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Original Article

Pulping and paper properties of Palmyra palm fruit fibers

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Abstract

Palmyra palm fruit fibers have the properties to be used as an alternative raw material of cellulosic pulps for papermaking. Acid and alkali pulping were investigated by using nitric acid and caustic soda on a laboratory scale, with the purpose of producing printing or writing grade pulp. The chemical composition of fiber strands from palmyra palm fruits were examined, such as holocellulose, cellulose, pentosan, lignin and extractives. The yields of acid and soda pulps were below 40%. The main physical and mechanical properties of hand sheets produced from acid and soda processes were evaluated on 80 g/m^2 test sheets as functions of the following parameters: tensile index, tear index, and brightness. The mechanical properties of soda pulps were developed by twin-roll press while it was not necessary to fibrillate acidic pulps through the beating step. The soda pulp sheets presented a lower brightness than that of acidic pulp sheets. The mechanical and physical properties of the acidic and alkaline pulps verified that they were of an acceptable quality for papermaking.

Keywords: Palmyra palm, pulping, papermaking, nitric acid, caustic soda

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. Non-wood plant materials include agricultural residues, natural growing plants, annual plants and crop fibers such as pineapple crown leaves, rice straw, kenaf and bagasse (Khristova *et al.*, 2002; Tran, 2006; Huang *et al.*, 2007; Rodriguez *et al.*, 2008). Non-wood fibers generally contain less lignin than wood fibers. The most common pulping process for non-wood fibers is the soda process. The soda pulping process is relatively simple and requires low capital investment. It is a chemical process that is environmental friendly and makes strengthened fibers for papermaking.

The chemical processes of pulp making can be divided into two main types the alkaline process and the acidic process. Conventional acid pulping is done by the sulfite process, which produces large amounts of polluting emissions. For this reason there is a need for new technologies of acidic pulping. These require the development of lower investment processes to obtain quality pulp products at lower costs, using non-sulfur formulas and low energy consumption, and using alternative materials such as agricultural residues. Acetocell (acetic acid-water), Milox (formic acid-hydrogen peroxide), Formacell (acetic acid-water-formic acid) and Acetosolv (HCl-water-acetic acid) are new acid pulping processes being used for delignification technologies (Poppiuslevlin *et al.*, 1991; Neumann and Balser, 1993; Saake *et al.*, 1995; Lehnen *et al.*, 2001; Jahan *et al.*, 2007; Ligero *et al.*, 2007; Sahin and Young, 2008). Nitric acid is another chemical solution used to determine cellulose content in plants. It reacts rapidly with and dissolves lignin to yield cellulose preparation in the Kurschner–Hoffner

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method (Browning, 1967). Nitric acid-water is a new pulping process, which modifies the Kurschner–Hoffner method to make paper pulps. This present research studied the pulping of non-wood plant materials, Palmyra palm fruit fibers, by using caustic soda and nitric acid-water pulping processes.

Palmyra or toddy palm (*Borassus flabellifer* Linn.) is a native plant of the Sathing-Pra Peninsula in southern Thailand. It is a plant of the *Palmae* family similar to oil palm and coconut. Palmyra palm is a 'miracle' plant due to the once wide utilization of most of its parts, such as the trunk, foliage, husk, nut, and flesh. The trunk can be used for furniture and handicrafts. Dried leaves and the flexible sticks in the frond are woven to make some crafts. Palm nectar is used to brew for wine and vinegar and to make sugar. The flesh inside the nut is edible. A natural food coloring substance can be extracted from the husk or mesocarp. The mesocarp of Palmyra palm fruits has been another fibrous source used as a raw material for pulp and papermaking. No references to the pulping of palmyra palm fruit fiber appear to exist in the scientific literature. The objective of this paper is to investigate the potential of another byproduct, husk or mesocarp, as a raw material for pulp and paper making. These experiments in this study explored the use of nitric acid and a soda pulping process, which produces no sulfur compounds and is well defined in terms of technology.

2. Materials and Methods

2.1 Raw materials

The ripened Palmyra palm fruits were collected from southern Thailand (Sathing-Pra Peninsula). They were cleaned and peeled after collection. The ripened flesh was pressed to separate the fiber from the flour paste. The pressed fibers were washed with water for the removal of the flour attached. All these samples were dried and the moisture content was determined for the subsequent experiment. They were stored in plastic bags in cold storage. The chemical composition of the samples was determined in accordance with the respective TAPPI standards ash content (T211-om-93), cellulose content (T203-om-93), lignin content (T222-om-98), hot water solubles (T207 om-88), 1% sodium hydroxide solubles (T212 om-88), ethanol-benzene extractables (T204 om-88) and pentosan (T223 om-84). Holocellulose was analyzed according to the peracetic method (Cordeiro *et al*., 2004).

