J Wood Sci (2000) 46:364–370

ORIGINAL ARTICLE

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Combination treatment of kenaf bast fiber for high viscosity pulp

Received: May 6, 1999 / Accepted: October 20, 1999

Abstract The viscosity of kenaf bast fiber has been found to be highly sensitive and variable with different pulping methods; therefore, it is important to choose proper chemicals and conditions for pulping and bleaching of kenaf bast fiber. From several pulping experiments, a nonconventional pulping method with a combination of ammonium oxalate pretreatment followed by soda pulping at normal pressure and then acidic chlorite delignification was developed to obtain high-viscosity pulp (162 centipoise). The optimum level of alkali dosage of soda pulping for high-viscosity pulp was found to be 15% (on pulp as NaOH). Pulps showed linear relations between viscosity and xylose or glucose contents, but the combination pulping method gave extremely high pulp viscosity, beyond the relations. The highest viscosity pulp from kenaf bast fiber demonstrated a tear index about twofold and a folding endurance 6.2-fold higher than those of Manila hemp pulp with comparable tensile and burst indexes. The high-viscosity pulp could be used in the production of high-quality currency paper or longevity paper for special uses.

Key words Kenaf bast fiber · Ammonium oxalate · Pulp viscosity · Tear index · Folding endurance

Introduction

Pulping of kenaf (*Hibiscus cannabinus* L.) bast fiber has been examined by most of the conventional pulping methods (e.g., kraft, soda, neutral sulfite) and unconventional pulping methods (e.g., normal-pressure pulping, biopulping).[1–4] There have been some reports seeking optimum treatment conditions for the highest-viscosity pulp.[5–9] Pulp viscosity is considered to be an important indicator of pulp damage in different pulping methods. In our earlier publication,[3] we examined a series of normal-pressure pulping procedures and found that alkaline hydrogen peroxide pulping of kenaf bast fiber pretreated with ammonium oxalate provides a pulp with good brightness and mechanical properties. However, damage to pulps during pulping at normal pressure was not characterized. In this report, we studied the viscometric property of kenaf bast pulps obtained by normal-pressure pulping and discuss their characteristics by comparing them with those derived by other treatments. An excellent pulp with extraordinarily high viscosity and good mechanical properties was obtained by a combination of ammonium oxalate (Amox) pretreatment, soda pulping at normal pressure, and acidic chlorite treatment.

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Part of this paper was presented at the 43rd Lignin Symposium, Fuchu, Tokyo, October 26–27, 1998

Experimental

Materials

Kenaf, variety sekko-ichi from China was cultivated at Tateda village in Kochi Prefecture, Japan. The field area was 0.2 hectares. Kenaf plant was harvested on November 15, 1997, 180 days after seed sowing, and the bast was separated by hand from the core during the green stage after removing the leaves and green fruits. The average plant height and diameter were 380 cm and 1.70 cm, respectively.

Pulping methods

During screening to get the optimum pulping method, various chemical combinations and conditions (Table 1) were examined for the kenaf bast fiber. In addition, pine kraft pulp was prepared to compare with the above pulping of kenaf bast fiber. All pulping methods were repeated. The pulping methods were symbolized alphabetically from methods A to K including pine kraft (method G) and

