

Full Length Research Paper

Extraction and characterization of *Retama monosperma* fibers

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The aims of this study were to determine the good conditions for fibers extraction from *Retama monosperma* leaves and their mechanical, physical and chemical characteristics. The fibers were extracted using a range of NaOH concentration from 1 to 16% in a period of treatment of 1 to 24 h, coupled with a physical treatment. For the evaluation of physico-mechanical characteristics, 200 samples were performed in the tensile test. The biochemical composition of the fibers was determined after separation of the parietal compounds. The results show that the best fiber yield was 11.51% obtained by a treatment of 14% NaOH for 8 h, followed by a physical treatment. The fibers biocomposition was 87.3% of cellulose, 7.5% of hemicelluloses and 1% of lignin. The Young's modulus was 13.3 GPa, tensile strength was 110 MPa and density was 1.3 g/cm³. The average fiber length was 155.7 mm. The fibers yield and characteristics showed that *R. monosperma* plant may in future be suitable source for natural fibers.

Key words: *Retama monosperma* young stems, fibers, extraction, characterization.

INTRODUCTION

The composites industry began since the 20th century, and in the same time, fibers industry saw an exponential growth. In that period, the most fibers used frequently in composite industry were synthetic fibers such as carbon, graphite, and glass fibers. The impact of this industry on the environment was very heavy. Today, natural fibers seem to be an effective solution for the production of fully biodegradable materials for replacing of some synthetic fibers (Belaadi et al., 2013; Mylsamy and Rajendran, 2010). Among the natural fibers, vegetable fibers have many advantages; availability, recyclability, low-cost, eco-friendly, no toxicity, biodegradability, mechanical

performance and easy extractability (Bledzki and Gassan, 1999; Reddy and Young, 2005; Béakou et al., 2008). Without cotton and wood fibers, the annual world production of vegetable fibers is 6200 kt (Rajendran, 2011). The Jute fibers are half of the global production, followed with coco 16 and flax 13% (FAO, 2010). At present, the most used plant in the extraction of fibers are sisal, hemp, flax and bamboo by using different plants parts such as: bast, leaf, seed, fruit, wood, stalk, and grass fibers (Mawaikambo, 2006). Currently, researchers do many studies on the characterization of new lignocellulosic fibers as Okra (De Rosa,

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2010; De Rosa, 2011), *Posidoniaoceanica* (Khiari et al., 2011), *Artichoke* (Fiore et al., 2011) and *Grewia tillifolia* (Jayaramudu et al., 2010). In Algeria, there are many plants which can be used in the extraction of fibers (Kaid-Harche, 1985; Kaid-Harche et al., 1990; Benahmed et al., 2006) but until now there are any studies on these plants. In this study we focused on *Retama monosperma*; this plant is natively from North Africa and some parts of southern Europe. In Algeria, this plant occupies a considerable area (Thoma, 1968). To the best of our knowledge, actually, the only use of this plant is for dune fixation and for fighting against desertification.

The aims of this study were the establishment of a fiber extraction protocol from *R. monosperma* leaves and characterization of extracted fibers. In general, for fibers extraction there are three different ways; chemical, mechanical and biological. In this study we used coupling techniques, chemical and physical. After extraction, the fibers were characterized with the different tests.

MATERIALS AND METHODS

Plant materials

In this study, we used a freshly harvested young leaves.

Fibers extraction

Physicochemical procedure

For the first time, an extraction protocol of *R. monosperma* fibers is established. The determination of the extraction parameters appears mandatory to define the appropriate protocol for the best fibers yield. The procedure used to obtain fibers is based on the principle of treatment combination, (chemical and physical) unfolding in four steps: pretreatment, chemical dissociation, physical dissociation and post-treatment.

Pretreatment

Pretreatment aims to eliminate the protoplasmic content. To this end three samples of 25 g were studied: i. the first sample (T1), was treated for 24 h with a mixture of chloroform / methanol (v/v); ii. the second sample (T2), was placed in an acetone bath for 10 min followed by another bath of isopropanol for 10 min as well. The sample was then transferred into an ethanol bath for 20 min at 90°C and last, it was washed with water; iii. The third sample (T), non-treated was used as a control.

