Preparation and Properties of Retted Kenaf Bast Fiber Pulp and Evaluation as Substitute for Manila Hemp Pulp

Abdolreza NEZAMOLESLAMI* , Kyoji SUZUKI* , Takashi KADOYA**

Using a retted Chinese kenaf bast fiber (CBF) as raw material, various pulps were prepared by using three cooking methods, i.e. soda-AQ pulping, kraft pulping and non-pressurized soda pulping. The capabilities of these pulps, as substitutes for manila hemp pulp which is used mainly as specialty paper applications, were investigated.

The soda-AQ and kraft pulps of retted CBF had lower Kappa number and ash content with greater pulp yield than those of unretted Japanese kenaf bast fiber (JBF). The CBF pulps prepared by the three cooking methods were bleached to about 80% Hunter brightness, by a two-stage hypochlorite process, and the relationship between strength properties and CSF was studied. Tensile index and burst index of those pulps at CSF 400ml were in the following order: non-pressurized soda> soda-AQ> kraft. The strengths of the non-pressurized soda pulp were superior to those of commercial manila hemp pulp. The folding endurance of the three pulps were lower than that of manila hemp pulp, while, that of the non-pressurized soda pulp became equal by 300ml CSF. Therefore, the non-pressurized soda pulp from retted CBF was found to be hopeful as substitute for manila hemp pulp.

Keywords : Kenaf, Retted bast fiber, Non-pressurized soda cooking, Soda-AQ cooking, Kraft cooking, Strength properties, Manila hemp pulp

1. Introduction

The utilization of non-wood plant fibers instead of wood fibers in the production of different kinds of pulps and papers is growing in many developing and developed countries. Since 1970 to present time the non-wood plant fiber pulping capacity has increased, on a global basis, two or three times as fast as the capacity for producing papermaking wood pulp[1].

Kenaf (*Hibiscus cannabinus* L.), a fast growing warm-season annual plant in the family of *Malvaceae*, has attracted attentions as a potential non-wood raw material for the pulp and papermaking over the last decade[1),2)]. Kenaf reaches 4-5m high with a basal diameter of 25-35mm during a growth cycle of 5-7months. The stem of kenaf is composed of two main fiber types ," bast fiber and inner woody core fiber. The average length of the bast fiber is 3-4mm, and that of the core fiber is 0.5-0.7mm[4]. The core fibers generally are short and wide with thin wall while the bast fibers are long and stiff. Generally, on an oven-dry basis, the bast part accounts for about

*Department of Forest Resources Sciences, Faculty of Agriculture Shizuoka University, 836 Ohya, Shizuoka-shi, Shizuoka, 422 Japan
**Kanagawa University, 2946 Tsuchiya, Hiratsuka-shi, Kanagawa, 259-12 Japan

1/3 of the stem mass and the woody core part for the remainder. To date, the whole kenaf stem has been commercially pulped by kraft method, however, most of the conventional and some non-conventional methods have given the required properties[5)-7)].

Because the kenaf bast and core fibers have different nature and structure, they show different behavior during pulping processes[3),6)] . Nowadays, researches on the kenaf pulping are mainly directed on the pulp and paper characteristics of core or bast part of kenaf in a separate system of prep aration.

Recently some mechanical methods for separating the kenaf parts have been described and practically performed[3)]. However, in some of the developing countries due to less expensive wages, kenaf fibers are separated manually rather than mechanically at present. One of the practical methods which facilitates the manually separation of bast fibers from core part and decreases the lignin content of the fibers is a method called "retting process" through which the kenaf stalks are immersed in marsh and undergo the lignin biodegradation by the enzymatic activities in the marsh during the treatment.

In the present paper, the pulping and papermaking characteristics of retted and unretted kenaf bast fibers under various cooking conditions are investigated. The obtained results are compared with those of commercial manila hemp pulp which is used mainly as specialty paper applications.

2. Materials and methods

2.1 Materials

The commercially available retted kenaf bast fiber, imported from China, was used. This will be simply referred to as Chinese bast fiber and designated as CBF. The epithelial layer has been almost removed in CBF. Also the unretted bast fiber and the woody core part, which had been harvested in Japan, were used in comparison with the CBF. Those will be referred to as Japanese bast fiber and Japanese core part, and designated as JBF and JCP, respectively. Some properties of CBF, JBF, and JCP are given in **Table 1**. JCP was not subjected to any pulping method and only was used for analytical comparison.

The commercial manila hemp pulp was compared to the kenaf pulps prepared in this study.

