibre extraction method with water treatment (T_{Wt}) on the tensile behaviour. For the same type of plant (same fibres), these curves are approaching each other, whatever the type of assembly (#1, #2 or #3).

Fig. 11c illustrates the effect of the under wet ground extraction method (T_{Gr}) on the behaviour of the composites according to the tensile curves. The first point that draws attention is the absence of composites with juncus fibres. It is also observed in Fig. 11c that the curves (of S1 and S2) belonging to this T_{Gr} method are very close to each other (a very small deviation) with respect to (S3) for the behaviour of this sisal fibre composite. However, they are all far from the Resin-pure reference curve. Nevertheless, it can be observed that the peak of the maximum stresses of these curves lies between the two previous cases (the maximums) of Figs. 11a and b.

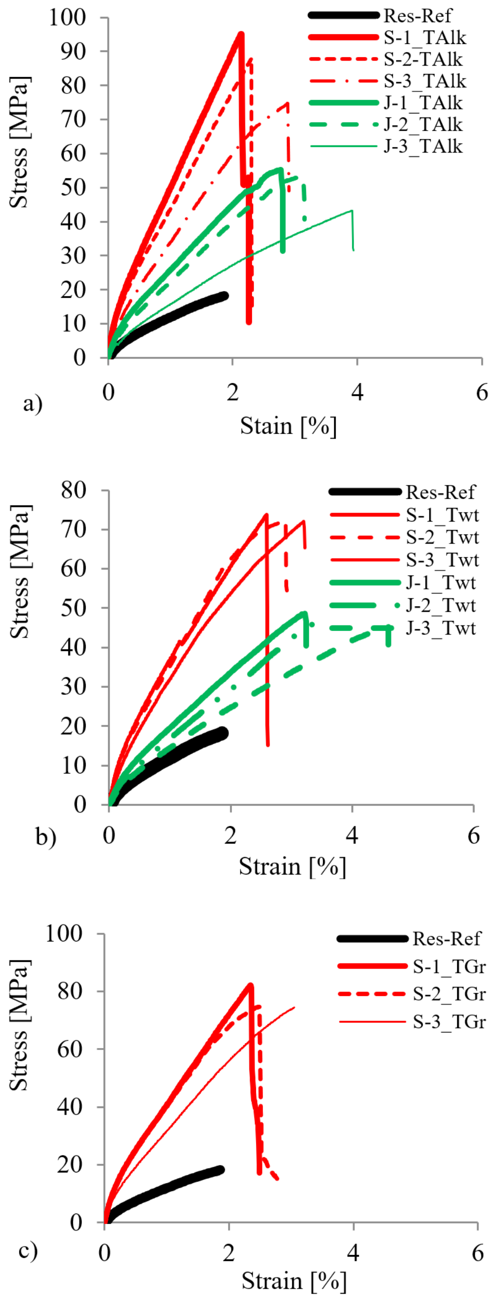


Fig. 11. *Tensile curves of the same composites showing the influence of extraction methods, a) T_{Alk}, b) T_{Wt} and c) T_{Gr}*

It is deduced that the T_{Alk} extraction method gives the best improvements on the mechanical properties of composites with both types of fibres (sisal and juncus). Since the NaOH treatment changes the topography of the fibre surface, it removes wax, pectin, and part of the lignin. Indeed, the removal of these components is necessary because their presence leads to lower tensile strength. It is concluded that the stress at break increases with the complexity of the extraction method while the strain at break decreases overall. This is attributed to the reduced mobility of hydrogen bonds between the matrix and fibres to roughen the fibre surface [28] and so improve the quality of the fibre/matrix interface.

2.5 Structural Characterization of the Elaborated Bio-composites

2.5.1 Observation of Different Fibres Used

Fig. 12 shows typical optic micrographs of sisal fibres obtained by the T_{Alk} and T_{Gr} extraction methods, respectively. The other measurements of the fibre diameters are summarized in intervals and in average values in Table 3, according to the nature of the plants and the extraction methods. The average diameters were determined from five samples (fibres) on the median part of their length. These measurements show the effect of the extraction method on the average value of the fibre diameter.