Table 1. Pulping conditions

Treatment methods	Chemicals and procedure	Liquor ratio	Time and temperature
Group 1[a]			
A: acidified sodium chlorite method	CH_3COOH + $NaClO_2$	1 : 10	24 h at 60°C
	1 N NaOH	1 : 10	1 h at 60°C
	0.5% HCl	1 : 10	1 h at 20°C
B: modified method from method A with Amox treatment	CH_3COOH + $NaClO_2$	1 : 10	24 h at 75°C
	0.5%$(NH_4)_2C_2O_4$ (Amox)	1 : 30	1 h at 85°C
	1% NaOH	1 : 10	2/3 h at 60°C
C: peracetic acid method	Equal quantities of glacial acetic acid and 20% H_2O_2	1 : 10	7 h at 98°C
D: peracetic acid method	Equal quantities of glacial acetic acid and 20% H_2O_2	1 : 10	24 h at 60°C
Group 2[b]			
E: soda pulping	17% NaOH[d]	1 : 5	1.5 h to 170°C/1.5 h at 170°C
F: kraft pulping	17% NaOH[d] and 25% sulfidity	1 : 5	1.5 h to 170°C/1.5 h at 170°C
G: soft wood kraft pulp (pine)	20% NaOH[d] and 25% sulfidity	1 : 5	1.5 h to 170°C/1.5 h at 170°C
Group 3[c]			
H: nitric acid pulping	10% HNO_3	1 : 10	1 h under refluxing condition
I: acetic acid pulping	95% CH_3COOH + 0.32% H_2SO_4	1 : 10	1 h under refluxing condition
J: alkaline hydrogen peroxide	15% NaOH[e] + 5% H_2O_2	1 : 10	1 h under refluxing condition
K: alkaline hydrogen peroxide	15% NaOH[e] + 10% H_2O_2	1 : 10	1 h under refluxing condition

[a] Sample preparation method for kenaf bast fiber length measurement 10–13
[b] High pressure pulping methods
[c] Normal pressure pulping methods after Amox pretreatment[3]
[d] Alkali charge as Na_2O
[e] Alkali charge as NaOH

categorized into three groups based on their treatment objectives and background.

Delignification of pulp for viscosity measurement

Pulps of group 1 (Methods A–D) and the nitric acid method (method H) with low kappa numbers were directly subjected to a viscosity measurement. A pulp of 0.5 g was delignified in a poly bag for group 2, with 10% acatic acid and 5% sodium chlorite solution buffering by 0.3 M sodium acetate at 65°C for an hour, and the pulps of group 3 (methods I–K) were delignified with acidic chlorite in a holocellulose procedure for viscosity measurements.[14]

Viscosity measurement

Pulp viscosities were measured by the Japan TAPPI test method.[15] An air-dried sample equivalent to 0.125 g oven-dried pulp was placed in a test tube, and 10 ml of distilled water was added. The mixture was stirred with a motor-driven copper rod for 5 min. Cupriethylenediamine solution (0.5 mol/l, 5 ml) was added, and the mixture was stirred for 5 min. Another 10 ml of cupriethylenediamine (1.0 mol/l) solution was added, and the mixture was stirred for 15 min. Pulp viscosity was measured by a capillary viscometer as 0.5% cellulose solution, using 0.5 M cupriethylenediamine as a solvent.[15] The pulp viscosity experiments were repeated more than three times, and the average value was used.

Pulp viscosities determined as centipoise (cp) were converted to degree of polymerization (DP) of polysaccharides according to the following formula[16]: $DP^{0.905} = 0.75[954 \log(X) - 325]$, where X is the TAPPI viscosity in centipoise.

Chemical analysis

Kappa numbers of the pulps were determined according to the SCAN-C1:77 test method.[17] For measuring ash, a 2-g sample was placed in a porcelain crucible and ignited at 600° ± 25°C in a muffle furnace for 2–3 h.[14] Pectin content was measured after extraction with 100 ml of 0.5% Amox solution at 85°C for 24 h on 1 g of moisture-free equivalent sample by the carbazole-sulfuric acid method.[14] Calcium was measured by ion chromatography (model HIC-6A; Shimadzu, Japan). Ashes were dissolved in 1 N HCl, and the solution was diluted to around 200 μS/cm to measure Ca ions by ion chromatography. Neutral sugar composition of the pulps was determined by the alditol acetate method.[14] The gas chromatograph used in this study was a Hitachi model 163 equipped with a flame ionization detector. A 2 m long and 3 mm i.d. column, packed with EGA 0.2% Silicone XF-1150 0.4% was employed with a nitrogen flow rate of 30 ml/min. The column was operated isothermally at 190°C with an injection port temperature of 230°C and a detector temperature of 195°C. Inositol was used as an internal standard. All experiments for chemical analysis including the kappa number were repeated.