Chemical dissociation

Alkaline dissociation was carried out by using sodium hydroxide (NaOH) at 14% for 24 h at 70°C. Then, the three samples were washed with water to neutralize them, and to finally separate the fibers.

Physical dissociation

It consists in proceeding to an autoclaving. Pressure and temperature are important factors in fibers dissociation. The three samples underwent the same treatment for 30 min at a pressure of

1.0 bar and a temperature of 121°C.

Post-treatment

After drying in ambient air, the fibers were separated by a manual carding. This step consists in removing the impurities and obtaining fine fibers.

Optimization of extraction conditions

Effect of NaOH concentration

Different concentrations of NaOH were tested (4, 6, 8, 10, 12, 14 and 16%) to determine the optimal concentration for the best dissociation of fibers. The treatment for each one has been 24 h.

Effect of processing time

The experience to determine the reaction time of the alkaline solution was carried out at different periods: 1, 2, 3, 4, 6, 12 and 24 h.

The effect of pressure

The experiment was conducted on two samples; one underwent a pressure of 2.2 bar at a temperature of 121°C, while the second (control) underwent no pressure.

Fiber characterization

Density

The fibers were dried at 100°C for 24 h, cut at the same length and put in pycnometer for density.

Fibers tenacity

The strength of fibers was determined with a Zwick tensile testing machine. The fibers were placed with clips between two rods separated with 2 cm. A tensile force was applied to the fiber breakage. The test was repeated 200 times.

Fineness

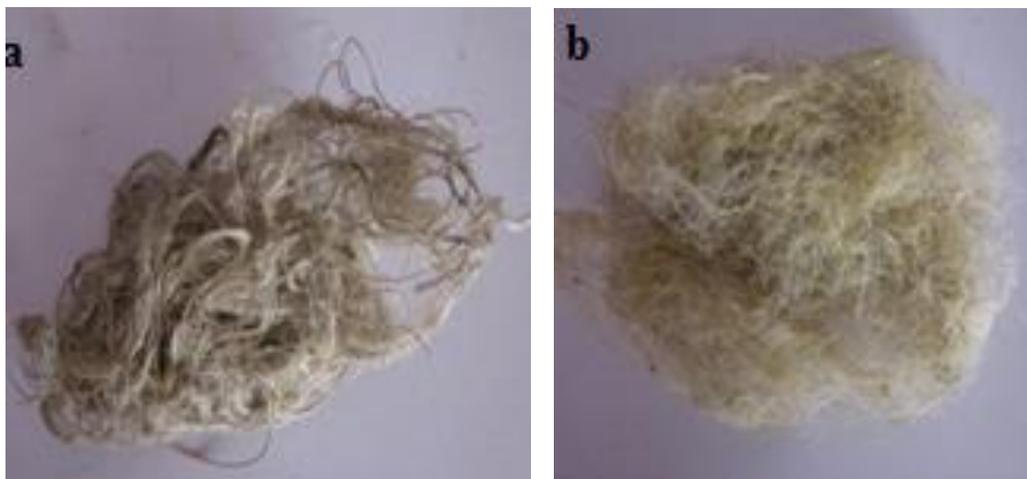
According to Fiore et al. (2011), fiber fineness was defined by the separation degree expressing the number of fiber bundles contained in 1 mg of raw material. The fibers were manually parallelized and cut to a length of 1 cm each. The fiber bundles were placed one by one on a balance with a clamp, until the weight of all fibers reached 1 mg. The number of fibers counted represents the separation degree.

Swelling test

Swelling of fibers due to water absorption was observed with a microscope provided with graduate objective. Three fibers removed from fiber bundles were placed in parallel direction on a glass slide. The fiber diameter was measured after 2 h distilled water immersion. Fibers diameter measurements were taken before and after immersion. The percentage in fiber diameter due to swelling was determined on 30 different fibers.

Table 1. The result of the extraction of fibers.