2.2 Pulping

The pressed fibers were pulped through the alkali and acidic processes. They were cooked at a 10:1 liquid-to-wood ratio in a digester autoclave. Cooking liquor for the alkali process was prepared by dissolving sodium hydroxide pellets in water and determining the concentration. A nitric solution for the acidic process was prepared by dissolving nitric acid (HNO_3) in water and determining the concentration. The cooking conditions in the alkali and acidic pulping are indicated in Table 1. The partially delignified pulps obtained were washed with water to neutralize the reaction. The pulps were mechanically disintegrated and screened on a flat screen with 0.25 mm slits in order to remove uncooked particles. Then the moisten soda pulps were pressurized with a twin-roll mill for fibrillation until the freeness of the fiber of the soda pulps was similar to unbeaten acid pulp (CSF 150 ml). The screened pulps were centrifuged and dried at 105°C in a hot air oven. The oven-dry weight of the pulps was determined as the screened pulp yield. The kappa number of screened pulps was examined according to the TAPPI standard (T236-cm-85). Fiber lengths were determined by a Kajaani FS-200 instrument.

2.3 Papermaking

Hand sheets were prepared from the dried pulp. The laboratory sheets were prepared in accordance with the Tappi Test Method T205 om-88, with a final grammage (mass of the product per unit area) of 80 g/m^2. The screened pulps were disintegrated and suspended in water to a dry content of 2.04 g/l. The pulp suspension was mixed with 8 l of water in the hand sheet mould for 10 seconds at 2 atm air pressure. The white water was emptied through the wire screen. The sheet was covered with three blotting papers and a pressure of 1 kPa was applied for 30 seconds to remove excess water. The sheet was thereafter removed from the wire screen, covered with a blotting paper and metal plate, and pressed at 500 kPa for 10 minutes to remove further excess water. Finally, the sheet was dried and conditioned at 25ºC and 50% relative humidity (RH) for 48 hours prior to testing. Ten hand sheets were used for testing the tensile index (Tappi T494 om-88), tear index (T414 om-88) and brightness (T452 om-92).

3. Results and Discussion

3.1 Chemical composition of Palmyra palm fruit fibers

The chemical composition of Palmyra palm fruit fibers is shown in Table 2. The ash content of the fibers is 0.64%. This is lower than those of non-wood fibers such as rice straw with 9.2% (Rodriguez *et al*., 2008), jute leaf with 8.8% (Basak *et al*., 1996), kenaf with 4% (Khristova *et al*., 2002) and bagasse with 1.5% (Khristova *et al*., 2006). Chemically, Palmyra palm fruit fibers are rich in holocellulose (68.52%)

Table 1. Pulping process variables

Item	Alkali pulping	Acidic pulping
Concentration (%)	5, 8, 10	5, 8, 10
Cooking time (min)	45, 60, 75	15, 30, 45
Cooking Temperature (°C)	150	100
Liquor to wood ratio	10	10

Table 2. Some chemical compositions of Palmyra palm fruit fibers

Chemical composition	Percentage based on bone dry weight
Ash	0.64±0.06
Holocellulose	68.52±1.38
Cellulose	37.01±0.92
Lignin	18.54±0.58
Pentosan	28.51±1.10
1% NaOH solubility	44.68±2.33
Hot water	21.36±2.30
Ethanol-benzene	4.26±0.58

and cellulose (37.01%). These are important parameters in determining the suitability of a raw material for papermaking. Lower lignin content (18.54%) is normally found in non-wood fibers. The pentosan content in Palmyra palm fruit fibers is 28.51%. This indicates the amount of hemicellulose, which contributes to the strength of paper pulp. Thus a higher pentosan contents is desirable.

The Palmyra palm fruit fibers contain higher solubilities in 1% sodium hydroxide and hot water. The high solubility in 1% sodium hydroxide indicates the extent of fiber degradation during the alkali pulping process and thus the screened yield of the chemical pulps would be low. The percentage of hot water soluble substances in Palmyra palm fruit fibers is 21.36%. This suggests that the higher content of sugars, coloring matters, starch and proteins could consume pulping reagents. The soluble extractives in ethanol-benzene show the lower level of extractable content in Palmyra palm fruit fibers, such as low molecular weight carbohydrates, salts, waxes, fats, and resinous substances.

3.2 Pulping process

The key performances from the optimal pulping are the screened pulp yield and quality of fiber sheets. The chemical pulp yields of the cooking process are shown in Table 3. It can be seen that the yield is highly dependent on the charge concentration, cooking time, cooking temperature, and type of pulping process. An increase in alkaline or acidic charge accelerates delignification as indicated in lower kappa number and a decrease in the pulp yield due to carbohydrate degradation. Alen (2000) has established that during the alkaline pulping of lignocellulosics, alkali catalysed reactions are mainly responsible for the loss of yield. This involves a step-like elimination of monosaccharide moieties from carbohydrates. This starts at their reducing ends and continues along the polymeric chain until an alkali stable end group is formed by a competing reaction (the stopping reaction).

