

## TUNISIAN DATE PALM RACHIS USED AS AN ALTERNATIVE SOURCE OF FIBRES FOR PAPERMAKING APPLICATIONS

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Every year, significant amounts of date palm rachises are accumulated in Tunisia. The rational valorisation of this renewable resource is therefore imperative, in order to fulfil the sustainability approach. In this context, this work aims to study the potential use of date palm rachises as a raw material for papermaking and to compare it with other sources of lignocellulosic fibres, such as wood, non-wood species, and agricultural wastes. For this purpose, soda-anthraquinone pulping of date palm rachis was performed giving rise to a yield of 45% (w/w). This value is similar to that obtained by pulping non-wood materials and is higher than that corresponding to the pulping of agricultural residues. The resulting pulps were subsequently refined using a PFI mill refiner at 0, 500, 1500, and 3000 revolutions, screened through a 0.15 mm mesh size sieve and used to produce conventional handsheets. Both pulps and papers were fully characterized in terms of morphological, chemical and physical properties, according to commonly used standards. The physical properties of the prepared handsheets were very similar to those displayed by other papers made of common lignocellulosic fibres. Furthermore, the pulps exhibited a good drainability together with excellent mechanical properties of the ensuing papers. For these reasons, date palm rachises could be considered as a potential source of fibres for papermaking applications.

*Keywords:* Date palm rachis, soda-anthraquinone pulps, pulp refining, paper properties.

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### INTRODUCTION

In the last few years, pulp production has been growing enormously, with a consequent increase in wood consumption and in pulp demand, which attained about 180 million tons in 2008. Hence, in order to preserve the forest resource and to satisfy the increasing demand in pulps, especially in the textile and papermaking industries, as well as for innovative materials such as cellulose fibres-based composites, new fast growing non-wood sources of lignocellulosic fibres must be sought. In fact, several works related to the study of annual plants from various geographical locations are available in the literature. They deal with *Cynara cardunculus L* and banana pseudo-stems (Antunes *et al.* 2000; Cordeiro *et al.* 2004; Abrantes *et al.* 2007), *Ipomea carnea* and *Cannabis sativa* (Dutt *et al.* 2008), kenaf (Chia *et al.* 2008), alfa (Belgacem *et al.* 1986), sorghum stalks (Jimenez *et al.* 1993), holm oak (Eugenio *et al.* 2006), bamboo (Khristova *et al.* 2006),

*Amaranthus caudatus* L. (Fiserova *et al.* 2006), *Atriplex hortensis* L. (Fiserova *et al.* 2006), *Helianthus tuberosus* L. (Fiserova *et al.* 2006), *Miscanthus sinensis* (Barba *et al.* 2002), *Arundo donax* L. (Shatalov *et al.* 2001) and so on. In most cases, the complete characterization of these new sources of fibres showed that they could be considered as promising materials.

In Tunisia, date palm (*Phoenix dactylifera*) is one of the most cultivated plants, and more than 4 million of palms occupy about 32 thousand hectares, as reported by the Tunisian Ministry of Agriculture. This culture produces a huge amount of date palm rachises, which are left on soils to biodegrade and thus to fertilise them and/or to be incinerated for energy recovery purposes. Date palm has not been extensively studied for papermaking applications. Ezzat (1974) showed that Egyptian date palm leaves could be a good source of cellulose fibres. Khristova *et al.* (2006) evaluated date palm rachises and leaves from Sudan for papermaking applications. For this purpose, an experimental work was performed in order to optimize the pulping conditions. The effect of refining on some physical properties (tensile, burst and tear indexes) of the papers produced was studied. The results obtained showed date palm could be a promising material for the paper industry. As reported in a previous paper (Khiari *et al.* 2010), Tunisian date palm rachis contains a high percentage of polysaccharides, *i.e.* 74% of holocellulose, and gives paper sheets with good mechanical properties, even if the initial pulps were unrefined. This work that is a logical continuation of the investigation undertaken before, aims to study the behaviour of a date palm rachis pulp during refining and to establish the effect of such a crucial operation on the properties of the produced pulps and papers. First, the impact of refining on the morphology of the fibres and their swelling behaviour was studied, before assessing the evolution of their surface charge. Then, the physical properties of conventional handsheets prepared for each refining level were determined. Finally, all these results were discussed and compared with data available in the literature and dealing with non-wood-, wood-, and agricultural crops-based papers.

## EXPERIMENTAL

### Materials and Methods

The date palm rachises used in this study were collected from the region of Monastir-Tunisia in September 2008 and dried under natural conditions in October of the same year. Date palm rachises, with dimensions of about 1 m length and 6-7 cm diameter, were cut into small pieces of 1 to 3 cm and extensively washed before being cooked.

### Pulping and Refining Conditions

The delignification stage of the palm rachis was performed according to experimental conditions described in a previous publication (Khiari *et al.* 2010). Briefly, soda-anthraquinone pulping was carried out using a 1000 mL volume reactor. The pulp cooking was performed at 160°C, with a total alkali charge of 20% expressed in NaOH (w/w, based on oven dried (o.d.) rachis), an anthraquinone concentration of 0.1% (w/w, with respect to o.d. material) and a cooking time of 120 min. The obtained pulp was

separated from black liquor by filtration on a nylon wire and washed several times, until attaining a neutral pH. Then, a sample of pulp (30 g) was disintegrated in 2L of water according to the standard method ISO 5263-1. After that, the suspension was filtered until a consistency of 10% was reached and refined in a PFI mill refiner (ISO 5264-2) at 0, 500, 1500 and 3000 revolutions. Finally, the pulp was screened through a 0.15 mm mesh size sieve in order to remove uncooked materials. The screening yields were determined as the ratio of the weight of o.d. pulp after and before screening.

### Pulp Characterization

The total amount of carboxylic groups in both the unrefined and the 3000 revolutions refined pulps was assessed by conductimetric titrations according to the procedure described by Katz *et al.* (1984) and Fras *et al.* (2004). Prior to titration, the investigated pulp (0.6 to 1 g in 300 mL of deionized water) was acidified by adding 1 mL of HCl solution (0.1 mol.L<sup>-1</sup>) and extensively washed. The conductivity of the pulp suspension was then adjusted to 600 μS.cm<sup>-1</sup> with a 0.5 mol.L<sup>-1</sup> NaCl solution. Finally, the titration of 500 mL of a pulp sample was performed with a NaOH solution (0.01 mol.L<sup>-1</sup>), after the addition of 0.5 mL of a solution of HCl (0.1 mol.L<sup>-1</sup>).

The charges of the dissolved and colloidal substances associated to the pulp suspensions were determined by using the colloidal titration method performed on a PCD02 (Mutek) apparatus equipped with an automatic titrator. After a two-steps filtration (through a 70 μm and a 20 μm Nylon wire in order to remove the fibres), samples from the unrefined and refined pulps were studied. The method consists of measuring the anionic charges of the filtrates, by adding slowly a cationic polyelectrolyte (PolyDADAMC) until obtaining a zero potential (equivalence point). This anionic charge is associated to the polyelectrolytes and colloids released in the water by the fibres, as well as to the fraction of cellulosic fine elements, which were not removed by the two-steps filtration.

