Kraft Pulping of Date Palm Rachis from Egypt

Adel M. Kamal El-Dean[a], Ahmed A. Geies[a], A. H. Mostafa[b], A.H. Felet[b].

[a] Chemistry Department, Faculty of Science, Assiut University, Assiut, Egypt
[b] Quena Paper Industry Company ,Ques, Egypt

Abstract

Non-wood fiber is one of the major sources for the pulp and paper industry due to their abundance and cost-effectiveness. Kraft pulping of date palm rachis from date palm trees from Ques city in Egypt was carried out under different conditions, and pulps with variable yields and mechanical properties were obtained. The date palm rachis gave best yields and mechanical properties more than bagasse and less than soft wood. Kraft pulping of date palm rachis with 14%,17% and 20% cooking liquor (which contains 90% sodium hydroxide and 10% sodium sulfide) and 12% for bagasse. Bleaching of pulps was carried out in three stages with chlorine dioxide gas in two stages and hydrogen peroxide in the middle stage, rachis pulps were obtained of high brightness and strength properties suitable for use in writing and printing papers. Beating of bleached pulps was done for comparison between date palm rachis, bagasse and wood pulps.

Keywords: Date palm rachis; Bagasse; Papermaking; Chemical composition; Pulping with additives; Bleaching; Beating.

1. Introduction

Paper is a sheet constituted of cellulosic fibers, which are normally produced by separating wood cells using mechanical or chemical processes. The isolated fibers are subsequently re-arranged and randomly distributed into a sheet-like structure. Normally, the pulp and paper industries obtain the cellulose pulp from hardwood and softwood. However, an insufficient wood supply for growing demand has caused the industries to search for alternative fiber sources, such as non-wood fiber plants.

Cellulose pulp for papermaking purposes is obtained largely from wood (55%), non-wood plants (9%) and recovered paper (36%) *(Brawn et al., 2000).* Overall, 90–92% of the original raw materials used to produce paper consist of hardwood or softwood, and only 8–10% of non-wood materials *(Jiménez et al., 2006).*The latter include agricultural residues such as rice straw, wheat straw, bagasse, grasses etc.

Non-wood fibers generally contain less lignin than wood fibers. The most common is simple and requires low capital investment. It is a chemical process that is pulping process for non wood fibers called soda process. The soda pulping process is relatively environmental friendly and makes strengthened fibers for papermaking. Kraft (or sulfate) pulping represents the main cooking process, accounting for nearly 90% of the global chemical pulp production as presented by *Gavrilescu and Craciun (2012)*. The term kraft refers to the fact that kraft pulp exhibits good strength properties. The cooking liquor (usually called white liquor) contains sodium hydroxide and sodium sulfide as the active alkali, which reacts with lignin and promotes degradation of the lignin polymer into fragments that dissolve in the cooking liquor. Kraft pulping parameters include those factors influencing the delignification rate, pulping selectivity, pulp properties and specific consumption of raw materials and energy. *MacLeod (2007)* showed that kraft pulping variables can be divided into key factors and minor factors. The key factors exert a significant influence on pulping results and they are: active alkali charge, white liquor sulfidity, cooking temperature and cooking time, wood species and chip quality. The minor factors are: liquor-to-wood ratio, black liquor addition and the presence of sodium carbonate in the white liquor. kraft pulping is used in Egypt to produce pulps from bagasse.

Date Palm is another such an agricultural residue. The main date-producing countries of the world are Iraq, Saudi Arabia, Egypt, Iran, Algeria, Pakistan and the Sudan *(von Maydell, 1986*). According to The Food and Agriculture Organization of the United Nations (FAO) Egypt has been the world's largest producer of dates since 1974 and reports very high average yields as compared to other countries (*FAOSTAT 2010)*. There are 14 million trees, occupying 30,934 ha, which represents 6.32 % of the fruit cultivated area in Egypt (*FAO 2002)*, and date production of Egypt represents about 20 % of the total world production (*FAOSTAT 2009)*. Only few studies evaluated the potential use of date palm by-products for pulp and paper manufacturing and most of them are dedicated to the pulping of date palm leaves *(Ezzat, 1974; El Morsy, 1980; El Morsy et al., 1981).* To the best of our knowledge, *Khristova et al.(2005)* compared pulps produced from rachises and leaves. The ensuing pulps are characterized in terms of yield, kappa number, brightness, freeness, degree of polymerization (DP) and chemical composition (residual lignin, holocellulose, cellulose and extractives).

Finally, the physical and mechanical properties of hand sheets are presented and discussed.

The obtained results (chemical composition of pulps and physical properties of papers) are compared to others of wood and bagasse.