Table 1 Comparison of some properties of Chinese (CBF) and Japanese (JBF) bast fibers with those of Japanese core part (JCP)

Components (%)	CBF	JBF	JCP
Eth-Ben. extractives	1.2	4.3	4.8
Klasson lignin	8.2	12.5	24.1
Ash	4.0	4.2	3.6
Holocellulose	76.4	72.8	78.7
Pentosans	23.1	21.3	17.4
Basic density (g/cm^3)	0.33	0.31	0.14

2.2 Methods

2.2.1 Pulping

Both of the bast fibers were cut into pieces of 2-3cm length. The kraft and soda-AQ methods were employed for them as conventional pulping processes. For CBF, due to having some certain characteristics such as low lignin content, a so-called non-pressurized soda method was employed. All the soda-AQ and kraft cooks were carried out with minidigester (capacity of 60ml, heated in a temperature-controlled oil bath system) and autoclaves (capacity of 1 and 2 ℓ , equipped with heater regulated by electrical transformer) in our laboratory. Charges of 100-200g bast fiber were charged in autoclave for cooking and quantities of 8g were charged for cooking in minidigester. For all the cooking tests, the chemical charges were based on percentage to oven-dried bast fiber and calculated as Na_2O base.

(1) Soda-AQ cooking

For pulping of the CBF, to achieve a proper condition, the cooking temperature and chemical charge were examined as independent variables using minidigester. The cooking temperature was examined within the range of 160-170℃ , and the total active alkali applied was within the range of 11-14%. In all of those soda-AQ cooks, the time at maximum cooking temperature was kept constant for 2 hr, and also the amount of anthraquinone addition was fixed at a same amount for all conditions, 0.2% (based on o.d. weight of bast fiber) .

The followings were the final conditions employed for the soda-AQ cooking of both bast fibers :

Liquor to solid ratio : 5/1 (ℓ /kg) ; Cooking temperature : 170℃ ; Time to cooking temperature : 1 hr ; Time at cookingtemperature : 2hr ; Anthraquinone charge : 0.2% ; NaOH charge : 13% for CBF and 16% for JBF.

(2) Kraft cooking

The sulfate cooking was applied for both bast fibers. Considering the lignin content and also the amount of chemicals charged in soda-AQ cooks, the conditions applied for kraft cooks were as follows :

Liquor to solid ratio : 5/1 (ℓ /kg) ; Time to cooking temperature : 1 hr ; Cooking temperature : 170°C ; Time at cooking temperature : 2 hr ; Active alkali charge : 13% and 16% for CBF and JBF, respectively ; Sulfidity : 10% for both bast fibers.

(3) Non-pressurized soda cooking

The CBF was subjected to non-pressurized soda cooking. At first the bast fiber was cut into the lengths of 3-4cm. For each cooking test, l00g bast fiber and the necessary amounts of sodium hydroxide in water were charged to a 2 ℓ glass beaker and heated to the boiling point at atmospheric pressure. To retain constant volume of the cooking liquor, required amount of hot water was gradually added into the beaker. The cooking conditions were as follows :

Time to cooking temperature : 20 min ; Cooking temperature : 100 ℃ ; Time at cooking temperature : 5hr ; NaOH charge : 21%.

In all pulping methods, the chemicals charged were based on the oven-dried weight of bast fibers. All of the cooking tests were carried out twice or more. At the end of cooking, the system was cooled and the recovered pulps were washed by tap water until no residual chemicals found and then passed over a laboratory flat screen. Then the screened pulps were dewatered and their yield and Kappa number were determined for every cooking conditions.

2.2.2 Conventional two-stage hypochlorite bleaching

A commercial sodium hypochlorite solution (containing about 5% active chlorine) was used as bleaching reagent. In order to reach the handsheet brightness of about 80% (near to that of manila hemp pulp sheets), the required amount of active chlorine (Cl) was added to each unbleached pulp sample. The necessary active chlorine for each sample was estimated from the Kappa number of each sample and about 70% of the chlorine demand was added as NaCIO solution in the first stage and 30% in the second stage. For each bleaching stage, the initial pH was adjusted to about 10. The consistency of the pulp slurry was controlled to be 10%, and the pulp suspension was kept in a polyethylene bag and placed in a water bath. The samples were heated at 60'C for 4 hr. At the end of each bleaching stage, the slurry was filtered and washed with hot tap water. Finally, the bleached sample was dewatered and kept in the refrigerator for the next procedure.