Measurement of fiber length

A 50-ml fiber suspension of 0.03% consistency was scanned by the Kajaani FS-200 fiber length analyzer

Table 2. Results of pulp comparison

Method	Pulp yield (% of raw sample)	Kappa no.	Kappa no. after treatment for viscosity measurement	Fiber length (mm)	Viscosity (cp)	Pulp DP	Pulp brightness (% ISO)
Group 1							
A	57.8	3.5	NT	2.30	24.6 ± 1	1506	82.0
B	53.0	10.2	NT	2.57	65.0 ± 5	2218	62.5
C	47.6	3.0	NT	2.18	4.2 ± 0.5	353	83.5
D	64.1	4.5	NT	2.50	10.0 ± 1	900	64.0
Group 2							
E	43.0	20.0	4.5	2.26	51.5 ± 3	2022	71.0
F	46.0	14.5	4.5	2.26	33.0 ± 1	1709	72.0
G	50.3	36.5	4.9	2.46	35.0 ± 2	1750	72.0
Group 3							
H	33.8	5.2	NT	0.64	10.8 ± 1	951	70.0
I	38.0	16.0	5.0	1.04	25.5 ± 1	1530	82.0
J	55.2	59.0	3.8	2.31	72.0 ± 5	2260	83.0
K	54.4	50.9	3.8	2.07	52.8 ± 3	2039	83.0

Refer to Table 1 for the symbols; NT, not treated; DP, degree of polymerization

(KAJAANI Electronics, Finland) for fiber length measurement.

Measurement of physical properties

Freeness, burst index, tensile index, folding endurance, and tear index were measured by JIS P-8121, P-8112, P-8115, and P-8116, respectively.[18] The brightness was in ISO standard, using Technibrite Micro TB-1C (Indiana, USA).

Results and discussion

Effects of pulping methods on pulp viscosity

Kenaf bast pulps with different treatment histories were prepared to evaluate their viscometric properties. The 11 pulps shown in Table 1 were analyzed for pulp yield, kappa number, viscosity, and brightness (Table 2). Method B showed the highest pulp viscosity (65.5 cp), which coincides with its highest fiber length value (2.57 mm) in the group, suggesting the least damage during the treatment used. Within the high-pressure pulping group (group 2), the soda method (method E: 51.5 cp) was higher than the kraft method (33.0 cp), which is comparable to softwood kraft pulp (35.0 cp). Pulp viscosities obtained by high-pressure pulping methods were also lower and high pressure and temperature may be some of the reasons for low pulp viscosity.[19]

Among the normal-pressure pulping group (group 3), nitric acid pulping (method H) showed the lowest value of 10.8 cp; the acetic acid pulping method (method I) also gave lower pulp viscosity than that of the wood pulp used as a reference (method G). These results indicate that acidic pulping is harmful for viscosity probably due to depolymerization by acid hydrolysis. The shorter fibers of the nitric acid pulp (0.64 mm) and acetic acid pulp (1.04 mm) may reflect damage to the fiber by acid pulping. We made optical microscopic observations on the damaged fiber due to acidic pulping.[3] Page and coworkers[20] reported that mineral acid, such as hydrochloric acid treatment during the vapor phase, causes significant reduction of the DP of cellulose. We carried out alkaline hydrogen peroxide treatments after Amox pretreatment, which effectively removes transition metallic ions such as Mn, Fe, and Cu.[3] Alkaline hydrogen peroxide pulping at 5% H_2O_2 dosage (method J) showed the highest pulp viscosity (72 cp). An increase in hydrogen peroxide dosage (at 10% H_2O_2) showed the lowest pulp viscosity. It is expected that we can obtain a higher viscosity when treatment is carried out at lower H_2O_2 dosage level.