The morphological properties of the date palm rachis fibres before and after refining were studied thanks to a MORFI (LB-01) analyzer, developed by Techpap – France (Passas *et al.* 2001). The main parameters (fibre length and width, content in fine elements) were measured by image analysis of a diluted suspension flowing in a transparent flat channel observed by a CCD video-camera. Fine elements are defined as particles with size less than 200 μm and their corresponding content is the ratio of the total length of fines to the total length of the elements present in the suspension. Arithmetic and weighted lengths (respectively  $\bar{l}_A$  and  $\bar{l}_W$ ) are calculated as follows:

$$\bar{l}_A = \frac{\sum_i n_i l_i}{\sum_i n_i} \quad \text{and} \quad \bar{l}_W = \frac{\sum_i n_i l_i^2}{\sum_i n_i l_i} \quad \text{Eqs. (1) and (2)}$$

Water Retention Values (WRV) were assessed by centrifuging wet pulp samples for 15 min at 3000 g according to Silvy's method (Silvy *et al.* 1968). Before and after drying, the samples were weighted and WRV were calculated by using the following equation:

$$WRV(\%) = 100 \times \frac{(M_1 - M_2)}{M_2} \quad \text{Eq. (3)}$$

where:  $M_1$  and  $M_2$  are the weights of the sample after centrifugation and after drying, respectively. The drying step was performed at 105°C during, at least, 24 hours.

The pulp drainability was determined by measuring the Shopper Riegler degree according to the standard method ISO 5267-1. All these measurements were replicated 3 times, apart from the WRV that was measured six times for each pulp.

### Preparation and Characterization of Conventional Handsheets

For each refining level, ten conventional handsheets with a basis weight of about 60g/m<sup>2</sup> were prepared on a Rapid Khöten sheet former, following the ISO 5269-2 standard method. The structure of the obtained papers was observed using a Zeiss ULTRA55 Scanning Electron Microscope (SEM) with an acceleration voltage of 15 kV. Each sample was prepared with a gold/palladium coating before the analysis. Regarding their physical properties, the handsheets were conditioned at 23°C and 50% of relative humidity before testing, as recommended by the ISO 187 standard. Then, the basis weight (ISO 536), thickness (ISO 534), bulk and permeability (ISO 5636-3) were measured. Finally, the main mechanical properties were assessed according to ISO standard methods. Tensile tests were performed with an L&W tensile apparatus. Adamel Lhomargy devices were used to determine the burst and tear strengths. Zero-Span Breaking Length (ZSBL), Internal Bond Strength (IBS), and Short-span Compression Test (SCT) were respectively assessed by using a Pulmac apparatus (TS-100), a IDM device and a Strip Compression Tester (Büchel – van der Korput). As recommended by the various standards used, all the measurements were repeated at least 10 times, thus allowing the determination of standard deviations. Paper thickness measurements were replicated 20 times.

## RESULTS AND DISCUSSION

### Pulp Properties

The chemical composition of the Tunisian date palm rachis was determined in a previous study (Khiari *et al.* 2010) which showed that it contains about 75% of holocellulose. This quantity is higher than that present in many other annual plants (Antunes *et al.* 2000; Cordeiro *et al.* 2004; Jimenez *et al.* 1992, 1993, 2006, 2008; Fiserova *et al.* 2006; Barba *et al.* 2002; Sarwar *et al.* 2006; Gominho *et al.* 2001; Jimenez and Lopez 1990). However, it is similar to that encountered in wood, which makes date palm rachis a competitive potential source of cellulose, especially for papermaking applications. Moreover, its significant content in hemicelluloses (30%) constitutes another positive feature, as this component may play a plasticizing role, thus increasing the refining kinetics and the inter-fibre bonding in the ensuing paper sheets. The soda-anthraquinone cooking stage of the date palm rachis is characterized by a cooking yield of 45%, as reported in Table 1.

**Table 1.** Cooking and Screening Yields for Date Palm Rachis Pulps

PFI revolutions	Cooking yield %	Screening yield %
0		94
500	44.8	96
1500		99
3000		100

(Each measurement was replicated 3 times. The deviation between the experimental values did not exceed 5%.)

After screening, the unrefined pulp contained 69% of holocellulose and 5% of lignin; its Kappa number was close to 50. As screening is performed after refining, the screening yield increased with the PFI mill revolutions (see Table 1) and reached 100% for the most refined pulp. This can be attributed to the mechanical action in the refining device, which disintegrates the remaining uncooked portions, thus recovering the whole material.

#### *Total charge and soluble and colloidal charge determinations*

The total charge and that associated to the soluble and colloidal substances are presented in Table 2. As previously explained, the former was determined by conductimetric titrations, for both the unbeaten and 3000 revolutions refined pulp whereas the latter was established by colloidal titrations for each refined pulp.

**Table 2.** Total and Surface Charges for Unrefined and Refined Pulps \*

PFI revolutions	Colloidal titration ( $\mu\text{eq}\cdot\text{L}^{-1}$ )	Conductimetric titration ( $\mu\text{eq}\cdot\text{g}^{-1}$ )
0	15.5	291
500	28.2	Nd **
1500	30.9	Nd **
3000	37.5	270

\* Each measurement is replicated 3 times. The deviation between the experimental values did not exceed 15% for the colloidal titrations and 5% for the conductimetric titrations.

\*\* Nd: not determined

This set of experiments shows that the date palm rachis pulp was highly charged, i.e.  $291 \mu\text{eq}\cdot\text{g}^{-1}$ . This content is similar to that of wood or high-yield pulps, but is dramatically higher than that encountered for common chemical pulps (see Table 3). For instance, unbleached chemical eucalyptus pulps, which are known to be highly charged compared with other chemical pulps, exhibit amounts of acidic groups close to  $180 \mu\text{eq}\cdot\text{g}^{-1}$  (Laine and Stenius 1995). Unfortunately, very few data related to the characterization of non-wood pulps are available in the literature, and no comparison is then possible. A similar total charge ( $270 \mu\text{eq}\cdot\text{g}^{-1}$ ) was obviously obtained for the refined pulp, despite a slight decrease probably due to the release of charged components from

the fibres during the refining stage and their removal during extensive washing. Thus, when the cell wall is exposed to mechanical and hydrodynamic shears, peptised hemicelluloses and polyuronic acids get partially transferred to the liquid phase (Goulet and Stratton 1990; Bhardwaj *et al.* 2004).