2.2.3 Pulp beating

The bleached pulps were beaten by a Tappi Standard Niagara beater in our laboratory. The amount of pulp charged for each beating trial was 90 g, pulp consistency is 1.2%.

2.2.4 Analytical methods

The chemical compositions of materials, i.e., ash, alcohol-benzene extractives, holocellulose, Klasson lignin and pentosans, were determined according to JIS P 8003, 8010, 8007, 8008 and 8011, respectively. The basic density of bast fibers and core part was calculated according to JIS P 8014.The Kappa number was determined according to the TAPPI T 452. Handsheets were prepared using a TAPPI Standard Sheet Machine. The brightness and opacity of the handsheets were determined with a Hunter-type reflectometer, according to TAPPI T 452 and JIS P 8138, respectively. Handsheet strength properties were determined according to TAPPI T 220.

3. Results and discussion

3.1 Preparation and properties of unbleached kenaf pulps

Figs. 1 to **3** show the changes in Kappa number, screened pulp yield, and brightness as a function of alkali charge in the soda-AQ cooking of CBF at three different cooking temperatures; 160℃, 165℃, and 170℃. The Kappa number values decrease with increasing the soda concentration and cooking temperature. On the other hand, the screened pulp yield increases with increasing the soda concentration, mainly up to 13%, and at larger concentration no significant variation is observed. The maximum screened yield value (about 63%) was obtained at 170 ℃ and alkali charge of 13-14% from the average of three measurements. However, alkali charge of 13% is preferable from economical point of view. The brightness is also improved with increasing the soda concentration and cooking temperature, but there is not very much change for the brightness at the concentration of 13-14%. The results indicate that an alkali charge of 13% and cooking temperature of 170℃ is the most suitable condition for the 2 hr soda-AQ cooking of CBF.

Some properties of the unbleached soda-AQ

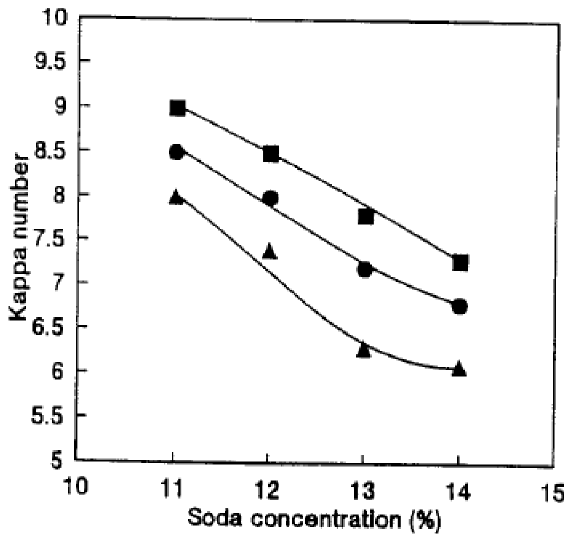

Fig. 1 Relationship between alkali charge and Kappa number in soda-AQ cooking of CBF at different temperatures

Legend : ■ :160℃, ●:165℃, ▲:170℃

Note : Anthraquinone charge was 0.2% (based on o.d. bast fiber) and the cooking time was for 2hr

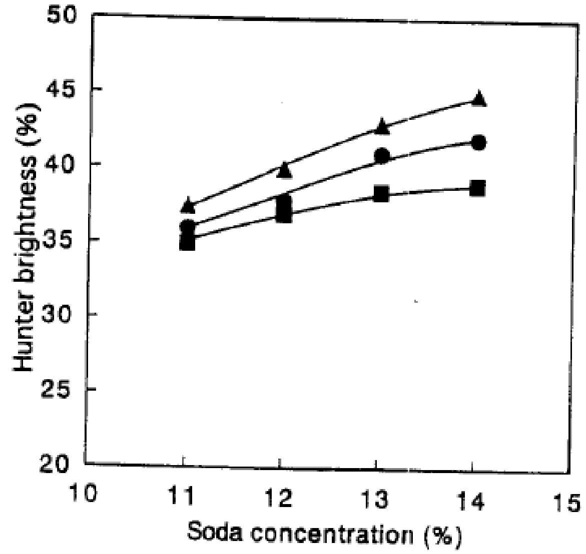

Fig. 3 Relationship between handsheet hunter brightness and alkali charge for soda-AQ pulping of CBF at different cooking temperatures