Sample	Weight (g)	Yield (%)
T	1.41	10.6
T ₁	1.27	9.6
T ₂	1.07	6.72

**Figure 1.** Fibers aspect. **A.** Before combing. **B.** After combing.

Floatability

Thirty single fibers were subjected to the following treatment: with a metal hook, one fiber was placed horizontally to the surface of deionized water till the fiber touched the water surface. The hook was removed gently, and the behavior of the fiber was observed (floating or sinking) for 5 min.

Chemical composition (Chaa et al., 2008)

A method based on the compounds selective dissolving was applied to determine the amount of each ligno-saccharide components (lignin, pectin, cellulose and hemicelluloses) presents in *R. monosperma* fibers.

Fibers biometrics

Fiber's length and width of 30 fibers and fibrils were determined under microscopes provided with graduated objective.

RESULTS AND DISCUSSION

Extraction of fibers

Chemical dissociation

In Table 1, the obtained results show that the first batch

of samples T (non-treated) presented a yield of 10.6% (w/w) which is higher than the batches T₁ (9.6%, w/w) and T₂ (6.72%, w/w). The batch T₂ gave the lowest yield of fibers. Treatment with acetone and isopropanol caused a considerable decrease in the yield which was estimated at 3.88% (w/w) compared with the untreated samples T (Table 1). T₁ also presented a low fiber yield compared to untreated samples. These results show that the preprocessing step for the removal of cellular contents leads to lower fiber yield. That proves that in the process of extracting fibers, sodium hydroxide was sufficient to eliminate the cell content and also to partially separate the fibers.

Physical dissociation

Autoclaving combines pressure and temperature. These two factors are important in the separation of fibers. Such treatment followed by a carding allows to separate fibers from impurities to obtain fine fibers ready to be used in the industry (Figure 1). Table 2 shows that most of the plants used in the production of fibers have yields which do not exceed 9% except for the case of banana leaves, while the *R. monosperma* gave a higher yield 10.6% (Table 1). This is explained by its high fiber content and their facility to extract.

Table 2. Extraction rate of fibers in several plant species (Arsene et al., 2007).

Fiber	Extraction (%)
Bagasse	4.00
Feuille de Bananier	9.84
Tronc de bananier	4.46
Coco	8.77
Tissu de coco	1.74
Eucalyptus	8.20
Sisal	1.33

Table 3. Yield fibers according to the concentration of NaOH.

NaOH (%)	Weight (g)	Output (%)
16	1.30	5.2
14	2.40	10.6
12	2.29	9.16
10	1.72	6.88
08	1.54	6.16
04	1.25	5.00
03	1.25	5.00
02	1.04	4.16
01	0.0	0.00

Optimization of the extraction of the fibers

Effect of the concentration of NaOH

The results obtained (Table 3) show that the yield of fiber was positively proportional to the concentration of sodium hydroxide. The best yield obtained was 10.60% with a concentration of 14% of Soda. Beyond this concentration, the yield decreases. It is also important to note that at a concentration less than 4% of sodium hydroxide, the separation of the fibers is achieved only if a mechanical pressure is applied, and at a concentration of 1%, the separation of fibers is impossible. These results show that the concentration of sodium hydroxide has an effect on the separation of fibers. This phenomenon is due to the swelling of the cellulose fiber after the relaxation of the natural crystalline structure of the cellulose. Fengel and Wegener (1983) have reported that the different alkali solution (KOH, Ca(OH)₂, NaOH) and its concentration have an effect on the degree of swelling and in the transformation into cellulose-II which affects the quality of the fibers. Also, author researchers reported that treatment with Ca(OH)₂ decreased the tenacity of fibers more than treatment with NaOH (Arsene et al., 2007). The treatment with sodium hydroxide changes the topography of the surface of the fibers, removing the components of the cuticle, the pectin, and partially the lignin and the hemicelluloses (Mwaikambo et al., 1999).

Table 4. Yield fibers according to treatment time.