For the acidic process, the nitric acid process results in a lower pulp yield than the soda process due to the higher efficiency of acid on delignification and carbohydrate hydrolysis. The optimum condition for alkaline pulping is 10% sodium hydroxide, a liquor ratio of 10:1 (liquor:fibers) at 150°C for 75 minutes (10/75/150). The screened pulp yield of this condition is the highest (40.7 % based on dried weight of pulp). In addition, the optimum conditions of acidic pulping are 8% nitric acid and a liquor ratio of 10:1 at 100°C for 30 minutes (8/30/100). The screened pulp yield of the optimum condition is 33.5% dried weight of pulp. Table 3 shows that the values of the kappa number are very low for all treatments, from both processes (acidic and soda processes). They never reached a value above 20, which suggests they have a strong tendency to whiten and to be more easily bleached. The kappa number would allow the pulp to be bleached without significant losses in term of cellulose fiber quality or length. The lowest values for the kappa number are for the acidic process conditions of 10% nitric acid and a temperature of 100°C for 45 minutes (10/45/100). The pulp sheets made under this condition show the highest brightness value of 42.36%. A higher brightness for paper from palmyra palm pulp is obtained from the acidic process. The pulp brightness increases with an increasing chemical charge, implying that some extractable alkali or acid components in the raw material are responsible for the high light absorption of the pulp.

The mechanical properties of pulp sheets are reported in Table 3. Tensile index and tear index are probably the most used ones for the direct measurement of the paper strength potential. The pulp sheets made of beaten soda pulp with 10% sodium hydroxide at a cooking temperature of 150°C for 75 minutes (10/75/150) show the highest tensile index. They have the tensile index and tear index of 13.8 Nm/g and 1.12 mNm^2/g, respectively. However, both the properties of unbeaten pulp sheets made of acidic pulp with 8% nitric acid at cooking temperature of 100°C for 30 minutes (8/30/100) are not significantly different to the beaten soda pulp (10/75/150). Its tensile strength at a given tear index is also higher, suggesting that its interfiber bonding strength is highest among the pulp studied. According to Gurnagul *et al.* (2006), fiber strength is directly proportional to α-cellulose content for fibers of low fibril angle and pulped by processes that do not degrade cellulose. The higher concentration of acidic charges has brought about the higher degradation of cellulose. On the other hand, the low concentration of chemical charges in cooking liquor (that is 5% sodium hydroxide or nitric acid) provides incompletely delignified pulp. This decreases the interfiber bonding so that its tensile index and tear index would be low.

Another physical property which has a strong influence on the papermaking potential of pulps is the dimensions of pulp fiber, particularly the length. Fiber dimensions, such as length-weighted average, weight-weighted average, and fiber coarseness, are shown in Table 4. The length-weighted averages of soda pulp and acidic pulp are 1.07 and 0.58 mm, respectively. The weight-weighted averages of soda pulp and acidic pulp are 2.11 and 1.01 mm respectively. Pulp fiber

Table 3. Physico-chemical characterization of the acidic and soda processes of Palmyra palm fruit fibers.

	Yield (%)	Kappa no.	Tensile index (Nm/g)	Tear index(mNm2/g)	Brightness(%)
Soda Process					
5 / 45 / 150	16.8a	19.8f	9.2a	0.62a	16.68a
5 / 60 / 150	18.5ab	20.1f	11.8c	0.80bc	17.30a
5 / 75 / 150	21.5c	19.7f	12.4d	0.85c	17.61a
8 / 45 / 150	18.9b	16.2d	12.0c	0.78bc	20.34b
8 / 60 / 150	24.4e	18.5e	12.7d	0.85c	21.70bc
8 / 75 / 150	27.2f	18.7e	13.2ef	0.91d	21.83bc
10 / 45 / 150	28.4f	15.7d	13.0e	0.88cd	22.26c
10 / 60 / 150	33.9h	16.2d	13.5f	1.04e	22.57c
10 / 75 / 150	40.7i	15.6d	13.8f	1.12e	23.05c
Acidic Process					
5 / 15 / 100	17.5a	12.5c	12.2cd	0.73b	20.29b
5 / 30 / 100	19.2a	11.3b	12.0c	0.82bc	22.65c
5 / 45 / 100	22.6c	10.8b	12.5d	0.80b	24.31d
8 / 15 / 100	27.3f	11.3b	13.6f	0.89cd	34.32e
8 / 30 / 100	33.5g	11.2b	13.7f	1.10e	38.85f
8 / 45 / 100	30.1f	10.5b	13.2ef	0.80bc	39.76f
10 / 15 / 100	28.4f	11.0b	13.4f	0.85c	39.54f
10 / 30 / 100	28.3f	10.5b	12.6d	0.85c	41.99g
10 / 45 / 100	22.8c	9.7a	11.0b	0.78bc	42.36g