2. Materials

2.1. Raw materials

Date palm rachis used in this study were collected from date palm trees with 8 years ages from Ques city and collected in August 2018. These wastes were dried under natural conditions during September 2018 (average temperature = 25^{o} C). The date palm rachises of about (1-2) m length and (3–5) cm diameter were then cut into (3–7) cm pieces before pulping. The pieces were washed in order to eliminate sand and contaminants and then soaking for 4h before cooking to decrease chemical consumption.

2.2. Characterization of raw materials.

Chemical composition of date palm rachis was determined. The evaluation of extractive substances was carried out in different liquids according to common standards *Tappi test methods*, namely: cold and hot water *(T207 om-93)*, 1% sodium hydroxide solution *(T212 om-93)* and ethanol–benzene *(T204 om-88)*. Ash content *(T211 om-93)* was determined. The amounts of lignin, holocellulose and cellulose were also assessed by using the following respective standard methods: *(T222 om-88*) and *(T203 om-93).* As recommended by the various standards used, all the experiments were duplicated and the difference between the two values was within an experimental error of 5%.

2.3. Pulping

The delignification of date palm rachis was carried out in laboratory circulation digester with a total active alkali charge of 14%, 17% and 20% as (Na_2O) expressed in mixture of NaOH and Na_2S (9:1) based on w/w oven dried rachis with dry weight 500 gram and cooking time of 20 min. at constant temperatures of 160° C. All the experiments were carried out in laboratory circulation digester, in which the heating time to reach the constant temperature was 2 h. The liquor to solid ratio was 8:1. For comparison between date palm rachis and bagasse, which used in Quena Paper Industry Company as raw material for paper production, one sample from fresh depithed bagasse was cooked at the same conditions with a total alkali charge of 12%. After cooking, the obtained pulps were washed several times through a 325-mesh wire bottom until obtaining a clear filtrate. After that, the slurry of washed cooked mass was made and disintegrated using motorized rotary cutter according to *ISO (the International Organization for Standardization) (ISO 5263: 95)*. The disintegrated cooked mass was thoroughly washed and pressed to obtain air dried cooked pulp. After thorough washing, the air dried pulp was screened in laboratory screen using mesh of 0.15 mm slot width to remove the rejects (shives).

2.4. Bleaching

Unbleached pulps were bleached in three stages using DEpD sequence (D is chlorine dioxide in acidic medium and Ep alkaline extraction with addition of hydrogen peroxide). Bleaching experiments were carried out in a high precision water bath. 100 gram of unbleached pulp on oven dried basis was packed into transparent polyethylene bags and put into water bath maintaining the conditions as shown in Table 1 to obtain the bleached pulp with above or around 85% ISO brightness. Prior to bleaching, the pulp was thoroughly mixed with water mechanically and then filtered using a vacuum pump to get pulp pads.

2.5. Beating

Beating of bleached pulps were carried out in laboratory PFI mill *(T 248 cm-85)* to evaluate pulp quality for papermaking. Laboratory beating of the pulp is a widely accepted method of simulating commercial refining practices. Physical testing of laboratory-beaten pulps provides significant data that aid in determining the ultimate performance of pulp when converted to paper. Beating of all date palm pulps, bagasse pulp and wood pulp were carried out to obtain Freeness of Pulp CSF 300.

2.6. Pulp and paper characterization

After cooking, pulps characterized in terms of yield, kappa number, brightness, freeness, shives and degrees of polymerization (DP). The cooking yield was calculated as the ratio of the weight of oven dried material after washing to that of initial raw material *(ISO 638:78).* kappa number to determine of the relative hardness, bleach ability, or degree of delignification of pulp *(T 236 cm-85).* Determination of the brightness of white, near-white, and naturally colored pulp (or directional reflectance) at effective wavelength of the spectral band is 457 nm *(ISO 2470:99).* The pulp drain ability was evaluated by measuring the Freeness of Pulp CSF (Canadian standard Freeness) *(T227 om-94).* Shives or uncooked materials were determined as the ratio between the weights of oven dried material before and after screening by using mesh of 0.25 mm slot width *(T275 sp-98).* The viscosity of pulp (η in mPa s) dissolved in the cupriethylenediamine solution was determined according to Tappi standard *(T230 om-99)*. These values were then converted into degrees of polymerization (DP) using to the following relation proposed by *Sihtola et al.(1963)*:

$$\mathbf{DP = [0.75\ (954\ Log_{10}\ \eta - 325)]^{1.105}}$$

The unbeaten screened pulps, bleached pulps and beaten pulps suspensions were diluted to 2 gram / L then, conventional hand sheets with a basis weight of 60 gram/m^2 were prepared on a Tappi sheet machine manual *(T 205 sp-95).* Prior to testing, the hand sheets were conditioned (23°± 1° C, 50%±2% relative humidity *(T 402 om-93)* and structural and mechanical properties were determined by measuring basis weight, thickness and bulk, as well as the tensile, burst and tear strength according to *(T 220 sp-96), (T 411 om-89), (T 494 om-88), (T 403 om-91)* and *(T 414 om-88).* Permeability and opacity of all pulps sheets were determined *(IS0 5636-3: 92),(ISO 2471: 98).*

