EFFECTS OF TCF AND ECF SEQUENCES ON WHOLE STEM KENAF (*HIBISCUS CANNABINUS*) PULP CHARACTERISTICS

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Received October 2004

ASHORI, A., RAVERTY, W. D. & JALALUDDIN, H. 2005. Effects of TCF and ECF sequences on whole stem kenaf (*Hibiscus cannabinus*) pulp characteristics. Totally Chlorine Free (TCF) and Elemental Chlorine Free (ECF) single- and multi-stage sequences were used to bleach whole stem kenaf pulp. The results indicated that in contrast to unbleached kraft wood pulps, kraft kenaf pulps could be easily bleached to a brightness of 91.4% using a four-stage TCF [Q_1(PO)Q_2P] bleaching sequence. The ECF bleached pulps had slightly higher selectivity and yield than those of TCF bleached pulps. The strength properties of the TCF [Q_1(PO)Q_2P] and ECF (D_1ED_2EP) bleached pulps were comparable, with the exception that tear index was slightly higher and tensile index slightly lower for TCF pulps.

Key words: Kraft pulp – pulp properties – Kappa number – chelating agents – selectivity

ASHORI, A., RAVERTY, W. D. & JALALUDDIN, H. 2005. Kesan urutan TCF dan urutan ECF terhadap ciri-ciri pulpa batang kenaf (*Hibiscus cannabinus*). Urutan bebas klorin sepenuhnya (TCF) dan urutan bebas unsur klorin (ECF) satu peringkat dan banyak peringkat digunakan untuk melunturkan pulpa batang kenaf. Keputusan menunjukkan bahawa berlawanan dengan pulpa kayu kraf yang tak luntur, pulpa kenaf kraf boleh diluntur dengan mudahnya kepada 91.4% keputihan menggunakan urutan pelunturan empat peringkat TCF [Q_1(PO)Q_2P]. Pulpa yang dilunturkan menggunakan urutan ECF menunjukkan kepilihan dan hasil yang lebih tinggi berbanding pulpa yang dilunturkan menggunakan urutan TCF. Ciri-ciri kekuatan pulpa TCF [Q_1(PO)Q_2P] dan pulpa ECF (D_1ED_2EP) agak sama kecuali pulpa TCF mempunyai indeks siat yang lebih tinggi dan indeks tegangan yang lebih rendah.

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Introduction

Chlorine and sodium hydroxide are traditionally used in sequential steps to remove the majority of residual lignin in kraft pulp. This process is highly efficient because it is cheap and selective at removing residual lignin. Recently, however, an enormous amount of attention has been focused on negative environmental consequences of using elemental chlorine in bleaching operations. The main reason is that chlorinated organic compounds are generated as by-products of chlorine bleaching of kraft and soda pulps, and many of these materials are difficult to remove from liquid effluents that flow from pulp mills. Furthermore, a number of these so-called organochlorine by-products are toxic to animals and plants. The two classes of organochlorine compounds that cause the greatest concern are two that are produced at very low levels (parts per trillion) in chlorine-based bleaching processes, namely, the polychlorinated dibenzodioxins (PCDDs or dioxins) and the polychlorinated dibenzofurans (PCDFs). These materials are formed as a complex mixture of tri-, tetra-, penta-, hexa-, hepta- and octa-chlorinated congeners that cause disease called chloracne in humans and have been linked to certain forms of cancer. As a result of these unwanted by-products, a great deal of research has been conducted to develop alternative bleaching processes that minimize formation of organochlorines.

The pulp and paper industry has been in the process of phasing out elemental chlorine from bleaching operations because of public opinion and government pressure. The increasing demand for pulps bleached without chlorine compounds contributes to the development of alternative pulping and bleaching processes. Chlorine use has been drastically reduced and Elemental Chlorine Free (ECF) as well as Totally Chlorine Free (TCF) bleaching processes are being developed. Processes in which chlorine dioxide is used in place of the element chlorine have become the most common method of achieving high pulp brightness with minimal organochlorine by-product formation. The ability of chlorine dioxide to selectively oxidize unsaturated carbon bonds in the presence of carbohydrates makes it very selective in removing lignin and in pulp brightening while retaining pulp strength. However, there are a few drawbacks to chlorine dioxide bleaching.