Values within columns followed by a different letter are significantly different ($P < 0.05$)

Table 4. Fiber characterization of acidic and soda pulps

	Value
Soda pulp	
Length-weighted average (mm)	1.07±0.02
Weight-weighted average (mm)	2.11±0.05
Coarseness (mg/m)	0.36±0.01
Acidic pulp	
Length-weighted average (mm)	0.58±0.01
Weight-weighted average (mm)	1.01±0.03
Coarseness (mg/m)	0.23±0.01

Length-weighted average ($Lw = Rn_iL_i^2/Rn_iL_i$);
weight-weighted average ($Lww = RniL_i^3/Rn_iL_i^2$).

properties could be a significant asset in controlling the papermaking potential of pulp. Depending on the physical properties, the weightweighted property distribution can be significantly different from the length-weighted distribution. In papermaking, fibers are used by weight. Therefore, it makes more sense to use weight-weighted average and distributions instead of length-weighted average.

The fiber coarseness of soda pulp and acidic pulp (shown in Table 4) are 0.36 and 0.23 mg/m respectively. Fiber coarseness is defined as the weight per unit length. The coarser fibers have thicker walls and lower specific surface area. The fiber coarseness influences virtually all pulp properties such as drainage, wet-web strength and optical properties of the dry sheets. Fiber coarseness and fiber length are very important factors in tear and tensile resistance. Both these properties increase with an increase in fiber length and fiber coarseness. Coarse and long fibers tend to produce higher tear and tensile resistance than fine fibers do. The fiber length presented as population distributions are shown in Figure 1.

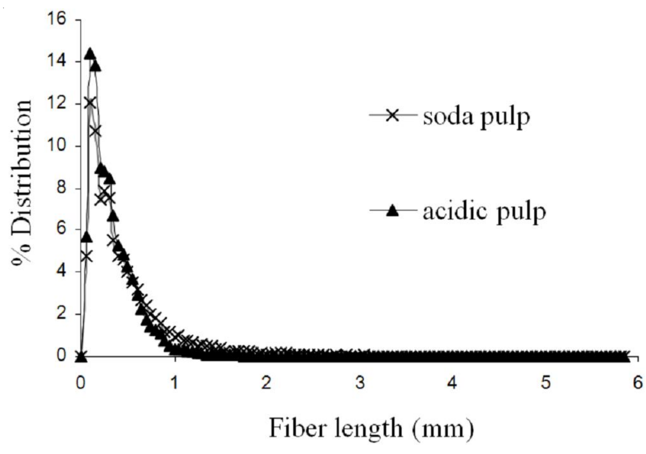

Figure 1. Population distribution of fiber length of soda pulp and acidic pulp.

The fiber length distribution curve in Figure 1 shows that the distributions of unbeaten acidic pulp and beaten soda pulp are not different. As the population distribution is strongly influenced by fines and short fibers, it is clear that either length-weighted or weight-weighted fiber length distributions have more practical use. Less desirable fibers could then be separated and then processed to change their dimensions and the properties of the fiber material, thus making them more suitable for papermaking.

4. Conclusions

The mesocarp of Palmyra palm fruits is an effective alternative source of cellulose for producing paper pulp. It could be cooked by either the soda process or the acidic process. However higher yields and acceptable properties are the requirements of chemical pulping. Unbeaten acidic pulp from Palmyra palm fruit fiber exhibits better kappa number and brightness than beaten soda pulp; this means that acidic pulp would be easily bleached. With its low chemical charges, unbeaten acidic pulp provides the paper sheets with acceptable strength. It has good tensile and tear indexes, with a good yield in terms of potential savings in energy and chemicals in the pulping process. Acidic pulp could be cooked in less cooking time and at a lower temperature or could be used to produce pulp at a lower chemical charge. However, soda pulping represents a more practical application because hand sheets can be produced normally. The soda pulp hand sheets possess relatively higher strength, especially the tear and tensile strengths, although the brightness of the soda pulp hand sheets remains in the lower range of around 23%.

The major mechanism that is responsible for the improving strength in soda pulps is probably the increase of interfiber bonding through the beating process (Rodriguez *et al*., 2008). The results of this study suggest that Palmyra palm fruit fibers constitute an effective alternative raw material for paper pulp. They provide pulp and paper sheets with acceptable properties and thus allow an agricultural residue from a major economic activity to be profitably exploited.

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