2. Results and discussion

3.1. Characterization of the raw material

The chemical composition of our date palm rachises and of other works are reported in Table 1. Table 2 summarizes chemical compositions collected from literature for various cellulosic biomasses such as hardwood and softwood, date palm rachises and bagasse.

Table 1 Chemical composition of date palm rachises

Amounts in % (w/w with respect to oven dried raw material)	This work	El Morsy (1980)	Khristova et al. (2005)	Bendahou et al. (2007)	Khiari et al. (2010)
Cold water solubility	6.1	n.d.	n.d.	n.d.	5
Hot water solubility	9.8	n.d.	8.7	n.d.	8.1
1% NaOH solubility	28	n.d.	25.6	n.d.	20.8
Alcohol - benzene extractives	2.7	n.d.	12.8[a]	4[b]	6.3[b]
Ash	4.7	3.4	5.6	2.5	5
Lignin	26.2[c]	25.8	23.8[d]	14	27.2[c]
Holocellulose	72.6	n.d.	n.d.	72	74.8

[a] In ethanol–cyclohexane **n.d.** not done

[b] Ethanol–toluene extractives

[c] Klason lignin.

[d] Residual lignin (Klason and soluble lignin).

Table 2 Chemical composition of date palm rachises, bagasse and woods

Amounts in % (w/w with respect to oven dried raw material)	CW	HW	NS	AB	Ash	LG	HC	Reference
Date palm rachises	6.1	9.8	28[b]	2.7	4.7	26.2	72.6	*This work*
Bagasse	4.5	7.2	36.4[b]	3.7	2.6	20.2	75.1	*This work*
Bagasse	6.4	12.2	38.2[a]	4.1	5.7	19.7	70	*Lal et al.(2012)*
Eucalyptus globules	—	2.84	12.42[b]	1.15	0.57	27.68	80.47	*Ferrer et al.(2013)*
Eucalyptus grandis	2.19	4.59	17.9[a]	2.89	0.72	27.1	72.8	*Sharma et al. (2011)*
Eucalyptus tereticornis	1.87	4.87	18.8[a]	3.02	0.45	28.8	71.6	*Sharma et al. (2011)*
Pine needle	2.98	7.53	47.42[a]	5.81	4.45	43.24	51.62	*Lal et al. (2013)*
Holm Oak (Quercus ilex)	—	—	—	—	—	16.3	71.2	*Eugenio et al. (2006)*
Brutia pine	2.2	2.8	16.1[b]	1.94	0.4	26.1	73.5	*Copur and Tozluoglu (2008)*
Olive wood	15.5	17	30[b]	10.4	1.4	15.64	65.83	*Jimenez et al. (1990,1992, 2008)*

CW: cold water solubility; **AB**: alcohol benzene solubility; **HW**: hot water solubility
NS: NaOH solubility; **HC**: holocellulose; **LG**: lignin.
a 0.1 N NaOH solubility; **b** 1% NaOH solubility

The comparison with the present work leads to several comments concerning the amount of extractives. Thus, in cold and hot water, the quantity of extractives for date palm rachis are higher than those found in hardwood and softwood but comparable to the amounts usually encountered in non-wood sources. The 1% NaOH extractives (28% for date palm rachis) are similar to those of wood sources, i.e. less than 20%, but are lower than those of annual plants. Finally, the amount of extractives in ethanol–benzene for the raw materials under investigation is relatively high, although in the same order of magnitude as those observed for other annual plants or agricultural crops. Considering the structural components, residual lignin for date palm rachis was also found to be quite high (26.2%), when compared to typical amounts encountered in annual plants, non-wood and hardwood sources which are close to 20%. Lignin content of date palm rachis is then close to that of softwood. In the same way, the amounts of holocellulose for the date palm rachis were similar to those found in wood and non-wood plants. Ash for the date palm rachis was high and more than wood. Degrees of polymerization (DP) for date palm rachis fibers was 1200 but 1090 for bagasse fibers. For comparison, the length of softwood and hardwood fibers is 2–3 mm and 1–2 mm respectively, The fibers from the date palm rachis were in the range of hardwood fibers, with short fiber length (1.3 mm), with medium thin walls (3.5 lm), narrow lumen (9.6 lm) and fiber width (16.6 lm) (*Khristova et al. 2005)*.