Treatment time (h)	Output (%)
05	0.0
08	5.6
12	4.4
21	4.4

The recent studies have shown that Na⁺ has a favorable diameter for penetrate between crystalline structures and with presence of water molecules to create spaces. In this structure, the -OH groups of the cellulose are converted to -ONa groups, expanding the dimensions of the molecules as it showed on the following reaction:



Subsequent washes with water will remove the Na-ion bonds. NaOH allows a complete transformation of cellulose I network to cellulose II, unlike other alkaline solutions that only lead to a partial transformation of the network (Johnson, 1979; Shenouda, 1979).

Effect of treatment time

Table 4 shows that treatment for 8 h in sodium hydroxide gave the best results than the others treatment time. During the extraction, the reaction of sodium hydroxide disassociates the fibers by breaking the bonds between lignin and polysaccharides of the cell walls (Wang and Sain, 2007). The extraction rate changes in relation to the concentration of NaOH and processing time. Table 4 shows that the ideal time for *R. monosperma* fibers extraction was 8 h. More or less than 8 h processing time, the yield was low, because less than 8 h was insufficient for the reaction of fiber extraction and over than 8 h, the NaOH has degraded the cellulosic fibers, which was undesirable. Sandy and Bacon (2001) reported that alkaline extraction can cause degradation of the cellulose leading to the extraction of nanofibers.

Effect of pressure

The pressure is also an essential element for a good separation of fibers; the pressure facilitates the separation of fibers. The Application of pressure of 2.2 bars gave a yield of 11.51%, whereas without autoclaving the yield was 9.48%.

Fiber's characterization

Mechanical characteristics

The results of tensile test shows that Young's modulus of

Table 5. Mechanical properties of *Retama monosperma* and some principal fibers (Bledzki and Gassan, 1999; Sandy and Bacon, 2001; Bismarck et al., 2005; Elenga et al., 2009; Elenga, 2009; Agu, 2014).

Fiber	Strength (MPa)	Elongation to failure (%)	Young's modulus (GPa)
<i>Retama monosperma</i>	110	4.6-4.7	13.3
Flax	345-1035	1.3-3.3	27.6
Sisal	600	3	12
Jute	396-773	1.5-1.8	26.5
Hemp	690	1.6	30-60
cotton	287-597	7-8	5.5-12.6
<i>Raffia textilis</i>	148-660	2	28-36
<i>Raffia farinifera</i>	500	4	12.3
Kenaf	700	3	55

Table 6. Density of some vegetable fiber (Sandy and Bacon, 2000; Béakou et al., 2008; Elenga et al., 2009).

Fiber	Density (g/cm ³)
<i>Retama monosperma</i>	1.3
Flax	1.5
Sisal	1.5
Jute	1.3
Hemp	1.15
Cotton	1.5-1.6
<i>Rhectohyllum camerunese</i>	0.947
<i>Raffia texillia</i>	0.75

13.3 GPa was found for *R. monosperma* fibers (Table 5). Compared to the *Raffia farinifera* (28-36 GPa), Young's modulus value of *R. monosperma* fiber was lower and it was about half that of Jute fiber (26.5 GPa) and Flax fiber (27.6 GPa). But it was higher than cotton which its Young's modulus ranged between 5.5 and 12.6 GPa according to the literature (Agu, 2014). Thus, the *R. monosperma* fibers appears to be more flexible than *R. farinifera*, flax and jute but more rigid than cotton. *R. monosperma* fibers tensile strength was 110 MPa (Table 5). Mechanical properties have a direct relationship with cellulose crystallinity (Sanadi, 2004; Sena Neto et al., 2013), length (Morlier and Khenfer, 1991), microfibrillar angle, cellulose content, molecular structure (Mukherjee and Satyanarayana, 1986), and fibers orientation (Djoudi et al., 2009). The elongation to failure was about 4.6 to 4.7%. It was higher than Flax (1.3 to 3.3%), Sisal (2 to 2.5%) and Hemp 1.6% but it was lower than cotton (7 to 8%).

Density

R. monosperma fibers density was 1.3 g/cm³. Table 6 shows that *R. monosperma* fibers density was same like that of jute and sisal fibers but lower than Flax and Sisal

Table 7. Water absorption of some vegetable fibers (Savastano et al., 1999; Ramakrishma and Sundararajan, 2005; Toledo Filho et al., 2005).