**Table 3.** Content in Acidic or Carboxylic Groups of Different Pulps and Fibres

(KN : Kappa Number)

Pulps	Acidic groups or carboxylic groups* ( $\mu\text{eq.g}^{-1}$ )	Dosage methods	Authors
Eucalyptus pulp (unbleached kraft pulp)	173*	Ion exchange	Mujé <i>et al.</i> 2006
Olivee tree pulp (organosolv pulp)	247*		
Spruce pulp (unbleached kraft pulp, yield:49%)	139* (KN=72.6)	Methylene blue dye adsorption	Goulet and Stratton, 1990
Spruce pulp (unbleached kraft pulp, yield:48%)	91* (KN=36.1)		
Spruce pulp (unbleached kraft pulp, yield:44%)	54* (KN=17.8)		
Spruce pulp (bleached kraft pulp)	31	Conductimetric titration	Fras <i>et al.</i> 2004
Cotton fibres	43.2		
Viscose fibres	48.6		
Modal fibres	27.2		
Lyocell fibres	20.6		
Unbleached kraft pulp (Yield : 66%)	201	Conductimetric titration	Katz <i>et al.</i> 1984
High-yield bisulfite pulp (Yield : 70%)	295		
Low-yield bisulfite pulp (Yield : 47%)	30		

By contrast, the dissolved and colloidal charge associated to the tested pulps depended strongly on the refining level, as reported in Table 2; the cationic demand of the most refined pulp (3000 revolutions) was 3 times greater than that of the unbeaten counterpart. As previously stated, this behaviour can be explained by the leaching of charged components from the fibres to the surrounding water. The very fine elements generated during the refining stage may also induce an increase of the cationic demand, even if a two-steps filtration is performed prior to the titration. Such a high cationic demand must be taken into account, as it can negatively impact the efficiency of the cationic wet end additives and limit the closure of water circuits during papermaking process.

#### Morphological Properties

Fibres' morphology was assessed for the unrefined and refined pulps. The average length (mm) and width ( $\mu\text{m}$ ) as well as the percentage in fine elements are summarized in Table 4a. The fibre length ( $\bar{l}_A=0.69$  mm –  $\bar{l}_w=0.89$  mm) and width (22  $\mu\text{m}$ ) of the unrefined date palm rachis pulp were found to be in the range of those associated with common agriculture plants, such as *Amaranthus caudatus* L. (Fiserova *et al.* 2006), orache (Fiserova *et al.* 2006), Jerusalem artichoke (Fiserova *et al.* 2006), holm oak (Eugenio *et al.* 2006), *Cannabis sativa* (Dutt *et al.* 2008) or *Cynara cardunlus* L.

(Antunes *et al.* 2000; Abrantes *et al.* 2007). Nevertheless, the fibre length was lower than that of most of wood fibres. It is also worth noting that the unrefined date palm rachis pulp was characterised by a high content in fine elements (30% in length). Besides, it appears that refining did not induce significant variations of the pulp morphology. In fact, the decrease of the fibre length was negligible and only a small increase (about 13%) in the percentage of fine elements can be observed for the pulps subjected to the higher refining levels (see Table 4a). Regarding the latter parameter, it is interesting to note that, for wood pulps, this increase can reach 30% for the same Schopper Riegler range. As expected, the fibre width of the different pulps slightly increases with the refining level: this classical behaviour can be related to the swelling and/or fibrillation of the fibres.

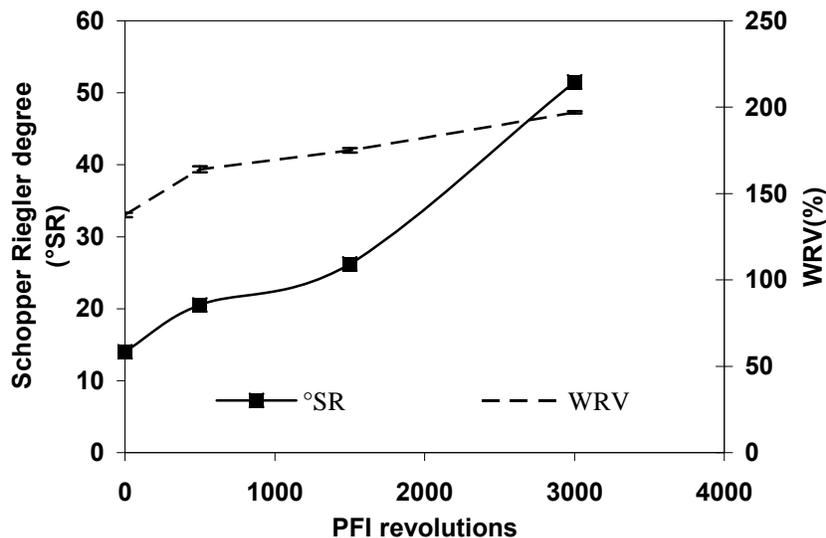
**Table 4a.** Effect of Refining on Fibres' Morphology, Drainability and WRV of Date Palm Rachis Pulps

PFI revolutions	Fibre length (mm)	Fibre width ( $\mu\text{m}$ )	Fine content (%)	Drainability ( $^{\circ}\text{SR}$ )	WRV (%)
0	$\bar{l}_A = 0.69 - \bar{l}_W = 0.89$	22.3	30.8	14	138
500	$\bar{l}_A = 0.69 - \bar{l}_W = 0.89$	23.0	31.7	21	164
1500	$\bar{l}_A = 0.68 - \bar{l}_W = 0.88$	23.5	32.0	26	175
3000	$\bar{l}_A = 0.67 - \bar{l}_W = 0.86$	24.1	36.2	52	197

#### *Drainability and Water Retention Values*

The Schopper Riegler degrees and the Water Retention Values (WRV) were determined for each pulp, as summarized in Table 4a and Fig. 1. The unrefined date palm rachis pulp exhibited a Schopper Riegler degree ( $14^{\circ}\text{SR}$ ) comparable to that of unrefined softwood pulps and lower than that of other annual plants, which usually lies around  $30\text{--}40^{\circ}\text{SR}$  (Dutt *et al.* 2008; Khristova *et al.* 2006). This value is surprisingly low if we consider the morphological properties of the pulps, in particular the fines content. We can suppose that it is related to the structure of the fibre mat formed during the filtration and maybe to the fibres stiffness. When the PFI refining increases from 0 to 1500 revolutions, the drainability slightly decreases from 14 to  $26^{\circ}\text{SR}$ . The refining effect was more pronounced after 1500 revolutions in the PFI mill: the Schopper Riegler degree of the most refined pulp reached a value of 52. From an industrial point of view, this good initial drainability is a very interesting property, as the date palm rachis pulp will not limit the runnability of the paper machine, as it is the case of other pulps made from annual plants.

Regarding now the refining kinetics, as depicted on Figure 2, it is observed that the date palm rachis pulp is easier to refine than wood pulps. Refining kinetics of other non-wood pulps are also reported for comparison: their kinetics are close to that of the date palm rachis pulp. This classical behaviour of non-wood based pulps is generally attributed to their high content in hemicelluloses and in charged groups, which facilitates the penetration of water in the fibre wall.