Optimization of alkaline hydrogen peroxide treatment for obtaining high pulp viscosity

Alkaline hydrogen peroxide pulping at boiling temperature (about 100°C) for 1 h at 5% H_2O_2 dosage was selected for the highest pulp viscosity in Table 2. To examine the optimization of H_2O_2 dosage, Fig. 1 was obtained during additional experiments. It can be estimated through the regression line that the highest viscosity with H_2O_2 treatment may reach 86.1 cp at zero H_2O_2 dosage (Fig. 1). We found that soda pulping without H_2O_2 addition gave surprisingly higher pulp viscosity (162 cp) after acidic chlorite treatment than that of the estimated value of 86.1 cp. (no. 3 in Table 3). It means that normal-pressure soda pulping and acidic chlorite treatment after Amox pretreatment is one of the ways to obtain a high-viscosity pulp from kenaf bast fiber. It indicates that addition of low H_2O_2 dosage during alkaline pulping at boiling is harmful to the viscosity.

The effects of various alkali (NaOH) dosages for normal-pressure soda pulping are shown in Table 3. The pulp viscosity was found to give the highest value at 15% alkali dosage. Excess alkali dosage reduced the pulp viscos-

Table 3. Results of normal pressure soda pulping[a] followed by chlorite treatment

No.	Alkali dosage	Pulp yield (% of raw sample)	Kappa no.	Kappa no. after acidic chlorite treatment	Fiber length (mm)	Viscosity (cp)	Pulp DP (estimated from viscosity)	Brightness (%ISO)
1	5%NaOH	52.6	74.5	4.5	NM	62.7[b] ± 15.0	NM	78.1
2	10%NaOH	50.7	74.0	4.2	NM	150.4 ± 10.0	2794	81.2
3	15%NaOH	49.0	72.2	3.8	2.73	162.0 ± 15.0	2850	83.0
4	20%NaOH	48.0	71.4	3.8	NM	159.5 ± 12.0	2835	83.0
5	25%NaOH	47.5	70.4	3.8	NM	147.7 ± 12.0	2779	83.0

NM, not measured

[a] After Amox pretreatment [0.5% Amox solution; sample/liquor ratio 1 : 25) for an hour at refluxing condition (about 100°C) at normal pressure

[b] At 5% NaOH dosage, pulp was not dissolved sufficiently in the cupricethyldiamine solution

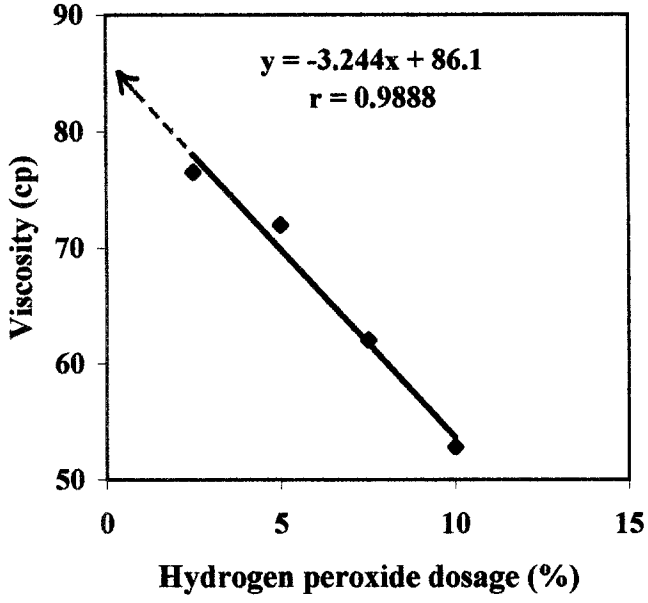

Fig. 1. Effect of hydrogen peroxide dosage on pulp viscosity during alkaline hydrogen peroxide pulping of kenaf bast fiber after ammonium oxalate pretreatment