Table 3. *Diameter measurement values for the fibres of sisal and juncus*

Fibre type	Extraction method	T_{Alk}	T_{Wt}	T_{Gr}
Sisal fibres	Diameters [μm]	210-330	270-360	340-560
	Average value [μm]	270	315	450

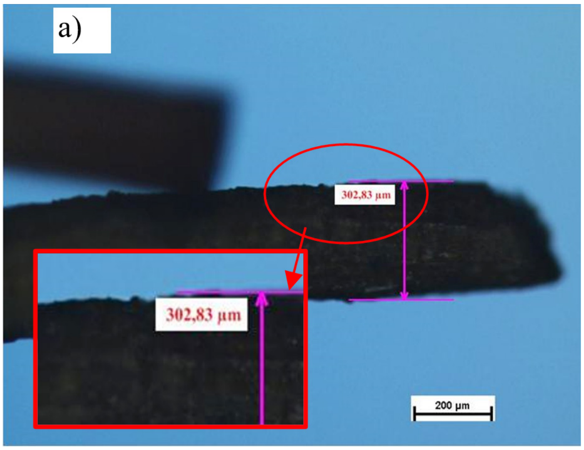
Fibre type	Extraction method	T_{Alk}	T_{Wt}
Juncus fibres	Diameters [μm]	110-220	170-260
	Average value [μm]	165	215

It is found that the first extraction method, T_{Alk} gives fibres with relatively small diameters compared to the other methods, such as 270 μm for sisal and 165 μm for juncus. This is probably due to the alkaline chemical treatment, which forces the fibres of the plant matrix to detach completely and results in fine fibres with smooth side surfaces. The second method T_{Wt} shows a remarkable increase in the measured fibre diameter values (Table 3).

The last method T_{Gr} reveals larger values of fibre diameters than before or sisal fibres. Thus, it is noted that with the last method, the increase ratio in average diameters compared to the first method is of the order of 66 %.

Other results can be summarised as follows.

On the surface of the fibres, traces of pectin and plant matrix cells were observed, despite a good rinsing. As a result, these traces of plant matrix participated in the increase of fibre diameters and thus reduced their number if used in the laminate for the same mass of fibres compared to other fibres obtained by other extraction methods and used in bio-composites. This resulted in weaker mechanical properties when testing the composites made by this method (T_{Gr}) and compared to the first treatment method (T_{Alk}).



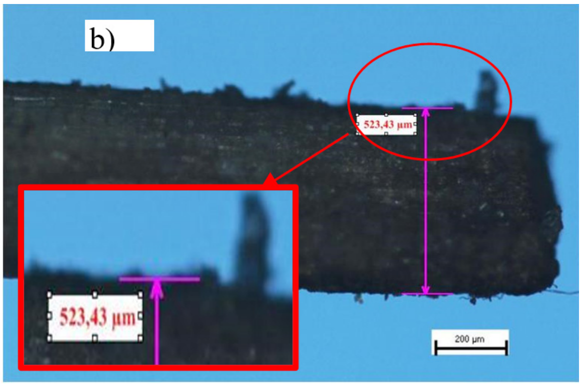


Fig. 12. *Typical optical micrographs of sisal fibres according to the above extraction methods, a)* T_{Alk}, *b)* T_{Gr}

Further measurements of the fibre diameter of any plant also show a small increase in the resin. Thus, the fibres in the resin were swollen (diameter increase of about 18 % to 20 %). For example, through some measurements of sisal fibres (T_{Gr}), the swelling ratio of this fibre is about 20.7 % (i.e., from 362 μm to 437 μm). This finding is also confirmed by Motaung et al. [9].

2.5.2 Structural Observation of the Developed and Tested Bio-composites

Longitudinal Observations of the Samples after Tensile Test

To characterize the interface between the fibres and the matrix after the tensile test and to see the fracture surfaces, the scanning electron microscope

(SEM) observation technique was used. Fig. 13 shows the fractured specimens of bio-composites reinforced with sisal fibres (front view), obtained by the T_{Alk} elaboration method and according to both fibre-assembling techniques (assembling in mono-linear technical and assembling in rope by twisting).

From the revealed images, it is globally observed that the failure that occurred in the samples was a combination of failure along the fibre-matrix interface and failure of some fibres, as well as matrix fracture. However, composite matrix failure after tensile failure generally exhibits brittle fracture. Fig. 13a shows a global view and its zoom of a rupture of the specimen obtained by the T_{Alk} extraction method and the fibres assembled with the mono-linear technique. It can be seen that there are some extended sisal fibres and some broken ones. The matrix has, in addition to its transverse fracture cracks, fragments attached to the fibres, showing the cohesion of the fibres with the matrix and the quality of the fibre-matrix interface.