This paper reports the laboratory work to produce whole kenaf (*Hibiscus cannabinus*) bleached pulp using environmental-friendly methods. Hence, the bleaching sequence implemented also has to be of this nature. TCF single- and multi-stage sequences were adopted. Conventional ECF bleaching methods were applied in order to compare the resulting pulps with those from TCF sequences. Different combinations of oxygen, ozone and hydrogen peroxide were used to design an appropriate bleaching sequence for production of TCF kenaf pulp suitable for making high quality printing paper. The main objective of this study was to determine the TCF and ECF sequence effects on strength properties of whole kenaf fibres.

Materials and methods

The unbleached pulp used in this study was whole kenaf kraft pulp, with a Kappa number of 15.5, ISO brightness of 24.0% and viscosity 22.2 cP prepared using a 17% active alkali charge and 25% sulphidity. Prior to bleaching, the pulp was first soaked in water, disintegrated, washed with distilled water, dewatered and then stored at 5 °C.

Pulp bleaching procedures

Overall, 12 different reagents were used under different conditions and various properties of the resulting pulp (Kappa number, viscosity and brightness) were determined. In each bleaching stage, following addition of specified chemicals, the pulp was stirred with a glass rod to ensure even mixing. The slurry (pulp mass and bleaching liquor) was periodically mixed as bleaching proceeded. At the end of each bleaching stage, the slurry was collected on a filter mesh and then thoroughly washed with deionised water. Finally, the pulp was placed in a muslin cloth and spin-dried and homogenized for 10 min.

Oxygen bleaching

Oxygen (O stage) delignification was carried out in a 650 ml autoclave equipped with a gas inlet and a stirrer. Magnesium sulphate solution (8.0 ml of 1% $MgSO_4.7H_2O$) was added to the pulp (15 g o.d.) to minimize cellulose degradation. An aqueous solution of 2% sodium hydroxide (0.35 M) was then added quantitatively to the pulp, followed by distilled water to a total combined weight of 150 g (10% consistency). The mixed slurry was then placed in an autoclave. After flushing the air inside the autoclave for 5 s, the pressure inside the autoclave was adjusted to 690 kPa and kept at this level as the temperature was raised to 100 °C with constant stirring speed (250 rpm). The pulp was maintained at 100 °C and 690 kPa oxygen pressure for 60 min.

Ozone bleaching

Ozone bleaching (Z stage) was carried out in a modified rotating vessel designed as an evaporator, connected with an ozone generator and an oxygen cylinder. The generator used was an SA-100P ozonizer (air-cooling type). All connections were firmly sealed so as to avoid leakage. The flow rate of ozone was 8 ± 0.2 ml s^{-1}.

Pulp (10 g o.d.) was soaked in acidic water of pH 3 (adjusted by addition of sulphuric acid, 2 M) for two hours. The pulp was then squeezed to a consistency of 25% and placed in a round bottom flask reaction vessel. The reaction vessel was rotated at 60 rpm to ensure homogeneous mixing between pulp and ozone. After the passage of ozone for 180 s (generation time), both the inlet and outlet vessels were closed immediately to trap the ozone gas in the vessel and allow it to react as far as possible with the pulp for 5 s (reaction time). The Z stage was run at room temperature.

Hydrogen peroxide bleaching

The stabilizer chemical and the reagent were added to the pulp (20 g o.d.) in the order as listed below:

(a) Magnesium sulphate solution, as 0.039 M solution (0.5% o.d. pulp)
(b) Sodium silicate, as 5% w/v solution (3% o.d. pulp)
(c) Sodium hydroxide standardized solution, as 0.33 M solution (3% o.d. pulp)
(d) DTPA, as 5% w/v solution (0.2% o.d. pulp)
(e) Hydrogen peroxide solution, as 35% v/v solution (3% o.d. pulp)

The charges of $MgSO_4$, Na_2SiO_3 and DTPA were applied in order to control or retard the rate of peroxide decomposition and cellulose degradation. The pH of the slurry was adjusted to 10.5–11. The consistency of the slurry was adjusted to 10% with distilled water and then placed in a sealable polyethylene bag where it was thoroughly mixed by manual kneading. The sealed bag was then placed in a water bath at 80 °C and heated for two hours.