**Fig. 1.** Evolution of Schopper Riegler degrees ( $^{\circ}\text{SR}$ ) and Water Retention Values (WRV) for different refining levels

The water retention value (WRV) of the unrefined pulp (138%) was significantly higher than that obtained for hardwood and softwood pulp (Silvy *et al.* 1968), which generally varies between 90 and 100%. Here again, the high hemicellulose and charge contents play an important role in the enhancement of hydration, as it was already observed for other annual plants (Antunes *et al.* 2000; Cordeiro *et al.* 2004; Abrantes *et al.* 2007; Dutt *et al.* 2008; Chia *et al.* 2008; Jimenez *et al.* 1993; Eugenio *et al.* 2006; Khristova *et al.* 2006; Fiserova *et al.* 2006; Barba *et al.* 2002; Shatalov *et al.* 2001; Khiari *et al.* 2010). Additionally, WRV increased with refining level, but the increase did not exceed 50% for the most refined pulp (52 $^{\circ}\text{SR}$ ) while, for hardwood and softwood pulps, such an increase may reach more than 100%.

It is worth noting that the date palm rachis pulp displayed both a good drainability and a suitable WRV. These two features permit a proper runnability of the machine and good mechanical properties of the ensuing papers. Moreover, the kinetics of refining is fast: fibrillation and hydration occur for low revolution numbers in the PFI mill, so that we could expect that energy consumption related to this operation will be reduced.

## Paper Properties

### *Paper structure*

Paper structure was qualitatively studied using Scanning Electron Micrography (SEM), as shown from Figure 3. SEM micrographs of the date palm rachis pulp handsheets refined at 0, 1500 and 3000 revolutions, show clearly the impact of refining. Figure 3a illustrates the increase of the fibre flexibility during refining: the sheet surface of papers made from refined pulps appears smoother owing to the better conformability of the fibres. Figure 3b allows observing individual fibres and confirms the previous discussion: the fibres appear to be less rigid and more fibrillated when the refining level increases. Finally, Figure 3c illustrates the effect of refining on the sheet density.

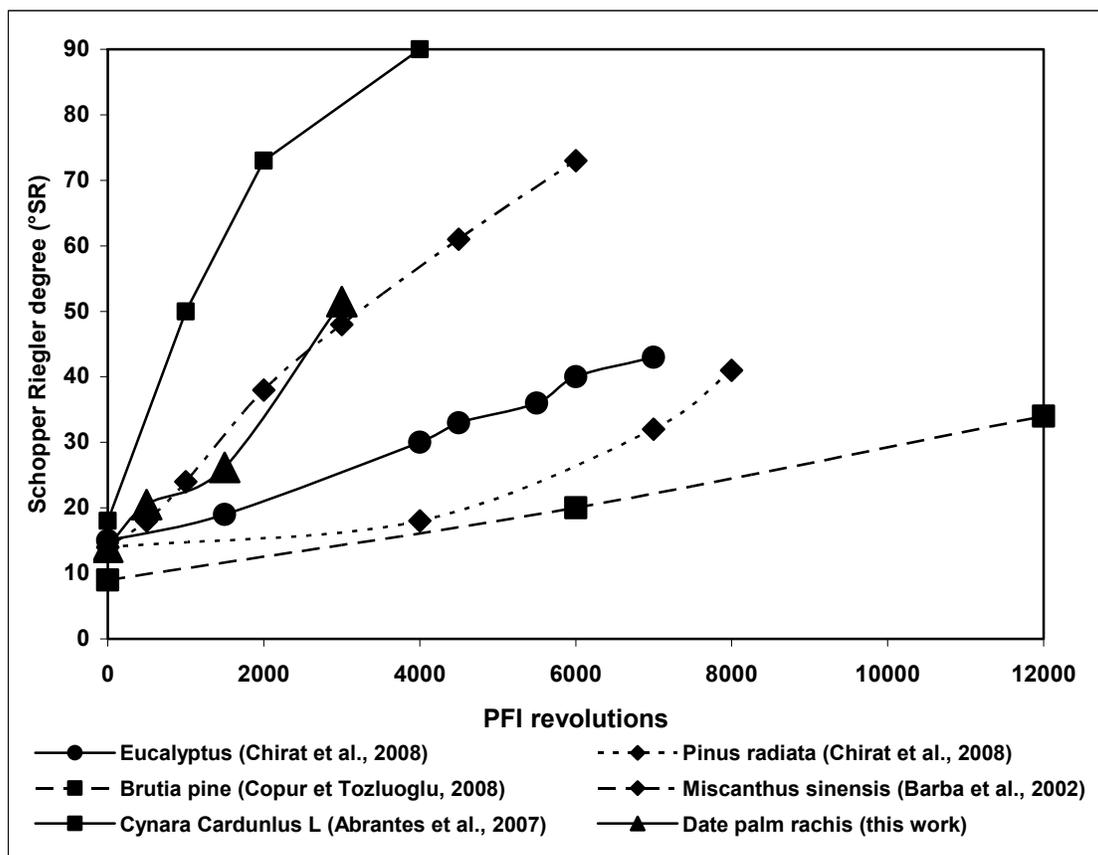
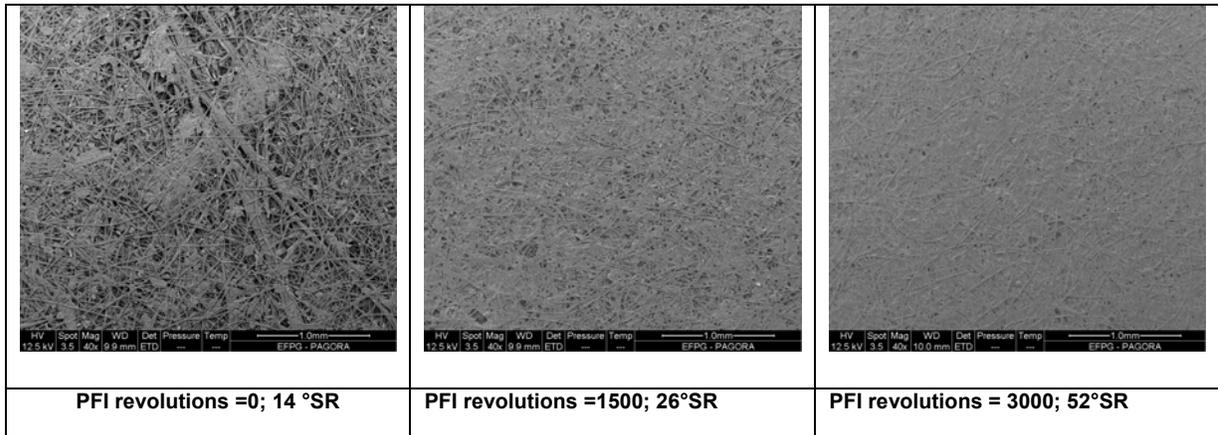