Fig. 13b shows a wide view and zoom of a specimen fracture obtained by the same extraction technique, but the fibres are assembled in rope. The same observations as before can be made in this case, but the fibres are additionally bent in flexion, and the matrix fragments are more numerous than before. The mechanism of rupture of the matrix is also shown by even more numerous longitudinal cracks. This demonstrates a strong attachment at the fibre-matrix interface. This is also affirmed by its tensile results, showing a clear improvement and thus giving the best case of the tensile tests.

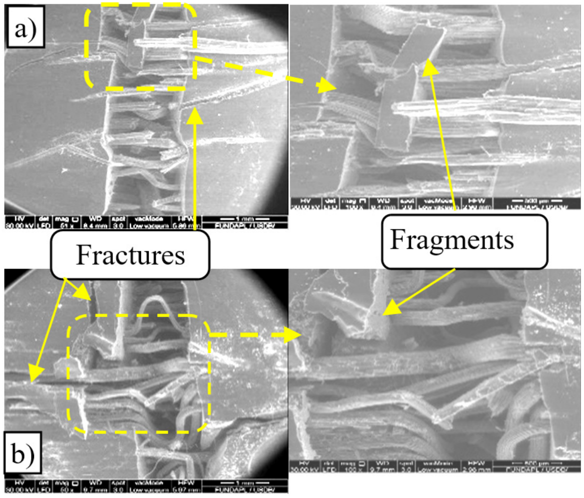


Fig. 13. *Global view and its zooms of a sisal specimen rupture obtained by the T_{Alk}; a) fibres assembled in mono linear technical and b) fibres assembled in ropes*

Fig. 14 shows laminated rupture specimens reinforced with the juncus fibres obtained via the T_{Wt} extraction method and assembled in mono-linear technical (T_{Wt} / Assemb-linear). It illustrates a global view and a magnified view of a part of the rupture. It is always observed after the tensile test that the polyester matrix fracture shows a brittle transverse break. It can be observed that the fibres underwent slippage, cracks and longitudinal with transverse breaks, showing a weak cohesion of the fibres with the matrix.

This resulted in a very low-quality fibre-matrix interface. Also, it is proved by the tensile tests, which give the lowest case for this juncus plant. It can be concluded that the T_{Wt} method does not give the best case of fibre-matrix junction in the composite. The same finding was obtained for the sisal plant with this method T_{Wt}.

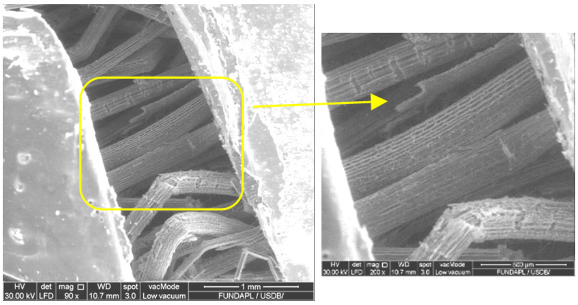


Fig. 14. *Global view and zoom of rupture specimen reinforced with the juncus fibres using the TWt /Assemb-linear production methods*

Cross-sectional Observations of the Specimens

Fig. 15 shows the transverse fracture surfaces of the bio-composite specimens reinforced with sisal. The portion of the specimen selected for observation corresponds to the best case of tensile test and complete rupture. This figure shows the shear surface morphology of the matrix and the sisal fibres state for two zones (Figs. 15a and b) in a selected specimen. It is observed that in Fig. 15a, the matrix surface is almost smooth, and there are fibre groups indicating a rope assembly. Fig. 15b shows some fibres having the burst tear with some micro-fibres and others in transverse rupture, as well as gaps on the matrix surface, which correspond to the location of other detached fibres. Therefore, the difference between Figs. 15a and b is that Fig. 15b illustrates more detail of the cross-sectional morphology of specimen rupture. Fig. 15c shows a magnified view of cut fibre that has a condensed capillary structure with a nearly flattened cross-section.

