BLEACHING ALKALINE PULPS FROM PINUS RADIATA

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ABSTRACT

Oxygen-delignified kraft, kraft-anthraquinone (kraft-AQ), and soda-AQ pulps from **Pinus radiata** D. Don were bleached with oxygen-based and chlorine-based treatments. Oxygen-based bleaching employed ozone (Z), alkali extraction (E), and peroxide (P) treatments in previously optimised ZEP and ZPZP sequences. Chlorine-based bleaching was with a conventional sequence. Comparative information was gathered on the effects of bleaching on pulp yield, viscosity, beating requirements, handsheet strengths, and brightness stability.

In general, the responses of the three alkaline pulps were similar for each bleach sequence though bleached soda-AQ pulps had slightly lower brightnesses. The yield advantages of AQ-additive pulping were preserved after full bleaching. Except for lower chemical demands with ZPZP bleaching, no noticeable differences were found between the effects of ZEP and ZPZP bleaching. Both sequences reduced pulp viscosity, pulp beating requirements, and handsheet tear and burst properties. In comparison, chlorine bleaching resulted in higher pulp viscosity and increasing pulp strength levels mainly because of enhanced handsheet tear properties, but had no effect on beating requirements and resulted in less stable brightnesses.

INTRODUCTION

Anthraquinone (AQ) addition to alkaline pulping has proved to be an effective and practical method of accelerating delignification and improving pulp yields. For kraft pulping, AQ addition has provided flexibility by allowing reductions in active alkali, sulphidity, and/or H-factor, any of which may be a problem area in a particular mill (Holton 1977a; Fossum *et al.* 1980a). For soda pulping, AQ addition has increased pulp yields and strengths to levels similar to those of kraft without the use of troublesome sulphur compounds (Farrington *et al.* 1977; Fossum *et al.* 1980b; Holton 1977b).

The specific effects of AQ addition during alkaline pulping are species-dependent as recent studies have shown. Some of these studies have also reported on the bleachability of AQ-additive pulps from the following softwood species: southern pine (*Pinus taeda* L.) (Blain 1980), Scandinavian [*sic*] pine (*Pinus sylvestris* L.) (Fossum *et al.* 1980a; 1980b), and various Canadian softwood species (Blain 1980; Holton 1977b; MacLeod *et al.* 1981). With the exception of Canadian black spruce (*Picea mariana* (Mill.) B.S.P.), several common trends were observed.

(1) Bleaching did not affect the yield advantages gained after AQ-additive pulping;

(2) Compared to control kraft pulps, the amounts of bleach chemical required to reach high brightness were similar for kraft-AQ pulps and greater for soda-AQ pulps;

(3) Bleached pulp strengths for kraft-AQ pulps were equivalent to the control kraft pulps while those for soda-AQ pulps were significantly less.

For black spruce pulps, kraft-AQ pulp strengths were intermediate to those of kraft and soda-AQ pulps both before and after bleaching (MacLeod *et al.* 1980). Bleach chemical demands for kraft-AQ pulps from black spruce were also greater than those for control kraft pulps (Liebergott & van Lierop 1981).

The present investigation was one in a series undertaken to characterise the response of alkaline pulps from *P. radiata* to AQ addition and subsequent delignification and bleaching with oxygen-based pulp treatments (Allison 1981 and in prep.; Fullerton & Kerr 1981). It was previously reported that AQ addition during kraft pulping of *P. radiata* increased pulp yields and pulping rates but to a lesser extent than that reported for other softwood species (Fullerton & Kerr 1981), and had little effect on handsheet properties.

Soda pulping of *P. radiata* with addition of 0.1 to 0.2% AQ on wood resulted in delignification rates and pulp yields at 30 Kappa number similar to those for kraft pulping (Allison 1981). More importantly, the handsheet strengths of *P. radiata* soda-AQ pulps, as measured by tear/tensile relationships, were considerably less than those of corresponding kraft pulps.

Oxygen delignification of *P. radiata* alkaline pulps was studied recently with the aim of maximising the degree of lignin removal without significantly affecting pulp strength properties (Allison 1981). Relative to kraft and kraft-AQ pulps, extended oxygen delignification of soda-AQ pulps (>50% lignin removal) resulted in less selective delignification, lower brightness at a given Kappa number, and pulps of lower handsheet strengths. However, with good operational control, oxygen delignification of all alkaline pulps from *P. radiata* could be safely extended to at least 60% lignin removal before significant strength losses occurred.

In additional studies, various ozone and peroxide bleach sequences were examined to determine which combinations of treatments and reaction conditions resulted in efficient bleaching to 85% brightness with maximum viscosity retention (Allison in prep.). Sequences with two ozone stages were more efficient for pulp brightening than those with one. Viscosity data gave no indication of the selectivity trends of these bleach sequences.

Evaluation of alkaline pulps from *P. radiata* after optimised bleaching is reported here. Kraft, kraft-AQ, and soda-AQ pulps at 43 Kappa number were oxygen delignified to 15 Kappa number then bleached to about 85% brightness with a one-ozone-stage sequence (ozone (Z), alkali extraction (E), and peroxide (P) treatments = ZEP sequence), a two-ozone-stage ZPZP sequence, and a conventional chlorine-based sequence (sequential chlorine dioxide / chlorine, alkali extraction, and chlorine dioxide treatments = D/CED sequence).