Fig. 2. Refining kinetics of pulps made from various lignocellulosic materials

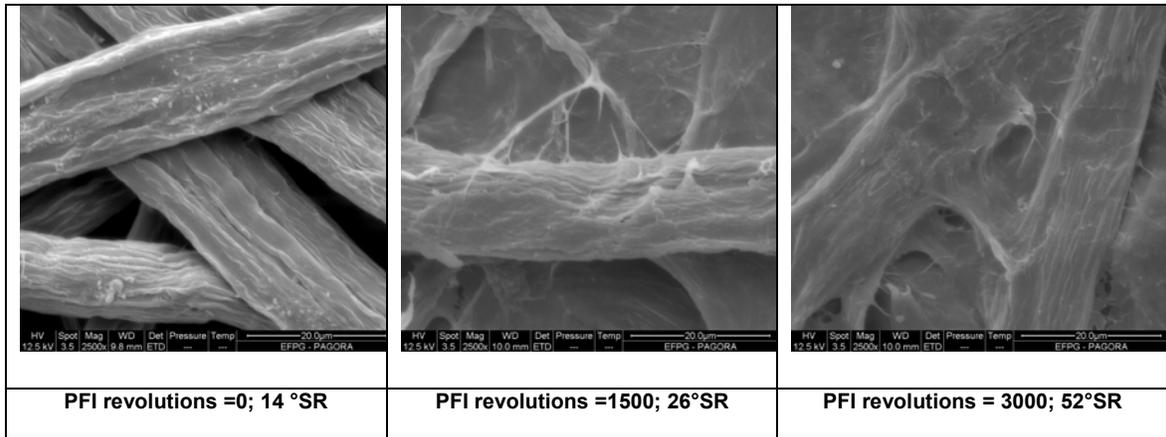
In addition, structural properties were assessed, as reported in Table 4b. The thickness of both the unbeaten and the 500 revolutions refined pulps was over-estimated due to the presence of a significant amount of uncooked materials. Consequently, the related bulks measured for the same pulps were also over-estimated. Nevertheless, the tendencies observed for both the bulk and the permeability of the produced papers are consistent with the SEM images analysis. Refining induces an increase of the paper density and then a decrease of the bulk and the permeability. But, it is worth noting that this decrease is limited and, even for papers produced from the most refined pulp (52°SR), the bulk and the permeability remained relatively high.

Table 4b. Effect of Refining on The Structure Properties of Handsheet Papers Made From Date Palm Rachis Pulps

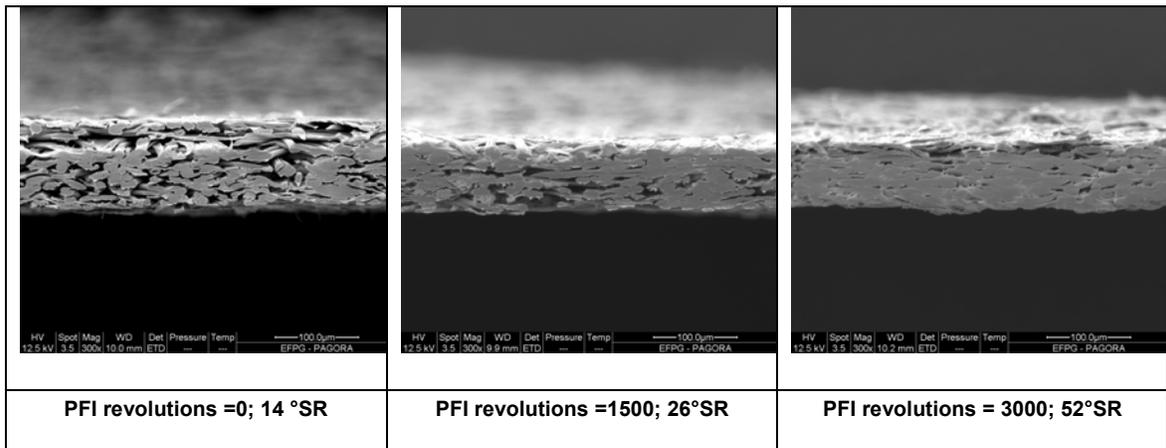
PFI revolutions	Basis weight (g/m <sup>2</sup> )	Thickness (μm)	Bulk (cm <sup>3</sup> .g <sup>-1</sup> )	Permeability (cm <sup>3</sup> .s <sup>-1</sup> .Pa <sup>-1</sup> .m <sup>-2</sup> )
0	63.9±1.89	141±6.11	2.15±0.03	449±0.042
500	57.7±0.51	112±1.02	1.94±0.017	244±0.018
1500	62.8±0.49	101±0.74	1.61±0.011	58±0.004
3000	64.4±0.37	99±0.81	1.42±0.008	7±0.0005



**Fig. 3.a.** Effect of refining on paper surface (date palm rachis pulps) – SEM images (Mag. 40<sup>×</sup>).



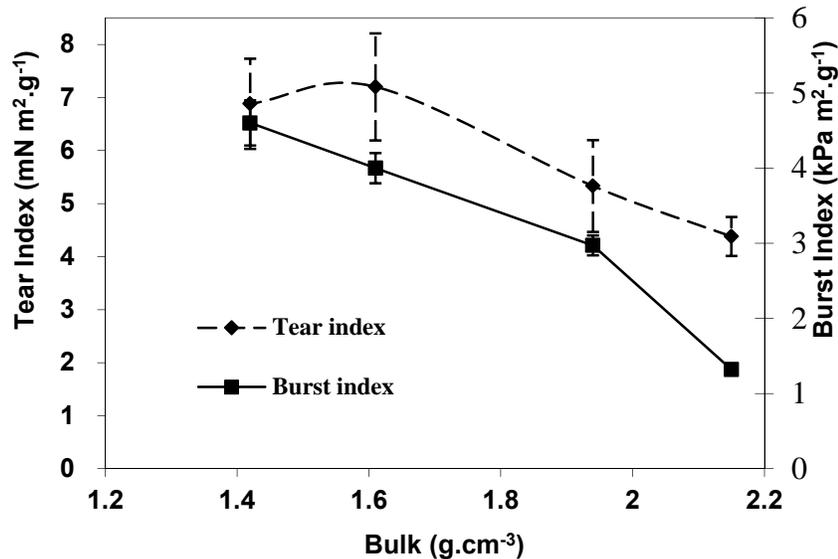
**Fig. 3.b.** Effect of refining on fibre structure (date palm rachis pulps) – SEM images (Mag. 2500<sup>×</sup>).



**Fig. 3.c.** Effect of refining on paper structure (date palm rachis pulps) – SEM images (Mag. 300<sup>×</sup>).

*Mechanical properties of paper*

The effects of refining on the main mechanical properties are summarized in Table 5 and illustrated in Fig. 4.