Legend : Refer to Fig. 1

Note : Refer to Fig. 1

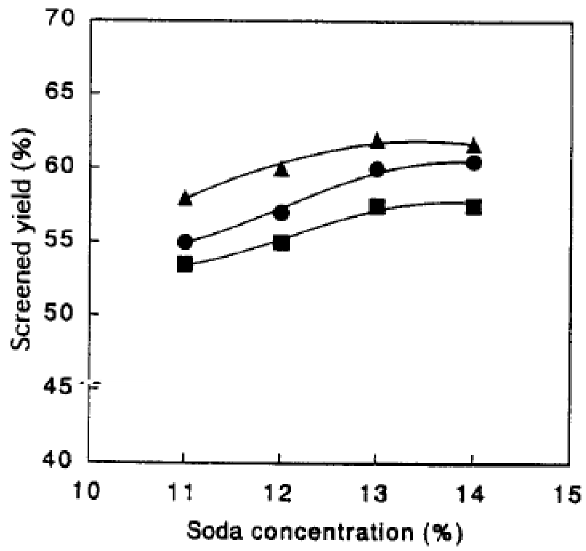

Fig. 2 Relationship between alkali charge and screened pulp yield in soda-AQ cooking of CBF at different temperatures

Legend : Refer to Fig. 1

Note : Refer to Fig. 1

and kraft pulps of CBF and JBF are compared in **Table 2**. The properties of the bast fiber pulps obtained were highly influenced by the differences in the chemical and fibrous compositions of two bast fibers. As shown in **Table 1**, the lignin content of CBF (8.2%) is lower than that of JBF (12.5%). Lower values of ethanol-benzene extractives and lignin content in the CBF, comparing to those of JBF, are considered to be mainly caused by the retting pretreatment of CBF done in China. **Table 2** shows that the Kappa number values of unbleached kraft and soda-AQ pulps of CBF (6.3 and 6.2, respectively) are significantly lower than those of unbleached JBF pulps (19.9 and 21.0, for the soda-AQ and kraft pulps, respectively), although the alkali charges were higher in JBF cooking (16%) than that of CBF (13%). Furthermore, the unbleached CBF pulps have lower ash content with a greater screened pulp yield than those of JBF. The Kappa number values of the soda-AQ and kraft pulps from both bast fibers were not significantly influenced by the type of cooking. On the other hand, the non-pressurized

Table 2 Characteristics of unbleached soda-AQ, kraft, and non-pressurized soda pulps prepared from Chinese (CBF) and Japanese (JBF) kenaf bast fibers

	CBF			JBF	
Characteristics	Soda-AQ	Kraft	Non-pre.	Soda-AQ	Kraft
Unbleached yield (%)	63	63	74	49	50
Kappa number	6.2	6.3	28.4	21.0	19.9
Ash (%)	1.3	1.3	1.9	2.9	2.7

soda pulp of CBF has higher Kappa number and yield than those prepared by the soda-AQ and kraft methods.

3.2 Properties of bleached pulps and handsheets

3.2.1 Pulp properties

All of the unbleached bast fiber pulps were bleached to a brightness level of manila hemp sheets (about 80%) by using a two-stage hypochlorite bleaching process. Some properties of the bleached pulps are given in **Table 3**. The yields of both kraft and soda-AQ pulps were decreased almost to the same extent after bleaching, but there exist a big difference between the yield values for CBF and JBF. However, among the three CBF pulps, at a similar handsheet brightness level, the non-pressurized soda pulp of CBF having 60% yield is superior to others. Considering the big difference existed in the Kappa number of unbleached samples (**Table 2**) , it would be reasonable to have higher bleached yield for the non-pressurized cooking.

3.2.2 Handsheet properties

To evaluate the effect of beating on different bleached pulps obtained, the strength properties of bleached handsheets were determined at different degrees of beating. **Figs. 4** to **7** show various strength properties of handsheets plotted against the CSF (Canadian Standard Freeness) for the soda-AQ, kraft, and non-pressurized soda pulps of CBF and the commercial manila hemp pulp. As shown, the tensile, folding, and burst strengths of CBF pulps improve as beating continues, but with different tendencies. The beating process is more effective for the non-pressurized soda pulp than for the soda-AQ or kraft pulp of CBF. Those three strength properties of non-pressurized soda pulp

Table 3 Characteristics of bleached pulps prepared from Chinese (CBF) and Japanese (JBF) kenaf bast fibers

	CBF			JBF	
Characteristics	Soda-AQ	Kraft	Non-pre.	Soda-AQ	Kraft
Bleached yield (%)	53	52	60	38	39
Initial CSF* (ml)	685	680	696	625	633
Brightness (%)	80.0	81.0	79.0	78.5	79.0