Oxygen pressurized hydrogen peroxide bleaching

Pressurized peroxide (PO) bleaching was conducted in a 650 ml stainless steel vessel, which was equipped with a gas inlet and a stirrer. The stabilizer chemicals and the reagent were added to the pulp (15 g o.d.) in the order listed below:

(a) Magnesium sulphate solution, as 0.039 M solution (0.5% o.d. pulp)
(b) Sodium hydroxide standardized solution, as 0.33 M solution (2% o.d. pulp)
(c) Hydrogen peroxide solution, as 35% v/v solution (0.2% o.d. pulp)

Distilled water was added to the pulp slurry to reach a total weight of 150 g (10% consistency). The slurry was well mixed and then placed in the autoclave. After flushing the air inside the autoclave for 5 s, the pressure inside the autoclave was brought to 690 kPa and kept at this level as the temperature was raised to 100 °C with constant stirring speed (about 250 rpm). The pulp was maintained in the autoclave at 100 °C for 60 min.

Chelation

Chelation was done by two methods, Q_1 and Q_2. In the Q_1 first step the pH was adjusted to 3 using sulphuric acid. The pulp was sealed in a plastic bag and immersed in a water bath at 50 °C for 30 min in order to dissolve the transition metals in the pulp. In the second step, the pH of the acidified pulp was raised to 5 using sodium hydroxide and DTPA (1% o.d. pulp basis) was added. The mixed slurry was placed in a sealable plastic bag and heated in a water bath at 50 °C for 30 min. The pulp consistency was maintained at 3% during treatment.

In the Q_2 method, pulp was treated with EDTA (0.5% o.d. pulp basis) at 3% pulp concentration in sealed polyethylene bags maintained in a water bath at 70 °C for 90 min. The pH of the pulp was adjusted to 4.5–5.

ECF bleaching

The conditions of chlorine dioxide (D), alkaline extraction (E), peroxide (P) and hypochlorite (H) stages, which were used in the ECF sequences are tabulated in Table 1. After adding the bleaching liquor, the pulp (20 g o.d.) was stirred evenly and distilled water was added to the slurry to reach the consistency stated in Table 1. The mixed slurry was then placed in a heat-sealed polyethylene bag and heated in a water bath. The pulp mass was thoroughly mixed by manual kneading.

Determination of bleached pulp properties

Brightness, selectivity and pulp yield were used in the determination of the optimum bleaching sequence. These properties were measured after each bleaching stage.

The Kappa numbers, viscosity and brightness of pulps were determined according to TAPPI Test Methods (TAPPI 2002) and TAPPI Useful Methods (TAPPI 1991). The yields were calculated based on the difference between the unbleached and fully bleached pulp weights.

The determination of viscosity was performed as per TAPPI T230 om-99—viscosity of pulp (capillary viscometer method). The preparation of handsheets followed TAPPI procedure T218 sp-02 — forming handsheets for reflectance testing of pulp. The brightness (% ISO) of handsheets was measured with a Technibrite Micro TB-1C instrument using a 457 nm filter. Selectivity can be expressed as a function of both the Kappa number drop and the loss of viscosity:

$$\text{Selectivity} = \frac{1 - \dfrac{K_f}{K_i}}{1 - \dfrac{h_f}{h_i}}$$

where K_f = Kappa number after bleaching
K_i = initial Kappa number
h_f = intrinsic viscosity after bleaching
h_i = initial intrinsic viscosity

Table 1 ECF bleaching conditions

Stage	Chemical	Charge (% o.d. pulp)	Consistency	Temp (°C)	Time (min)
D_1	ClO_2	2	10	60	120
D_2	ClO_2	1.5	10	60	90
E	NaOH	1.5	8	80	60
H	NaOCl	1.5	10	38	90
P	H_2O_2	1.5	10	90	90

Handsheet making and paper evaluation

Handsheets of 80 g m^{-2} were made according to TAPPI T205 sp-02. Ten pieces of handsheets were tested for physical testing in terms of burst index, tear index, tensile index, stretch and tensile energy absorption as per TAPPI procedure (TAPPI 2002). Prior to the physical–mechanical tests, pulps were refined in a PFI mill. Refining of whole kenaf pulps was conducted carefully at a low number of revolutions (500, 1000, 1500 and 2000) because of the short fibre component and the slow draining characteristics of these pulps.