**Fig. 4.** Evolution of burst and tear indexes, as a function of bulk

Whatever the characteristics considered, the obtained values showed that rachis date palm based papers exhibited very good strength properties and that refining operation enhanced them in a great extent. For instance, the pulp refined at 1500 revolutions (26°SR) seems to correspond to an acceptable compromise between paper properties and drainability, because the values of the breaking length (6920 m), the Young modulus (4380 MPa), and the internal bond strength (570 J.m<sup>-2</sup>) are comparable or even better than those obtained with wood-based papers, refined in similar conditions. In addition, SCT values are high enough to envisage the use of these pulps in packaging applications. From the wet zero-span breaking lengths, which reveal the intrinsic strength of the fibres, one can assume that cooking was conducted in optimal (or at least close to) conditions as the fibre strength appears to be quite good, whatever the refining level. The dry zero-span breaking lengths were also measured for each series. These values include the effect of fibre-fibre bonds formed during the drying stage performed under constraint.

Figure 4 depicts the evolutions of the burst and tear indexes, as a function of the bulk and shows that these two properties increased, when the sheet density decreased. For instance, the burst index reaches a value of 4.6 kPa m<sup>2</sup>.g<sup>-1</sup> for the most refined pulp, and the tear index was close to 7 mN.m<sup>2</sup>.g<sup>-1</sup>. Nevertheless, the tear index seemed to reach a plateau after 26 °SR, which has to be related to the shortening of the fibres, as well as, the increase of the fine content observed for the most refined pulp. This behaviour tends to confirm that refining has to be limited to a range in which its detrimental impact on fine content and fibre length is reduced, i.e., below 30 °SR in our case.

**Table 5.** Effect of Refining on The Mechanical Properties of Handsheet Papers Prepared From Date Palm Rachis Pulps

PFI revolutions	Elongation (%)	Breaking length (km)	Young modulus (GPa)	Specific tensile energy (mJ.g <sup>-1</sup> )	Internal bond strength (J.m <sup>-2</sup> )	Short-Span Compression Test (kN.m <sup>-1</sup> )	Zero–Span breaking length (km)	
							dry	wet
<b>0</b>	1.09±0.09	3.13±0.23	2.51±0.14	222±37	94±8.8	1.32±0.14	13.4±0.91	10.8±0.66
<b>500</b>	1.84±0.12	5.87±0.25	3.64±0.09	709±79.3	305±23.9	1.55±0.12	13.8±0.95	11.1±0.69
<b>1500</b>	2.79±0.10	6.92±0.14	4.38±0.12	1290±58.3	566±35.8	1.84±0.11	14.2±0.80	11.5±0.81
<b>3000</b>	3.07±0.24	7.81±0.33	5.29±0.12	1620±190	1010±43.6	2.26±0.12	14.6±0.62	11.3±0.28

In view of a comparison, the main properties of plants, wood and non-wood materials available in the literature are collected in Table 6. The mechanical properties of the rachis date palm-based papers are similar or even better than those of samples made of wood and non-wood species, with the exception concerning the tear index that may be slightly lower. Moreover, it confirms that the pulp produced from date palm rachis is characterized by a good drainability when compared to non-wood species and annual and perennial plants.

**Table 6.** Physical Properties of Papers Made From Various Lignocellulosic Materials. ( $^{\circ}$ SR: Schopper Riegler degree; F: Tensile force; BL: Breaking length; Elong: Tensile elongation; a: revolution number – PFI refining; b: refining time (minutes) -Valley beater or Jokro mill)

	Pulp			Paper					
	Cooking yield %	Refining level	$^{\circ}$ SR	Basis weight (g/m <sup>2</sup> )	F (N)	BL (km)	EI (%)	Burst index (kPa.m <sup>2</sup> .g <sup>-1</sup> )	Tear index (mNm <sup>2</sup> .g <sup>-1</sup> )
<b>Wood</b>									
Olive (Jimenez <i>et al.</i> , 1992)	47		21	67		1.6	1.1	3.33	
<i>E. citriodora</i> (Jimenez <i>et al.</i> , 2008)	43	1500 <sup>a</sup>		67				3.2	7.0
<i>E. tereticornis</i> (Jimenez <i>et al.</i> , 2008)	43	1500 <sup>a</sup>		67				2.5	5.4
Brutia pine (Copur and Tozluoglu, 2008)	46	0 <sup>a</sup>	9	75	15.6			0.75	9.3
		6000 <sup>a</sup>	20	75	59.1			3.95	8.0
		12000 <sup>a</sup>	34	75	59.7			4.2	7.8
<b>Non wood</b>									
<i>Ipomea carnea</i> (Dutt <i>et al.</i> , 2008)	44		44	60	70.2			3.62	3.5
<i>Cannabis sativa</i> (Dutt <i>et al.</i> , 2008)	50		45	60	76			5.7	4.7
<i>Sesbania aculeata</i> (Dutt <i>et al.</i> , 2008)	48		45	60	71			4.92	4.4
<i>Oxytenanthea abyssinica</i> (Khristova <i>et al.</i> , 2006)	42	0 <sup>b</sup>	15	60	55.4			3.4	10.9
		25 <sup>b</sup>	20	60	60.8			4.1	12.4
		30 <sup>b</sup>	27	60	64			4.3	12.4
		40 <sup>b</sup>	41	60	68.8			4.5	11.7
<i>Bambusa vulgaris</i> (Khristova <i>et al.</i> , 2006)	37	0 <sup>b</sup>	15	60	46			2.6	14.8
		25 <sup>b</sup>	38	60	49.4			3.1	19.8
		30 <sup>b</sup>	46	60	55.4			3.6	18.7
		40 <sup>b</sup>	58	60	61.4			4.1	19.2
Hemp (Zomers <i>et al.</i> , 1995)		0 <sup>a</sup>	38	60	48.7			2.7	10.2
		1000 <sup>a</sup>	45	60	60.1			3.6	9.5
		2000 <sup>a</sup>	55	60	63.3			4	9.2
<b>Annual and perennial plants</b>									
Jerus. Artichoke (Fiserova <i>et al.</i> , 2006)	30		40	75	67			4.5	4.1
Amaranth (Fiserova <i>et al.</i> , 2006)	38		40	75	70			4.1	3.9
Orache (Fiserova <i>et al.</i> , 2006)	45		40	75	59			3.7	3.3
Bagasse (Jimenez <i>et al.</i> , 2008)	43	1500 <sup>a</sup>		67				9.1	7.2
<i>Miscanthus sinensis</i> (Barba <i>et al.</i> , 2002)		0 <sup>a</sup>	14	75	31	3.2	1.4	1.23	10.1
		500 <sup>a</sup>	18	75	51	5.2	2.2	2.25	11.5
		1000 <sup>a</sup>	24	75	58.4	6	2.5	2.67	11.3
		2000 <sup>a</sup>	38	75	66	6.7	2.7	3.56	10.5
		3000 <sup>a</sup>	48	75	72	7.3	2.8	4.21	10.0
		4500 <sup>a</sup>	61	75	84	8.6	3.5	4.87	9.1
<i>Cynara Cardunlus L</i> (Antunes <i>et al.</i> , 2000; Abrantes <i>et al.</i> , 2007)	37	0 <sup>a</sup>	18	130			2.5	1.4	
		1000 <sup>a</sup>	50	130			4	5.6	
		2000 <sup>a</sup>	73	130			4.1	5.8	
		4000 <sup>a</sup>	90	130			4	5.8	
Date palm from Sudan (Khristova <i>et al.</i> , 2006)	42	0 <sup>b</sup>	60			4.4		1.9	10.0
		30 <sup>b</sup>	60			7.9		4.7	10.2
		35 <sup>b</sup>	60			8.1		4.6	10.3
		40 <sup>b</sup>	60			8.4		4.6	10.4
This work	45	0 <sup>a</sup>	14	60	29.4	3.1	1.1	1.32	4.4
		500 <sup>a</sup>	21	60	49.9	5.9	1.8	2.97	5.4
		1500 <sup>a</sup>	26	60	63.9	6.9	2.8	4	7.2
		3000 <sup>a</sup>	52	60	79.8	7.8	3.1	4.6	6.9