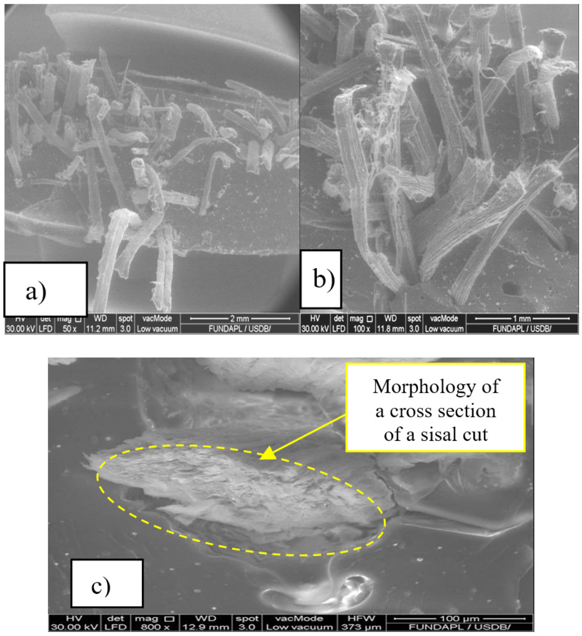


Fig. 15. *a) and b) global view of shear surface morphology of the matrix and the sisal fibres for two zones, and c) zoom of cut fibres*

3 CONCLUSIONS

This work aims to develop and elaborate the bio-composites with sisal and juncus fibres, analyse the effect of the elaboration of these materials on their mechanical properties and thus recommend the best case. The fibres are obtained via three extraction methods and assembled in three types of assembly, two of which are new techniques to form ropes in the matrix of these composites. The elaborate matrix is made of unsaturated polyester.

The first method of extraction is based on the effect of water alone for a long time (T_{Wt}), while the second method is based on an alkaline chemical treatment by a NaOH solution of 7 % concentration (T_{Alk}). The last method consists in exploiting the effect of the humidity under the ground (T_{Gr}). The selected fibres are assembled in three types, such as the first one in mono-linear fibres without overlaps, then groups of fibres in rope by twisting and in rope by braiding.

The tensile tests reflect a bilinear behaviour with a brittle fracture in the majority of the elaborated bio-composites. The classification according to the average mechanical characteristics of the different combinations in decreasing order is as follows: the case of assembly by twisted rope, then braided rope, and the last case in mono linear fibres. The classification by the extraction method is the Alkaline treatment with NaOH (T_{Alk}), treatment under wet ground (T_{Gr}), and treatment with water alone (T_{Wt}).

The mechanical properties of these bio-composites depend on the type of fibre, its diameter, the way of assembly, and the method of extraction of these fibres. The improvements resulting from the treatment with NaOH solution have changed the topography of the lateral surface of the fibres, eliminating the wax, pectin, hemicellulose and part of the lignin. The composites with sisal fibres show the best mechanical behaviour in tensile than those elaborated with juncus fibres. The difference is more than 200 %. The case of twisted rope assembly presents the highest values of Young's modulus compared to the other forms of assembly. The Young's modulus of the composite reinforced with sisal is twice as high as that reinforced with juncus for the same characteristics.

Optical microscopy analysis allowed the measurement of the diameter of the sisal and juncus fibres. The fibres in the resin underwent a swelling of about 18 % to 20 %. These measurements show the effect of the extraction method on the average value of the diameter of a fibre. The best fibres are those with more cleanliness without residual impurities and smaller diameters. Structural analysis of the bio-composites by SEM showed that those obtained by the NaOH solution extraction method (T_{Alk}) present the best cohesion of the fibres with the matrix. The post-tensile morphology of the specimen failures shows that the failure was a combination of failure along the fibre-matrix interface and fracture of some fibres as well as a brittle transverse failure of the polyester matrix.

Finally, the best bio-composite material developed and recommended is the one obtained by combining the extraction method based on NaOH treatment (7 %) with the sisal fibres assembled in rope by twisting. This combination contributed to reducing the thickness of bio-composites compared to the bibliography and improved their mechanical properties.

4 ACKNOWLEDGEMENTS

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5 NOMENCLATURE

T_{Alk} Treatment with NaOH (Alkaline),
T_{Wr} Treatment with water alone,

T_{Gr} Treatment in the wet ground,
#1, #2 Rope Assembly by twisting, by braiding,
#3 Mono Linear Assembly of the fibres,
E Young's modulus, [GPa].

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