Results and discussion

Single- and multi-stage bleaching

Table 2 shows that single application of chemicals has a limited effect on brightness improvement and delignification. The brightness of the single-stage (D or O) bleached pulp was still very low and Kappa number was high compared with conventional ECF and TCF fully-bleached pulps.

Multi-stage applications of bleaching chemicals provided greater benefits. The inter-stage washing, which removes dissolved bleaching by-products (e.g. oxidized lignin) is partially responsible for improvement in the extent and efficiency of bleaching. In addition, multi-stage sequences take advantage of the different reactivities of each chemical towards different bonds in the lignin and provide synergy in bleaching and lignin removal.

Since the partially bleached OZ and OQP pulps were found to contain high residual lignin, rather extreme bleaching conditions were necessary in order to completely remove the residual lignin. In these cases, an oxygen-pressurized hydrogen peroxide (PO) operating at a higher reaction temperature (100 ∞C) was employed instead of the normal atmospheric pressure hydrogen peroxide bleaching. The potential brightening benefit from pressurized peroxide bleaching is realized because the increased temperature allows the peroxide bleaching reaction to proceed at a faster rate. If the lignin content in the peroxide stage is fairly high, such as after an oxygen stage, the addition of oxygen will allow delignification reactions to proceed along with the peroxide delignification and brightening reactions.

Efficient brightness increase in the peroxide stage depends on chelation of traces of transition metals in the pulp. Hence, chelation was performed before the first stage of peroxide bleaching. It was assumed that there may be some residual transition metal ions present in the pulp fibre after the first stage of chelation. Hence, the pulp was chelated again before the second peroxide stage. The inclusion of the second chelation stage resulted in a significant increase in the efficiency of the second peroxide stage (Table 2).

Table 2 The properties of whole kenaf bleached pulp using TCF and ECF techniques

Bleaching sequence	Kappa No.			Viscosity (cP)			Selectivity	Yield (%)	Brightness (% ISO)
	Before	After	% Drop	Before	After	% Drop			
O	15.5	10.4	32.9	22.2	39.1	32.9	0.84	98.1	44.8
OZ	15.5	9.1	41.3	22.2	43.2	41.3	0.96	97	55.1
Q_1(PO)	15.5	6.2	60.0	22.2	43.2	60.0	1.40	96.3	71.4
OQ_1P	15.5	4.9	68.4	22.2	50.4	68.4	1.36	94.3	75.6
Q_1(PO)P	15.5	3.1	80.0	22.2	50.9	80.0	1.57	92.9	85.7
OZQ_1P	15.5	2.8	81.9	22.2	45.9	81.9	1.78	92.6	84.5
Q_1(PO)Q_2P	15.5	1.1	92.9	22.2	47.7	92.9	1.95	91.7	90.4
D	15.5	7.6	51.0	22.2	36.0	51.0	1.42	97.1	56.7
D_1ED_2	15.5	3.7	76.1	22.2	38.7	76.1	1.97	93.5	73.4
D_1ED_2EH	15.5	1.6	89.7	22.2	45.5	89.7	1.97	90.7	82.2
D_1ED_2EP	15.5	0.8	94.8	22.2	42.3	94.8	2.24	93.9	88.9

Values are means of three replicates.
Unbleached pulps had ISO brightness of 24%.
O = oxygen, Z = ozone, P = hydrogen peroxide, PO = pressurized peroxide, D = chlorine dioxide, E = alkaline extraction, H = hypochlorite

Pulp brightness

A single stage of oxygen and chlorine dioxide can raise the brightness by 20 and 32% units respectively, without affecting the yield. However, these values are well below the acceptance brightness level (normally 80–90% ISO) for printing paper manufacture. To achieve this high level of brightness, multi-stage bleaching sequence is required.