ity, but the magnitude of reduction was small. This implies that the alkali dosage does not have a large effect on pulp viscosity during normal-pressure pulping. In contrast, with pressurized pulping methods (soda and kraft pulping: group 2 in Table 1) the pulp viscosity declines rapidly when the alkali charge is increased at constant cooking time and temperature.[5,6] The three-stage treatment – a combination of Amox pretreatment, soda (15%) pulping at normal pressure, and acidic chlorite treatment – is called method N, a new pulping system.

The yield of bleached pulp with method N is higher than the yields of unbleached soda and kraft pulps (methods E and F in Tables 2 and 3). The pulp DP of the new method is calculated to be 2850, which is much higher than that of commercial market pulp and comparable to the generally accepted value of native plant cellulose DP of 3000.[21] The fiber length of pulp treated with method N is longer (2.73 mm) compared with method B, suggesting less physical damage to the fibers by method N.

Relation between viscosity and chemical composition

To determine the causes of surprisingly high viscosity of the pulp with method N, we checked the relation between viscometric properties and neutral sugar compositions and the pectin, ash, and calcium ion (Ca^{2+}) contents. Neutral sugar compositions of kenaf bast fibers prepared by various treatments are shown in Table 4. Amox pretreatment increased neutral sugar contents of kenaf bast fiber due to removal of impurities such as pectin, ash, and extractives. Figure 2 shows the relation between glucose content and pulp viscosity. The pulp obtained with the nitric acid method (method H), having the highest glucose content but the lowest pulp viscosity, may be depolymerized by acid hydrolysis. The pulp obtained with method J, having low glucose content, showed high pulp viscosity; this can be explained by the fact that pulp with high hemicellulose content undergoes low cellulose degradation, which results in high pulp viscosity. All the pulps except those treated by method N showed a significant correlation between glucose content and pulp viscosity (Fig. 2). The pulp viscosity with method N is extraordinarily high when compared to that of pulps treated by the other methods. Page and coworkers[20] reported that for an individual treatment high cellulose content gave high fiber strength. Pulp viscosity of kenaf bast fiber after different treatments, however, demonstrated that the pulp viscosity was low when the glucose content was high. Removal of hemicellulose might have a major influence on cellulose degradation; and the degraded pulp (low-viscosity pulp) even with high glucose content shows low viscosity, as in the case of dissolving pulp (average viscosity 11.7 cp).[22] This reflected a tendency toward high viscosity with high residual hemicellulose (Table 4). As shown in Fig. 3, the xylan content of the pulp treated by method H had the lowest value with low pulp viscosity. In contrast, method J with the highest xylan content showed the highest pulp viscosity when method N was excluded. The xylan content and pulp viscosity showed a highly linear relation when method N was excluded. Genco et al. reported that increased hemicellulose retention in the pulp had a positive impact on strength properties.[23] Cao et al.[24] also noted that for recycled paper pulp strengths decreased significantly with a decrease in xylan contents. The high xylan content with method N showed a positive effect on the strength properties (Table 5).

Table 4. Carbohydrate composition of the pulps in relation to pulp viscosity

Method	Arabinose (%)	Xylose (%)	Mannose (%)	Galactose (%)	Total hemicellulose (%)	Glucose (%)	Total sugars (%)	Pulp viscosity (cp)
Alcohol benzene-extracted sample	1.9	12.7	2.2	1.3	18.1	59.1	72.2	–
Amox-treated sample	1.3	14.7	2.0	1.1	19.10	61.7	80.8	–
Group 1:								
B	1.8	19.0	2.5	1.0	24.3	74.2	98.5	65.0
Group 2								
E	1.0	16.4	1.3	0.6	19.3	77.2	96.5	51.5
F	0.3	12.9	0.7	0.3	14.2	83.2	97.4	33.0
G	0.6	13.2	7.3	0.8	21.9	71.6	93.5	35.0
Group 3								
H	–	9.10	1.7	–	10.8	90.3	101.0	10.8
I	0.9	10.2	2.9	0.6	14.6	83.1	97.7	25.5
J	1.7	21.6	3.8	1.0	28.1	72.7	100.8	72.0
K	1.7	19.0	3.8	1.0	25.5	74.2	99.7	52.8