Analysis of pulping, oxygen delignification, and bleaching results, as well as determinations of pulp yields and handsheet properties, allowed the following to be compared:

(1) The bleach responses of kraft, kraft-AQ, and soda-AQ pulps after similar oxygen delignification;

(2) The effectiveness of oxygen-based *versus* chlorine-based bleaching;

(3) The selectivity of single-ozone-stage (ZEP) *versus* double-ozone-stage (ZPZP) bleaching.

EXPERIMENTAL

Pulping

Commercial chips of *P. radiata* at 462 kg/m^3 basic density were screened in the laboratory prior to pulping in a 10-*l* Weverk rotary digester.

Pulping conditions are presented in Table 1. A proprietary preparation of a 50% suspension of AQ in water was used. Pulps were defibred at approximately 2% consistency with a propellor stirrer operating at 1425 revs/min for 10 min. Defibred pulps were screened through a 0.25-mm slotted Packer flat screen and total yields and screened rejects were determined.

Oxygen Delignification

Oxygen delignification was performed according to procedures outlined previously (Allison 1981). Fluffed pulp at 25% consistency was oxygen treated for 30 min at approximately 118°C maximum temperature with appropriate applications of NaOH (Table 2). Maximum oxygen pressure was 690 kPa and addition of MgO complex was 0.1% MgO on pulp.

Bleach Treatments

Ozone treatments were performed according to laboratory procedures described elsewhere (Allison in prep.). Prior to ozonation, pulps were soaked at pH 3.0 and 1% consistency for 15 min. They were then dewatered to 35% consistency and double fluffed. For two-ozone-stage bleaching, 65% of the total ozone was applied in the first stage and 35% in the second stage. Alkaline extractions were performed with appropriate amounts of NaOH at 10% consistency and 70°C for 60 min. Standard peroxide treatment conditions were 0.4% diethylenetriaminepenta-acetic acid (DTPA), 15% consistency, 70°C, and 180 min, and 1% total H_2O_2 was applied. For two-peroxide-stage bleaching, 60% of the total peroxide was applied in the first stage and 40% in the second stage. Adequate NaOH was applied to reach an initial pH of 11.7.

For sequential chlorine dioxide / chlorine treatments, total available chlorine applied was equal to (0.22) × (Kappa number) with 30% of the total as chlorine dioxide. Chlorine dioxide was applied for 2 min at 5% consistency and 20°C, and was followed by chlorine applications for 63 min at 3% consistency and 20°C. About 10% of the total available chlorine applied was unreacted after 65 min. Chlorine dioxide treatments were performed at 10% consistency and 70°C for 120 min. Reaction pH was maintained

TABLE 1—Pulping conditions and results

Pulp type	AQ charge (%)*	Active alkali (% Na_2O)	Sulphidity (%)	Kappa number	Total pulp yield (%)*	Screenings (%)*	Brightness (%)	Viscosity (mPa.s)
Kraft	0	14.7	24.0	45.2	50.2	1.8	24.9	30.6
Kraft-AQ	0.05	14.0	25.0	43.4	50.3	1.9	24.0	37.6
Soda-AQ	0.16	13.7	0	42.3	49.8	1.2	23.8	23.5

Standard conditions: 1000 g.o.d. wood
4 : 1 liquor to wood
177°C max. temperature
90 min to max. temperature
60 min at max. temperature

* Based on oven-dried wood

TABLE 2—Oxygen delignification conditions and results

Pulp type	Applied NaOH (%)*		Temperature (°C)		Yield (%)		Kappa No.		Brightness (%)	Viscosity (mPa.s)
	Calc.	Actual	Initial	Max.	Oxygen*	Total†	Oxygen	Δ%		
Kraft/O_2	4.0	5.2	105	118	94.2	47.2	15.4	65.9	39.7	17.0
Kraft-AQ/O_2	4.0	5.3	106	116	94.6	47.6	15.9	63.4	38.4	20.3
Soda-AQ/O_2	4.0	5.4	105	118	93.5	46.6	14.7	65.1	36.9	16.9

Standard conditions: 25% consistency
30 min reaction time
0.1% MgO
690 kPa O_2

* Based on o.d. pulp

† Based on o.d. wood

at between 3.5 and 4.0 by additions of NaOH during the course of the treatment. After the final stage in all bleach sequences, pulps were treated with SO_2-water at pH 5.5 at 1% consistency and 20°C for 15 min.

Pulp Evaluation

Yield was determined by duplicate consistency measurements from samples dried for 4 hours in a 105°C dry oven. Kappa number was determined by a half-scale modification of Appita standard method P201 M-59 and viscosity was determined according to Tappi standard method T-230. Brightness was determined on samples formed according to Tappi standard method T-218 and measured at 457 nm with a Zeiss "Elrepho" reflectometer on an absolute basis. Handsheet properties were determined according to standard Appita methods on pulps beaten in a PFI mill at 10% consistency with an applied load of 1.8 N/mm. Comparisons between handsheet brightness and the brightness of standard samples gave some indication of brightness stability since pulps had aged from 1 to 2 weeks between these two measurements.