Fiber	Water absorption (%)
<i>Retama monosperma</i>	53
Sisal	190-250
Bamboo	145
Jute	281

fibers density. *Raffia texillia* fibers density is lower than all vegetable fibers (Elenga et al., 2009). There are a negative correlation between the density and Young's modulus. When the density is lower, the young's modulus and strength are higher. In general, vegetable fibers present densities lower than synthetic's fiber like glass fibers (2.5 g/cm³) (Bledzki and Gassan, 1999).

Swelling test using optical microscope

The absorption capacity of *R. monosperma* fibers was lower than that of all vegetable fibers represented in Table 7. It was three times lower than Bamboo fibers.

Floatability

The test shows that *R. monosperma* fibers had a hydrophobic character, which was due to its hydrophobic surface. The hydrophobic surface and the limited absorptive character may be due to treatment with NaOH.

Chemical composition

Polysaccharides composition of *R. monosperma* fibers was: 87.3% cellulose, 7.5% hemicelluloses, 4.2% pectin

Table 8. Fiber wall chemical composition (Bledzki and Gassan, 1999).

Fiber	Lignine	Hemicellulose	Cellulose
Flax	2	12	64.1
Sisal	9.9	12	65.8
jute	11.8	12	64.4
Ramie	0.6	13.1	68.6
<i>Retama monosperma</i>	1	7.5	87.3

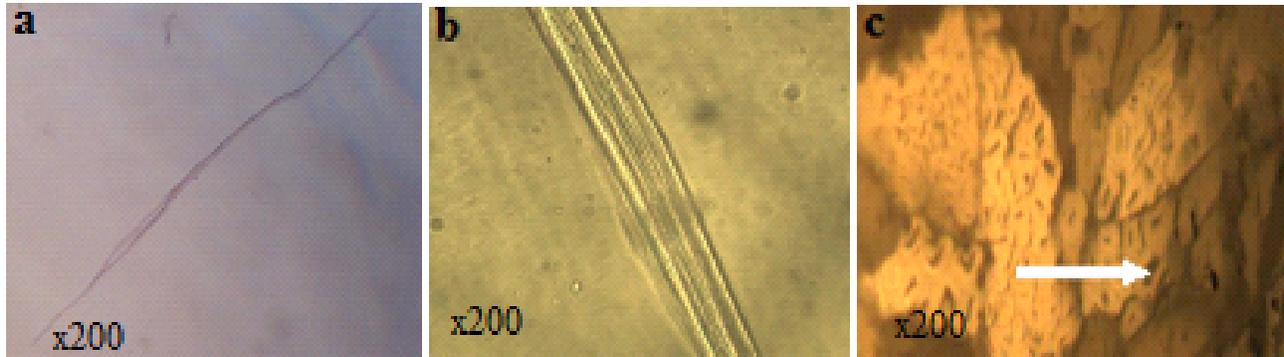


Figure 2. Microscopic observation of *Retama monosperma* stems and fibers. **a)** Microscopic observation of fiber cell of *Retama monosperma* (x200). **b)** Cross section of fibrous bundles *Retama monosperma* (x200). **c)** Microscopic observation of *Retama monosperma* fibers cross-section (x200).

and 1% lignin. The results show that *R. monosperma* fibers were rich in cellulose 87.3%; richer in cellulose than Flax 64.1% and Ramie 68.6%. Fibers with a higher cellulose fraction are more suitable for fibrous applications (Sena Neto et al., 2013). Cellulose is the main structural component of the lignocellulosic fibers, as it provides strength and stability to the cell walls and to all the fiber structure (Paster et al., 2005). Therefore, the cellulose content in a fiber or fiber bundle affects its properties and consequently, its applications. On the contrary, the percentage of the fraction of the hemicelluloses of *R. monosperma* fibers was lower compared to other plants (Table 8).