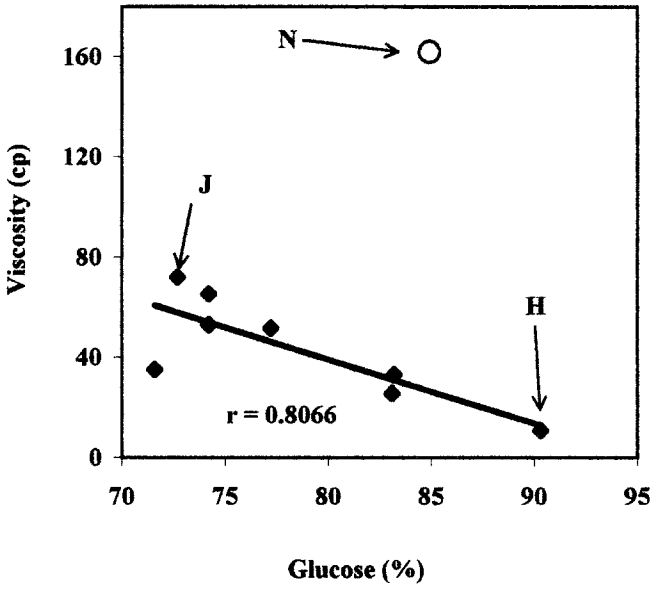

Fig. 2. Relation between pulp viscosity and glucose content. *r*, correlation coefficient significant at 5% level. Letters (*H*, *J*) are symbols for the pulping methods shown in Table 1. Regression coefficient (*r*) was calculated excluding method N

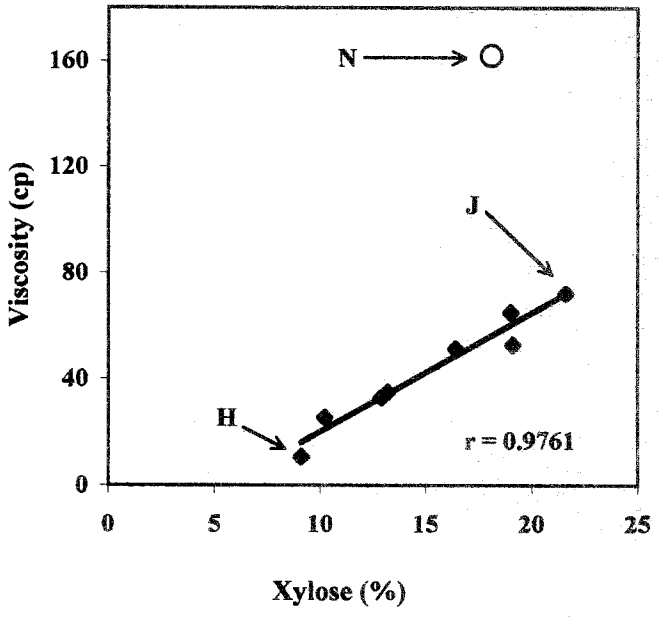

Fig. 3. Relation between pulp viscosity and xylose content. *r*, correlation coefficient significant at 1% level. See Fig. 2

Kenaf bast fiber has high pectin and ash contents.[3] The pectin contents of the pulp via method A (9.42%) and method D (10.23%) (Fig. 4) were high compared with the other pulps; but the pulp viscosities of these methods are at the lowest levels. The pectin content in the pulp via method N is at the same level as that of pulp from method B (viscosity 65 cp), but their viscosities are significantly different, suggesting that pulp viscosity does not correlate with residual pectin content.