*CSF : Canadian Standard Freeness

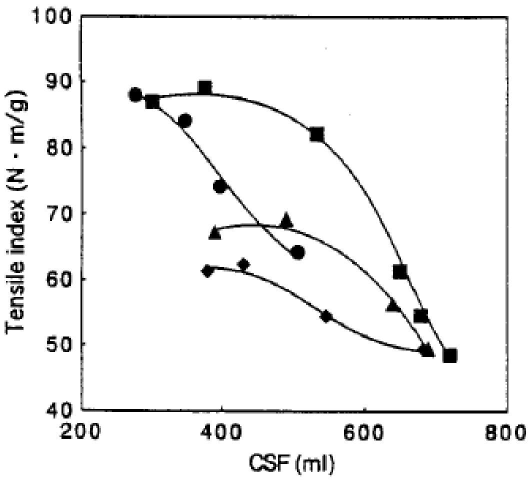

Fig. 4 Relationship between CSF and tensile index of manila hemp pulp and different bleached pulps of CBF

Legend : ■ :Non-pressurized soda pulp of CBF, ▲ :Soda-AQ pulp of CBF, ◆ :Kraft pulp of CBF, ● :Manila hemp pulp

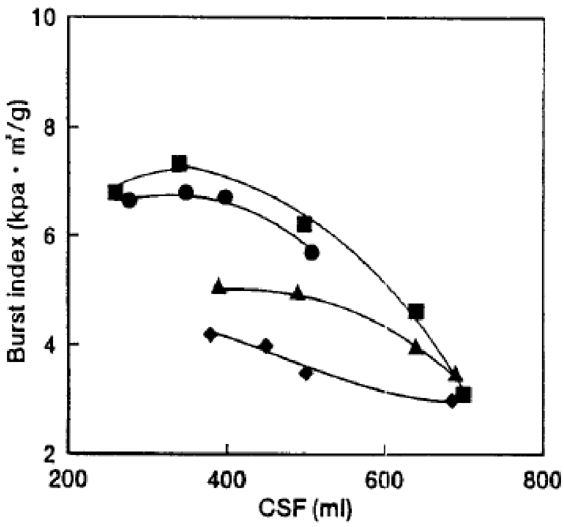

Fig. 6 Relationship between CSF and burst index of manila hemp pulp and different bleached pulps of CBF

Legend : Refer to Fig. 4

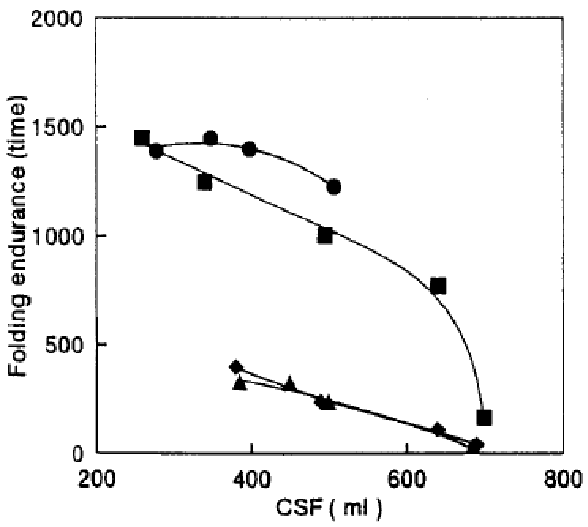

Fig. 5 Relationship between CSF and folding endurance of manila hemp pulp and different bleached pulps of CBF

Legend : Refer to Fig. 4

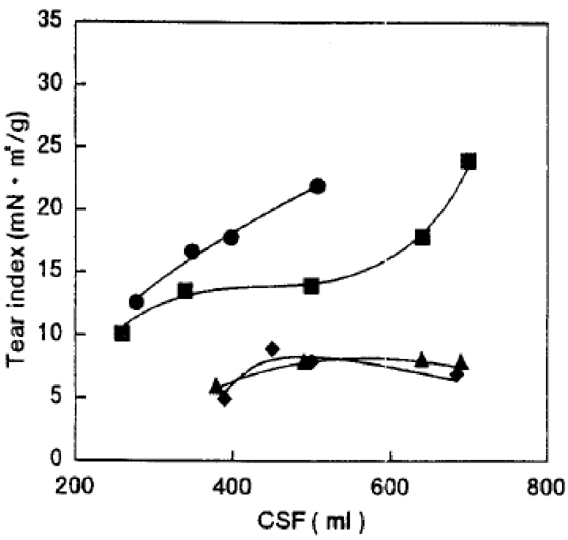

Fig. 7 Relationship between CSF and tear index of manila hemp pulp and different bleached pulps of CBF