3.2. Date palm rachis pulping

Kraft pulping of date palm rachis (Table 3) with chemical charge of 14–20% of active alkali (90% sodium hydroxide and 10% sodium sulfide) and maximum temperature of 160° C for 20 min and kraft pulping of bagasse with chemical charge of 12% of active alkali at the same conditions gave kappa numbers in the range of (19.5-30.5)for date palm rachis and 12.3 for bagasse at screened yields between(38.9- 39.7) % for date palm rachis and 47.3% for bagasse. For date palm rachis the increase of the alkali charge from 14% to 20% at 160°C resulted in a slightly lower screened yield, but the pulp had higher viscosity, better brightness and lower kappa number. In the initial period of cooking huge amounts of alkali are consumed for the neutralization of acids derived from the polysaccharides and for neutralizing lignin degradation products.

Nevertheless, too high alkali concentrations must be avoided, otherwise over-proportional degradation and dissolution of hemicelluloses and cellulose might take place, resulting in reduced yield and viscosity. Brightness of date palm rachis pulps was from (25.5-36.5) less than bagasse pulp (47) due to high kappa no for date palm rachis pulps (19.5-30.5) than bagasse(12.3), while DP for date palm rachis pulps (994-1085) more than bagasse (832).

The mechanical properties (Table 3) indicated the strength of date palm rachis pulps and bagasse pulp. Tensile strength and Burst strength of date palm rachis pulps less than those of bagasse pulp but tear strength was higher than bagasse pulp. Air presence for date palm rachis were high as the same of wood pulp more than 5000 ml/min but for bagasse was 440 ml/min. The mechanical properties indicated the superior tear strength for date palm rachis (10.4-11.5) pulps to those of bagasse pulp (6.2). Tensile and burst strength for date palm rachis were less than of bagasse.

Table 3 Date palm rachis and bagasse pulping conditions and unbleached pulps evaluation

Pulping process	Date palm rachis			Bagasse
	A_1	A_2	A_3	A_4
Pulping conditions				
Active alkali as Na_2O %	14	17	20	12
Dry sample weight, gram	500	500	500	500
Liquor to oven dry sample ratio	8	8	8	8
Maximum temperature, °C	160	160	160	160
Time for pulping, min	120	120	120	120
Time at max. temperature, min	20	20	20	20
Pulping results				
Total yield, %	45.8	44.6	43.5	50.5
Screnned yield, %	39.7	38.9	39.3	47.3
Rejects, %	6.1	5.7	4.2	3.2
Kappa number	30.5	25.8	19.5	12.3
ISO brightness, %	25.5	31.2	36.5	47
Opacity % ptg	97.7	96.9	96.4	87.5
SC Coefficient m^2/kg	35.5	37.2	40.1	30.8
Yellowness %	42.9	38	33	25.5
Air permeance ml/min	›5000	›5000	›5000	440
DP(degrees of polymerization)	1085	1003	994	832
Pulp evaluation				
Initial pulp freeness CSF	680	660	650	510
Tensile index, Nm/g	33.8	36.6	39.4	50.3
Tear index, mNm^2/g	11.5	10.9	10.4	6.2
Burst index, $kPam^2$/g	2.6	2.7	3.1	3.8

3.3. Bleaching of pulps

Unbleached pulps were bleached in three stages using DEpD sequence (Table 4). D is chlorine dioxide and Ep alkaline extraction with addition of hydrogen peroxide. By this sequence brightness of date palm rachis pulps increases to (85.1-86.5) with good for that mechanical properties but brightness of bagasse pulp was 87.5. Yields of pulps after bleaching (93.8-95) % for date palm rachis and 95.2% for bagasse. DP(degrees of polymerization) were high for date palm rachis pulps (880-910) and more than for bagasse (770).