Brightness obtained with TCF sequences was higher than that obtained with the ECF sequences (Table 2). This is the opposite of what normally happens with bleaching of wood pulps, whereby it is more difficult to reach the commercially important 90% brightness level using TCF sequences. This relative ease of bleaching kenaf kraft pulps with TCF sequences does not appear to have been reported. This is a significant advantage to producers of kenaf kraft pulps who may be able to offset some of the additional costs of slower paper machine speeds (as a result of lower freeness of kenaf pulps compared with wood pulps). The ability to bleach to higher brightness with TCF sequences is associated with lower effluent treatment costs. The highest brightness (90.4% ISO) was obtained for the pulp treated with Q_1(PO)Q_2P sequence. This is about 1% unit higher than the brightness of the D_1ED_2EP bleached pulp. The use of peroxide in the last stage, in both methods, provided a higher level of brightness. Pulp brightness increased with increased chemical charges. Three sequences of TCF [(Q_1(PO)P, OZQ_1P, Q_1(PO)Q_2P] and two sequences ECF (D_1ED_2EH, D_1ED_2EP) could raise the brightness to an acceptable level for manufacture of printing papers.

The combined application of only 0.2% hydrogen peroxide plus oxygen [Q_1 (PO)] had a much higher impact on increasing the brightness (71.4% ISO) than bleaching using oxygen alone (44.8% ISO). The potential brightening benefit from pressurized peroxide bleaching was also realized in practice.

Catalytic decomposition of hydrogen peroxide by metal ions, such as potassium, manganese and copper, is well-known. Hence, the hydrogen peroxide was stabilized with sodium silicate and chelating agents (DTPA and EDTA). A comparison of the results of the Q_1(PO)P (85.7% ISO) and Q_1(PO)Q_2P (90.4% ISO) sequences showed that the use of the second chelation stage (Q_2) increased pulp brightness by almost 5 units. Therefore, a second stage of chelation is very important in removing the remaining transition metal ions from kenaf pulps, thus improving the peroxide treatment.

Selectivity and viscosity

Selectivity is a good indicator for determining effectiveness of a bleaching method, while retaining strength properties of pulp. Table 2 shows that ECF bleaching has higher selectivity than TCF bleaching. This was supported by the viscosity data, where the viscosities of TCF bleached pulps were lower than those of the ECF bleached pulps. This may be attributable to the intensive use of hydrogen peroxide in the TCF processes, as it is well-known that peroxide can decompose to give free radicals that cause cellulose depolymerization (Anderson & Amini 1996). On the other hand, ozone is a powerful oxidizing agent that oxidizes and solubilizes most of the residual lignin. However, it also reacts indiscriminately with all organic materials including carbohydrates. During ozone bleaching, many carbonyl groups are formed, which make the cellulose more susceptible in an alkaline medium to rapid degradation and therefore a decrease in viscosity, as recorded in Table 2, was not unexpected. Reduction in viscosity by ozone can cause a decrease in the physical–mechanical properties of paper (Roncero *et al.* 2002). The ability of chlorine dioxide to oxidize unsaturated carbon bonds selectively in the presence of carbohydrates makes it very efficient at lignin removal and pulp brightening. The selectivity values of D_1ED_2EP and $Q_1(PO)Q_2P$ sequences were 2.24 and 1.95 respectively.

Viscosity is a measure of the average degree of polymerization of the cellulose in pulp and consequently can indicate the degree of carbohydrate degradation during pulping and bleaching (MacLeod *et al.* 1994). Although the viscosity results obtained in ECF and TCF bleachings were apparently good, there was no guarantee that the mechanical strength of the paper produced from those pulps would be acceptable. Ryynanen *et al.* (1995) found that with chemical pulp there is no firm relationship between viscosity and handsheet strength properties. In order to confirm that the fully bleached paper strength properties were acceptable, the physical–mechanical properties of the ECF and TCF bleached pulps were investigated after pulp refining.

Pulp yield

Pulp yield decreased as the bleaching sequence progressed (Table 2). The highest and lowest yield belonged to single O and D_1ED_2EH respectively. The use of multi-stage bleaching in both methods caused a considerable reduction in bleached pulp yield. The slight difference between the yield of ECF (D_1ED_2EP) bleached pulp (93.9) and that of the TCF [$Q_1(PO)Q_2P$] pulp (91.7) was not significant.