The relations of pulp viscosity with ash and Ca ion contents are shown in Figs. 5 and 6, respectively. The acid pulping methods (methods H and I) had good ability to remove ash, whereas method D was least able to remove ash content. No significant relation between pulp viscosities and residual ash contents was found in this study. Calcium is known to be a major metal ion in kenaf bast fiber,[3] but the Ca ion content did not have positive or negative effects on pulp viscosity (Fig. 6). Jameel et al. reported that some low alkalinity earth metals, such as Ca and Mg, can behave as carbohydrate protectors during alkaline pulping,[26] but we found that Ca ions in the pulp had no protective effect on pulp viscosity for kenaf bast fiber.

Mechanical properties of high-viscosity pulp

Mechanical properties of high-viscosity pulp obtained by method N were compared with those of softwood kraft pulp

Table 5. Physical strength properties of high-viscosity pulp of kenaf bast fiber in comparison with wood and Manila hemp pulps

Pulp property	Kenaf (high-viscosity pulp)	Softwood bleached pulp (pine)[a]	Manila hemp[b]
Bleached yield (% of raw sample)	49.0	NA	NA
Basis weight (g/m^2)	68.2	61.4	61.3
Kappa no.	3.8	NA	NA
Pulp viscosity	162.0	35.0	NA
PFI revolution	600	1500	NA
CSF (ml)	300	396	400
Tensile index (Nm/g)	72.0	71.3	72.6
Burst index (kPa.m^2/g)	6.1	7.0	6.7
Tear index (mN.m^2/g)	33.0	11.5	17.2
Folding endurance (time)	8635	745	1386
Brightness (%ISO)	83	86	NA
Air resistance (Gurley porosity: s/dl)	16	NA	NA

NA, data not available
[a] Data from Mazumder et al.[3]
[b] Data from Peralta[25]