Morphometric characterization

R. monosperma fibers had an average length of 155.7 mm (Table 1). Compared to kenaf fibers (3 to 7 mm) (James et al., 1999) and to cotton fibers (0.83 mm) (Ververis et al., 2004), *Retama monosperma* fibers were longer. This characteristic interested the textile and biocomposites industry. The morphological characteristics of fibers, length and width are important factors in mechanical characteristics of fibers (rigidity or flexibility). In general, the length of plant fibers is between 100 and 150 mm and width from 10 to 50 μm (Fogtdal, 1990) (Table 9).

Morphology and ultrastructure of *R. monosperma* fibers

Figures 2a and 2b shows the morphological difference between fiber and fiber cell. A fiber (Figure 2b) is composed of many fibers cell called elementary fibers (Figure 2a). The microscopic observation (Figure 2c) shows that elementary fibers have a lumen (indicated with arrow). This characteristic is very interesting for thermal and acoustic insulation. Although, no study has been performed on the insulation performance for each plant fiber. Kymäläinen and Sjöberg (2008) and Hepworth and Brus (2000) reported that there are a link between fiber porosity and thermal property. The SEM observations show clearly the morphology, shape and microstructure of *R. monosperma* fibers (Figure 3a, b and c). One of these fibers was separated from the bundle (Figure 3, a, arrow).

Conclusion

This is the first published paper on the extraction of *R. monosperma* fibers from leaves. Our study shows that, this species is very rich in fiber and it is easy to extract them with an interesting yield compared to several plants already exploited, which makes possible its valuation for industrial purposes, especially, if it is a wild plant

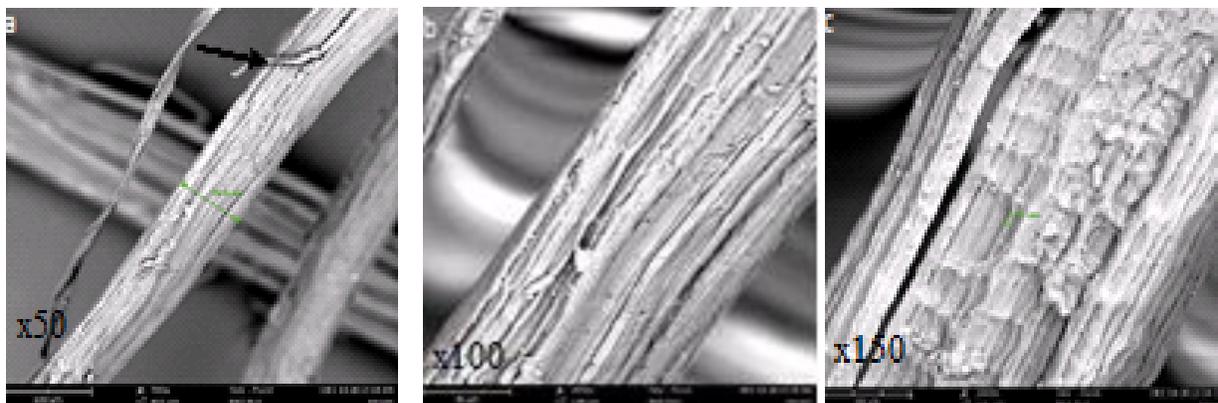


Figure 3. Scanning electron microscopy observation of *Retama monosperma* fibers. a) X50. b) X100. c) X150.

Table 9. Morphological characteristics of *Retama monosperma* fibers.

Average length of elementary fibers (μm)	Average length of fibers (mm)	Average width of elementary fibers (mm)	Average width of fibers (mm)
700	155.7	0.002	0.116

widespread in Algeria and which requires little water. Also, their fibers exhibit interesting properties such as higher cellulose content (86%), high elasticity (4.6 to 4.7%) and low density ($1.3\text{mg}/\text{cm}^3$). These characteristics enable *R. monosperma* fibers to be the preferable plant fibers in textile and composite industry. Finally, we recommend further studies for better understanding of the chemical, physical and mechanical characteristics. Furthermore, structural studies such as cellulose crystallinity as well as plant age and seasonal variation are needed for efficient exploitation of this species in Algeria.

Conflict of interests

The authors did not declare any conflict of interest.

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