Preliminary screening

The results of this preliminary screening showed that the whole stem pulp could be bleached to a high brightness using TCF and ECF processes. Only two TCF sequences [OZQ_1P and $Q_1(PO)Q_2P$] and two ECF sequences (D_1ED_2EH and D_1ED_2EP) were able to obtain high ($\geq$ 80% ISO) brightness with a relatively good yield, Kappa number and viscosity. Although it is also possible to achieve the desired brightness with other bleach sequences at lower pulp viscosities and strengths, for producing high quality printing paper high ($\geq$ 80% ISO) brightness along with high physical–mechanical properties are required.

In order to focus on pulps produced by the most promising processes, those sequences leading to low brightness ($\leq$ 80% ISO) and low viscosity ($\leq$ 1.5 cP) were not examined further. Hence, for the next sequence of experiments, only the four bleached pulps listed above were used for refining, making handsheets and testing.

Paper properties

Tensile strength is an important property, especially for printing papers, and is dependent on the fibre strength and strength of inter-fibre bonding (Page 1969). It can be greatly improved by refining the pulp because this procedure promotes fibre collapse and flexibility, generates fines and increases inter-fibre bonding. The fine content of pulp contributes to the bonding of fibres by bridging the gap between the networks of long fibres. The improved bonding enhances the strength properties of the paper.

No significant differences were observed between the various bleaching sequences within the TCF and ECF groups. However, there were significant differences between TCF and ECF pulps (Figure 1). The tensile strength results obtained from TCF pulps were slightly lower and were in accordance with the lower viscosity values obtained for these pulps.

Considering the importance of tensile index and its close relationship with pulp freeness, all other properties were expressed relative to tensile index. There were no significant differences between the ECF bleaching sequences evaluated. The same trends were observed for all TCF sequences. From these results, it is possible to infer that the TCF [Q_1(PO)Q_2P] bleached pulp properties were similar to those of ECF (D_1ED_2EP) sequence.

With an increase in refining and also tensile index, there was a decrease in tear index (Figure 2). With increased refining, the fibres were extensively cut and weakened, decreasing their tearing resistance. The lowest values of tear index were achieved at about 95–100 N m g^{-1} tensile index. These were equivalent to 230–270 CSF for both ECF and TCF pulps, which corresponded to 2000 PFI mill revolutions. TCF bleached pulps had slightly superior values to ECF pulps.