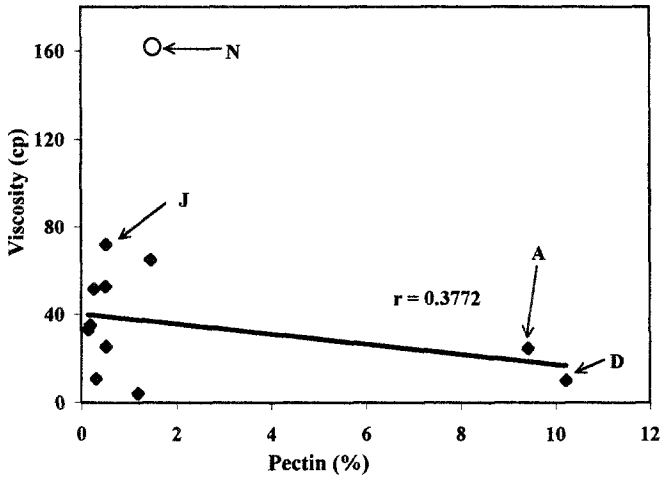

Fig. 4. Relation between pulp viscosity and pectin content. See Fig. 2

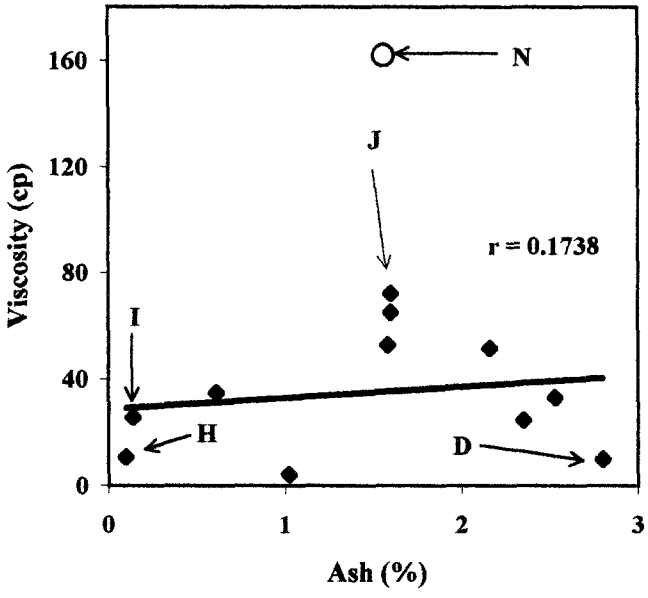

Fig. 5. Relation between pulp viscosity and ash content. See Fig. 2

and Manila hemp pulp (Table 5). The tear index and folding endurance of the method N pulps are 2.0 times and 6.2 times higher than those of the Manila hemp pulp (abaca)[27] with comparable tensile and burst indexes, respectively. The air resistance (Gurley porosity, s/100 ml) of the pulp from method N is 16, comparable to that of highly porous abaca pulp (Gurley porosity, 15.5 s/100 ml).[25] The pulp of Manila hemp is well known for its use in the production of paper with high porosity and excellent tear, burst, and tensile strength; but the tear index and folding endurance of pulp (from method N) of kenaf bast fiber are much higher than those of the Manila hemp pulp.[25] This implies that kenaf pulp from method N could be applied to the production of unique high-quality currency paper or high-longevity paper for special use, such as for dictionaries, encyclopedias, and bond paper.

Conclusions

The pulp viscosity of kenaf bast fiber is highly dependent on the type of treatment. A combination of ammonium oxalate pretreatment, soda pulping at normal pressure under reflux, and acidic chlorite treatment was found to give the highest-viscosity pulp. The optimum alkali dosage level of soda pulping was found to be 15% (on pulp as NaOH). The combination method creates an extraordinary position in the relations between pulp viscosity and carbohydrate compositions and the pectin, ash, and Ca ion contents. Kenaf pulp with high viscosity demonstrated a high tear strength and folding endurance with good tensile and burst indexes when comparing with Manila hemp pulp. Kenaf pulp with high viscosity could be used in the production of high-quality currency paper or high-longevity paper for special uses. The combination method for high-viscosity pulp from kenaf bast fiber needs further study to determine the optimum conditions for practical application.

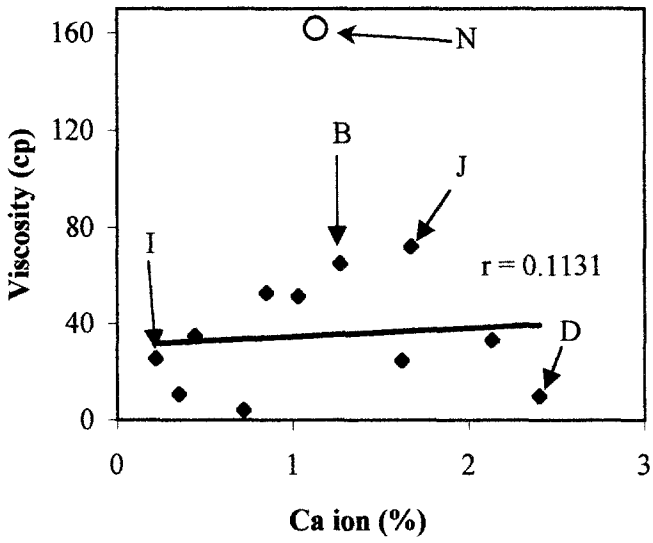

Fig. 6. Relation between pulp viscosity and Ca ion content. See Fig. 2

Acknowledgments This work was partly supported by a Sasakawa Scientific Research Grant of The Japan Science Society and a Grant-in-Aid for Scientific Research (10660166) from the Ministry of Education, Science, and Culture of Japan.

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