Legend : Refer to Fig. 4

smoothly increase with decreasing the CSF and then leveled off, except the folding endurance. Comparisons of the strength properties of various bleached handsheets at 400ml CSF are given in **Table 4**. As indicated, the strength properties of the non-pressurized soda pulp are sufficiently comparable to those of manila hemp pulp. However, the tear index value of the non-pressurized soda pulp at the CSF of 400ml is lower than that of manila hemp pulp, but it become nearly equal at 300ml CSF (**Fig. 7**).

Table 4 Comparison of strength properties of various bleached pulp handsheets at 400ml CSF

Characteristics	CBF			JBF		Manila hemp
	Soda-AQ	Kraft	Non-pre.	Soda-AQ	Kraft	
Basis weight (g/m^2)	65.0	63.0	62.3	60.0	62.5	61.3
Density (g/cm^3)	0.62	0.62	0.64	0.63	0.61	0.63
Breaking length (km)	6.8	6.4	9.0	5.5	6.0	7.4
Burst index ($kPa \cdot m^2/g$)	5.1	4.2	7.2	3.8	4.0	6.7
Tear index ($mN \cdot m^2/g$)	7.0	6.7	14.0	12.0	15.0	17.2
Folding endurance (time)	295	324	1200	540	455	1386

4. Conclusion

From this research the following conclusions can be given;

1) The contents of Klasson lignin and ethanol-benzene extractives of CBF were lower, and the amounts of holocellulose and pentosans were a little higher than those of JBF. These differences are considered to be caused by the retting pretreatment of CBF done in China.

2) On the kraft and soda-AQ pulping, it is determined that the CBF can be pulped with lower cooking chemical charges than for JBF, and with greater pulp yields.

3) An alkali charge of 13% and cooking temperature of 170℃ is the optimum for the 2 hr soda-AQ cooking of CBF.

4) The pulping properties of either bleached or unbleached soda-AQ pulps of both bast fibers were satisfactorily similar to those of kraft pulps, and this indicates that the soda-AQ method can be a substitute for kraft method in pulping of the kenaf bast fibers.

5) On the evaluation of strength properties of handsheets of all bleached pulps, the non-pressurized soda bleached pulp from CBF was superior to other pulps produced by soda-AQ or kraft from both bast fibers.

6) In comparison with commercial manila hemp pulp, the handsheets prepared from non-pressurized caustic pulp of CBF showed quite comparable strengths to those of manila hemp pulp, at similar levels of freeness and brightness. Therefore, the non-pressurized soda pulp of retted CBF can be used a hopeful substitute for the manila hemp pulp.

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精錬発酵ケナフ靭皮繊維パルプの製造と特性および マニラ麻パルプ代替品としての評価

アブドレザ・ネザモルエスラミ*　鈴木恭治*　門屋　卓*

精錬発行処理を受けた中国産ケナフ靭皮繊維(CBF)を用いて、異なる蒸解方法により得られたパルプの特性を評価するとともに、特殊紙用に用いられているマニラ麻パルプ代替品としての可能性を検討した。蒸解方法は一般に行われているソーダAQ法、クラフト法に加えて蒸圧ソーダ蒸解法も行った。

ソーダAQ法、クラフト法ともにケナフ靭皮繊維に対し良質なパルプを与えたが、CBFから調製されたパルプは精錬発酵処理されていない日本産ケナフ靭皮繊維（JBF）より低活性アルカリ条件にも拘らず高パルプ収率、低カッパ価、低灰分量であった。

各パルプ化方法により調製したパルプを漂白した後ピーターで叩解し、濾水度と強度特性の関係を調べた結果、CSF４００での引張りおよび破裂指数は常圧ソーダ、ソーダAQ、クラフトの順に高い値となり、常圧ソーダパルプはマニラ麻を上回る強度を示した。耐折強さは3種パルプともマニラ麻パルプより低い値となったが、常圧ソーダパルプはCSF３００付近でマニラ麻と同様の値となった。したがって常圧ソーダ蒸解したCBFパルプはマニラ麻パルプの代替品として有望であることが分かった。

キーワード：ケナフ、精錬発酵靭皮繊維、常圧蒸解、ソーダAQ蒸解、クラフト蒸解、強度特性、マニラ麻パルプ