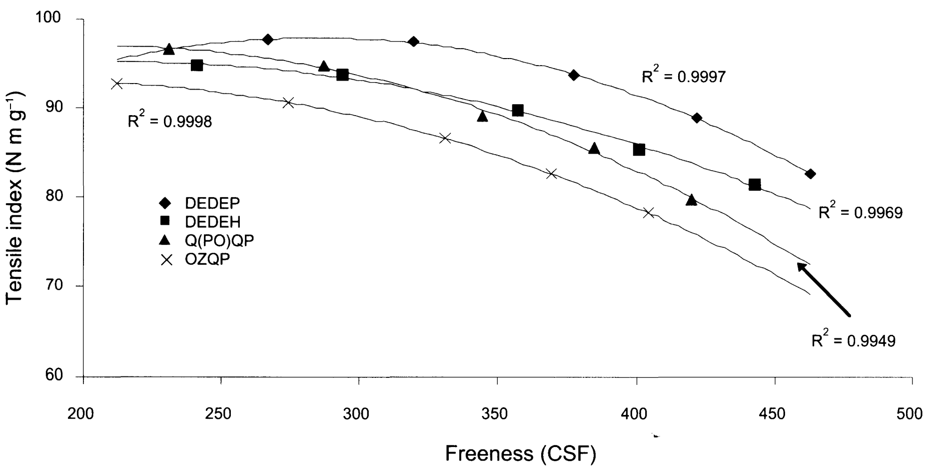

Figure 1 Variation of tensile index with bleached pulp freeness

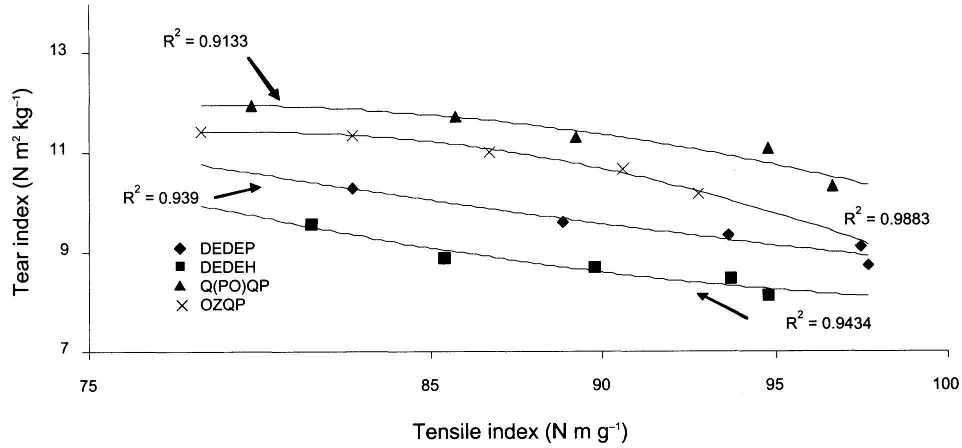

Figure 2 Variation of tear index with tensile index

Pulp refining enhances fibre collapse and promotes a greater contact area between fibres. This leads to higher values of burst index for both ECF and TCF pulps (Figure 3). ECF bleached pulps showed slightly superior burst results to the TCF pulps. From a statistical viewpoint, the type of ECF and TCF bleaching sequences did not affect burst index.

The properties of the TCF [Q_1(PO)Q_2P] and ECF (D_1ED_2EP) bleached pulps were comparable, with the exception of tear index and tensile index, which were slightly higher and lower for TCF pulps respectively. Moreover, TCF [Q_1(PO)Q_2P] bleached pulps could be easily refined to good strength levels.

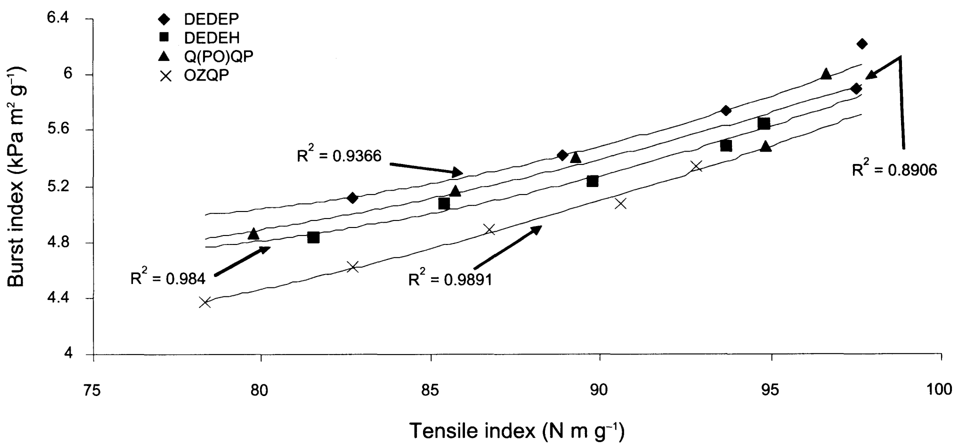

Figure 3 Variation of burst index with tensile index

Conclusions

Kraft kenaf pulps could be easily bleached to a brightness of 91.4% using a four-stage TCF bleaching sequence. The TCF [Q_1(PO)Q_2P] sequence is simple and only two main stages of bleaching are involved using only one major bleaching chemical, together with two chelation stages. The strength properties of the TCF [Q_1(PO)Q_2P] bleached pulp and the ECF (D_1ED_2EP) bleached pulp were not significantly different. In addition, TCF [Q_1(PO)Q_2P] bleached pulp could be easily beaten to good strength levels.

There was no definite relationship between viscosity and handsheet strength properties. Although the viscosities of TCF pulps were lower than those of the ECF bleached pulps, the strength properties were similar. Thus, the papermaking properties of pulps should be measured rather than viscosities when pulp quality for papermaking is being evaluated.

Acknowledgements

The authors thank Mohd Nor Mohd Yusoff of the Forest Research Institute Malaysia and Wan Rosli Wan Daud of Universiti Sains Malaysia for technical support.

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