Regarding now the non-wood species and the annual plants, the data originated from the literature as well as our experimental data are reported on Figs. 5a and 5b, which depict the evolution of the burst and the tear indexes, for different levels of refining, as expressed in terms of Schopper Riegler degrees. Even if few data are available, these figures show that results obtained for Sudanese and Tunisian date palm rachis are quite close. They, therefore, allow confirming the previous discussion about the potentiality of this source of lignocellulosic fibres.

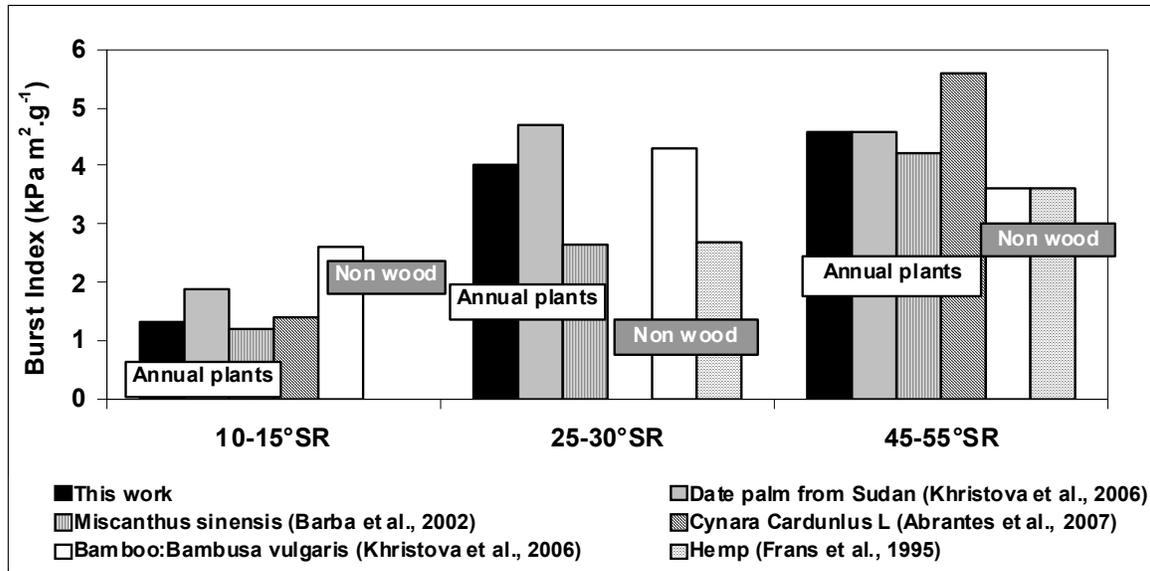


Fig. 5.a. Burst index of papers made from non-wood species and annual plants for different Schopper Riegler degrees

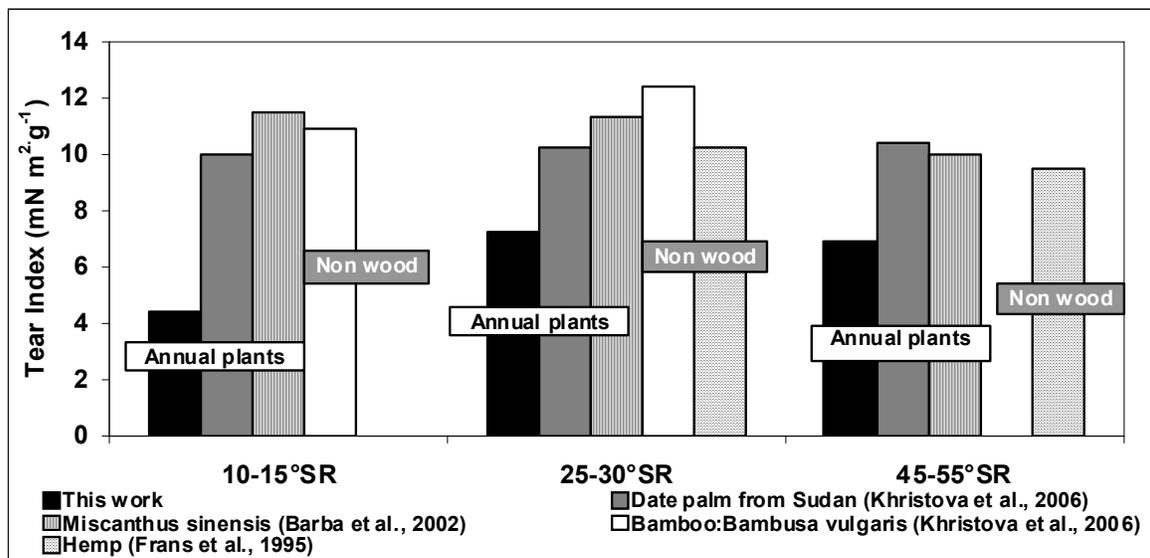


Fig. 5.b. Tear index of papers made from non-wood species and annual plants for different Schopper Riegler degrees

## CONCLUSIONS

In a context where the demand of pulp for classical papermaking applications as well as for the production of chemicals is growing, the availability of lignocellulosic fibres may become a restraint. From these considerations, we studied the properties of an agricultural residue, the Tunisian date palm rachis. The pulp, obtained by a conventional soda-anthraquinone cooking, is characterized by a very good drainability. Its morphological properties are similar to those of other non-wood materials, apart from the fibre length that is relatively low. All these properties are not deeply modified by the refining process, except for the drainability, which significantly decreased after a certain level of refining. On the other hand, refining greatly enhanced the mechanical properties of the produced laboratory paper sheets. To conclude, Tunisian date palm rachises may constitute a promising raw material for the production of lignocellulosic fibres, especially in the field of papermaking applications.