Table 4 Bleaching results and bleached pulp evaluation from date palm rachis and bagasse

Pulping process	Date palm rachis			Bagasse
	A_1	A_2	A_3	A_4
First stage bleaching				
chlorine dioxide				
concentration g/l	5	5	5	5
pH	2	2	2	2
Temperature, °C	75	75	75	75
Time, min	120	120	120	120
Addition kg/ton	20	20	20	20
Dry sample weight, gram	100	100	100	100
Consistency of pulp %	6	6	6	6
Second stage bleaching				
Hydrogen peroxide/soda				
concentration g/l / %	195/17.5	195/17.5	195/17.5	195/17.5
pH	11	11	11	11
Temperature, °C	80	80	80	80
Time, min	120	120	120	120
Addition kg/ton	7/20	7/20	7/20	7/20
Consistency of pulp %	6	6	6	6
Third stage bleaching				
chlorine dioxide				
concentration g/l	5	5	5	5
pH	3.5	3.5	3.5	2
Temperature, °C	75	75	75	75
Time, min	120	120	120	120
Addition kg/ton	5	5	5	5
Consistency of pulp %	6	6	6	6
Pulping results				
Total yield, %	95	94.6	93.8	95.2
ISO brightness, %	85.1	85.7	86.5	87.5
Opacity % ptg	84.9	83.2	82.2	77.1
SC Coefficient m^2/kg	40.7	44	44.6	32.2
Yellowness %	6.2	5.4	4.5	3.1
Air permeance ml/min	›5000	›5000	›5000	290
DP(degrees of polymerization)	910	905	880	770
Pulp evaluation				
Initial pulp freeness CSF	590	580	550	510
Tensile index, Nm/g	51.1	52.3	54	54.5
Tear index, mNm^2/ g	10.3	9.7	9.6	6.1
Burst index, $kPam^2$/ g	3.3	3.3	3.6	4.3

3.4. Beating of pulps

Beating of date palm pulp (A3), bagasse pulp (A4) were carried out in laboratory PFI mill to obtain freeness of Pulp CSF 300 and summarized in (Table 5). Comparison between date palm rachis pulps, bagasse pulp and wood pulps (Reference to Quena Paper Industry Company results) were listed. The results show that the fiber of date palm rachis is close to that of hardwoods and better than of bagasse.

Table 5 Beating of bleached pulps

pulps	Date palm rachis	bagasse	Soft wood*	Hard wood* *(Eucalyptus)*
Freeness CSF	550 300	510 300	700 300	540 300
Tensile index, Nm/g	54 86.1	54.5 62.2	26.2 108.4	20.2 60.1
Tear index, mNm²/ g	9.6 11.7	6.1 5.86	16.3 10.7	3.5 8.1
Burst index, kPam² /g	3.6 4.5	4.3 4.6	2.1 8.5	1 2.8
Air permeance ml/min	›5000 550	290 40	›5000 120	›5000 845

* *Reference to Quena Paper Industry Company results*

4. Conclusions

The fiber of date palm rachis is close to that of hardwoods. The carbohydrate and lignin contents indicated acceptable yields from the date palm rachis with high amount of alkali at 160°C. Bleaching the rachis pulps with DEpD sequence method resulted in bleached pulps of high brightness and strength properties then pulp suitable for writing and printing papers. Strength properties for date palm rachis pulps were higher than that of bagasse pulp which used in Egypt in paper industry.

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الملخص العربي

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عادل محمد كمال الدين[1] ، أحمد عبده جعيص[1]، عاطف حسن مصطفي[2]، عبده حماده فليت[2]

قسم الكيمياء – كليه العلوم – جامعه أسيوط

شركه قنا لصناعه الورق

تعتبرالألياف غيرالخشبية أحد المصادرالرئيسية لصناعة اللب والورق نظرًا لوفرة هذه المنتجات وفعاليتها من حيث التكلفة. حيث تم إجراء عملية طبخ لجريد نخيل البلح المجمع من مدينه قوص في مصربطريقه الكرافت في ظروف مختلفة، وتم الحصول على اللب ذو الحصيلة والخواص الميكانيكية المختلفه. وأعطى جريد نخيل البلح حصيلة جيده وخواص ميكانيكية أفضل من قش قصب السكر وأقل من الخشب. تم طبخ جريد النخيل بسائل الطبخ الذي يحتوي على90٪ هيدروكسيد الصوديوم و 10٪ كبريتيد الصوديوم بنسب 14٪ و17٪ و20٪ وطبخ قش القصب بنسبه 12٪ سائل طبخ. تم تبيض اللب على ثلاث مراحل بغاز ثاني أكسيد الكلور على مرحلتين وفوق اكسيد الهيدروجين في المرحلة الوسطى ، وتم الحصول على بياض عالي للب المصنوع من جريد نخيل البلح وخصائص ميكانيكيه عاليه تؤكد صلاحيته للاستخدام فى صناعه ورق الكتابة والطباعة. وقد تم طحن اللب المبيض المصنوع من جريد النخيل وقش القصب للمقارنة بين بينهم وبين اللب المصنوع من الخشب.