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## REFERENCES CITED

- Abrantes, S., Amaral, M. E., Costa, A. P., Duarte, A. P. (2007). “*Cynara cardunculus* L. alkaline pulps: Alternative fibres for paper and paperboard production,” *Bioresour. Technol* 98, 2873-2878.
- Antunes, A., Amaral, E., and Belgacem, M. N. (2000). “*Cynara cardunculus* L.: Chemical composition and soda-anthraquinone cooking” *Ind. Crop. Prod.* 12, 85-91.
- Barba, C., De la Rosa, A., Vidal, T., Colom, J. F., Farriol, X., and Montane, D. (2002). “TCF bleached pulps from *Miscanthus sinensis* by the impregnation rapid steam pulping (IRSP) process,” *J. Wood Chem. Technol.* 22(4), 249-266.
- Belgacem, M. N., Zid, M., Nicolski, S. N., and Obolenskaya, A. V. (1986). “Study of the chemical composition of alpha from Tunisia,” *Chim. Technol. Drev., Mej. Sbor. Trud.* 8, 111-114.
- Bhardwaj, N. K., Kumar, S., and Bajpai, P. K. (2004). “Effects of processing on zeta potential and cationic demand of kraft pulps,” *Colloids Surf. A* 246, 121-125.
- Chia, C. H., Zakaria, S., Nguyen, K. L., and Abdullah, M. (2008). “Utilisation of unbleached kenaf fibers for the preparation of magnetic paper,” *Ind. Crop. Prod.* 28(3), 333-339.
- Chirat, C., Lachenal, D., Mishra, S. P., and Pipon, G. (2008). “Including an ozone stage in bleaching sequence: Effect on pulp properties,” ABTCP 2008, The International Pulp and Paper Congress and Exhibition, Transamérica Expo Center, Sao Paulo, Brazil, 13-16, October 2008.

- Copur, Y., and Tozluoglu, A. (2008). "A comparison of kraft, PS, kraft-AQ and kraft-NaBH<sub>4</sub> pulps of Brutia pine," *Bioresour. Technol.* 99(5), 909-913.
- Cordeiro, N., Belgacem, M. N., Torres, I. C., and Mourad, J. C. V. P. (2004). "Chemical composition and pulping of banana pseudo-stems," *Ind. Crop. Prod.* 19, 147-154.
- Dutt, D., Upadhyaya, J. S., Tyagi, C. H., Kumar, A., and Lal, M. (2008). "Studies on *Ipomea carnea* and *Cannabis sativa* as an alternative pulp blend for softwood: An optimization of kraft delignification process," *Ind. Crop. Prod.* 28, 128-136.
- Eugenio, M. E., Alaejos, J., Diaz, M. J., Lopez, F., and Vidal, T. (2006). "Evaluation of Holm oak (*Quercus Ilex*) wood as alternative source for cellulose pulp," *Cellul. Chem. Technol.* 40(1-2), 53-61.
- Ezzat, S. (1974). "Leaves of date palm tree (*Phenix dactylifera*) as a technical feasible source of raw material for paper production," *Cellul. Chem. Technol.* 8, 627-634.
- Fiserova, M., Gigac, J., Majtnerova, A., and Szeiffova, G. (2006). "Evaluation of annual plants (*Amaranthus caudatus* L., *Atriplex hortensis* L., *Helianthus tuberosus* L.) for pulp production," *Cellul. Chem. Technol.* 40(6), 405-412.
- Fras, L., Laine, J., and Dolecek, V. (2004). "Determination of dissociable groups in natural and regenerated cellulose fibers by different titration methods," *J. App. Polym. Sci.* 92, 3186-3195.
- Gominho, J., Fernandez J., and Pereira H. (2001). "*Cynara cardunculus* L.: A new fibre crop for pulp and paper production," *Ind. Crop. Prod.* 13, 1-10.
- Goulet, M. T., and Stratton, R. A. (1990). "The effect of pulping, bleaching, and refining operations on the electrokinetic properties of wood fines," *Nord. Pulp Pap. Res. J.* 5(3), 118-125.
- Jimenez, L., and Lopez, F. (1990). "Characterization of Spanish agricultural residues with a view to obtaining cellulose pulp," *Tappi J.* 73(8), 173-176.
- Jiménez, L., Lopez, F., and Martinez, C. (1993). "Paper from sorghum stalks," *Holzforchung* 47, 529-533.
- Jiménez, L., Lopez, F., and Alaejos, J. (2006). "Materias primas alternativas para pastas de papel. Tipos, características, procesos y situación actual," *Ingeniería Química* 435(4), 76-90.
- Jimenez, L., Rodriguez, A., Perez, A, Moral, A., and Serrano, L. (2008). "Alternative raw materials and pulping process using clean technologies," *Ind. Crop. Prod.* 28(1), 11-16.
- Jimenez, L., Sanchez, I., and Lopez F. (1992). "Olive wood as a raw material for paper manufacture," *Tappi J.* 11, 89-91.
- Katz, S., Beatson R. P., and Scallan, A. M. (1984). "The determination of strong and weak acidic groups in sulfite pulps," *Svensk Papperstid.* 6, R48-R53.
- Khiari, R., Mhenni, M. F., Belgacem, M. N., and Mauret, E. (2010). "Chemical composition and pulping of date palm rachis and *Posidonia oceanica* – A comparison with other wood and non-wood fibre sources," *Bioresour. Technol.* 101, 775-780.
- Khristova, P., Kordsachia, O., Patt, R., and Karar, I. (2006). "Comparative alkaline pulping of two bamboo species from Sudan," *Cellul. Chem. Technol.* 40(5), 325-334.
- Laine, J., and Stenius, P. (1995). "Surface properties of bleached kraft pulp fibers and their comparison with the properties of the final paper products," 8<sup>th</sup> international Symposium on Wood and Pulping Chemistry (ISWPC), Helsinki, 1, 589-596.

- Mutje, P., Pelach, M. A., Carcia, J. C., Presta, S., Vilaseca, F., and Jimenez, L. (2006). "Comparison of cationic demand between olive and organosolv pulp and eucalyptus kraft pulp," *Process Biochem.* 41, 1602-1607
- Passas, R., Eymin, G., Voillot, C., Tarrajat, G., Caucal, G., and Khelifi, B. (2001). "MorFi: Analyseur morphologique des fibres," *Récents Progrès en Génie des Procédé* 15(78), 259-264.
- Sarwar, M. J., Nasima, C. D. A, and Russe, M. (2006). "Alkaline sulfite-anthraquinone-methanol (ASAM) pulping of corn stalks," *Cellul. Chem. Technol.* 40(7), 531-536.
- Shatalov, A. A., Quilho, T., and Pereira, H. (2001). "Arundo donax L. reed: New perspectives for pulping and bleaching. 1. Raw material characterization," *Tappi J.* 84(1), 96.
- Silvy, J., Romatier, G., and Chiodi, R. (1968). "Méthodes pratiques de contrôle du raffinage," *ATIP* 22(1), 31-53.
- Zomers, F. H. A., Gosselink, R. J. A, Dam, J. E. G. V., and Tjeerdsma, B. F. (1995). "Organosolv pulping and test paper characterization of fiber hemp." *Tappi J.* 78(5), 149-155.

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