RESULTS AND DISCUSSION

Pulping and Oxygen Delignification

Pulping and oxygen delignification results were in general accord with earlier findings (Allison 1981; Fullerton & Kerr 1981). Anthraquinone additions to kraft pulping of *P. radiata* resulted in reductions in active alkali and enhanced total yields when pulping to a given Kappa number (Table 1). Soda-AQ pulping also showed a slight yield/Kappa number advantage over conventional kraft pulping.

Extended oxygen delignification (~ 65% Kappa number reduction) of similar kraft, kraft-AQ, and soda-AQ pulps resulted in final Kappa numbers of 15.4, 15.9, and 14.7 respectively (Table 2). Treatment yields were similar for the three types of alkaline pulp (~ 94% on pulp). Brightness response and viscosity reduction were greater for the kraft and kraft-AQ pulps than for the soda-AQ pulp.

Bleach Response

Oxygen-based ZEP and ZPZP bleaching was performed at optimum conditions as determined previously (Allison in prep.). Though ozone and peroxide bleaching was able to achieve over 85% brightness if sufficient ozone was applied, ozone applications were minimised here since excessive ozone treatments can adversely affect pulp strengths (Perkins 1978). Variations in ozone application levels made comparisons difficult between the three alkaline pulps, although they generally responded similarly to ozone and peroxide bleaching with respect to brightness (Table 3). The total yield advantages of AQ-additive pulping were preserved after oxygen-based bleaching. Both kraft-AQ and soda-AQ pulps had total yields of about 44.5% *versus* 43.6% total yield for bleached kraft pulps. Viscosity reductions after oxygen-based bleaching were similar for the three types of alkaline pulps.

There were no consistent differences in yield or viscosity between pulps bleached with either one ozone stage (ZEP) or two ozone stages (ZPZP) (Table 3). However, the

TABLE 3—Bleaching conditions and results

Pulp type	Sequence*	Total chemicals applied (%)†					Yield (%)		Brightness (%)	Viscosity (mPa.s)
		H_2O_2	O_3	NaOH	Cl_2	ClO_2	Bleach†	Total‡		
Kraft/O_2	ZPZP	1.0	0.90	1.9	—	—	92.4	43.6	83.3	8.6
	ZEP	1.0	1.21	3.0	—	—	92.4	43.6	84.2	8.1
	D/CED	—	—	2.2	2.4	1.2	93.6	44.2	88.6	16.5
Kraft-AQ/O_2	ZPZP	1.0	0.86	1.9	—	—	93.1	44.3	79.5	11.0
	ZEP	1.0	1.12	3.0	—	—	94.9	45.2	83.4	10.2
	D/CED	—	—	2.2	2.5	1.2	93.8	44.6	87.7	16.0
Soda-AQ/O_2	ZPZP	1.0	0.89	1.9	—	—	95.8	44.6	85.7	7.1
	ZEP	1.0	1.15	3.0	—	—	96.0	44.7	83.0	8.6
	D/CED	—	—	2.2	2.3	1.2	96.2	44.8	86.9	13.7

* See Experimental section for details

† Based on o.d. pulp

‡ Based on o.d. wood

ozone and alkali demands of the ZPZP sequence were about 25% and 36% less, respectively, than those of the ZEP sequence when bleaching to a given level of brightness (Allison in prep.).

Chlorine-based D/CED bleaching was more easily controlled and consistently surpassed the target brightness of 85% (Table 3), but AQ-additive pulps were somewhat more resistant to D/CED bleaching and final brightnesses were in the order kraft > kraft-AQ > soda-AQ. Differences were small and reflect brightness differences in the original oxygen-delignified pulps (Table 2). Again, total yield was lowest for the kraft pulp.

Ozone and peroxide bleaching resulted in more stable brightness (Table 4); ZEP- and ZPZP-bleached pulps lost 2.5 to 5.1 points of brightness while D/CED-bleached pulps lost 7.1 to 8.5 points of brightness. The superior stability of ozone- and peroxide-bleached pulps was due to final peroxide treatments which are well known for resulting in stable brightness (Singh 1979).

TABLE 4—Differences between handsheet brightness and the brightness of standard pads

Bleach sequence	Pulp type	Std. pad brightness (%)	Handsheet brightness @ 2000 PFI rev* (%)	Brightness difference (%)
ZPZP	Kraft	83.3	78.2	5.1
	Kraft-AQ	79.5	77.0	2.5
	Soda-AQ	85.7	82.3	3.4
ZEP	Kraft	84.2	80.0	4.2
	Kraft-AQ	83.4	80.0	3.4
	Soda-AQ	83.0	79.5	3.5
D/CED	Kraft	88.6	81.5	7.1
	Kraft-AQ	87.7	79.2	8.5
	Soda-AQ	86.9	78.4	8.5

* Pulp stored from 1 to 2 weeks at 4°C prior to handsheeting

Beating Requirements

The development of handsheet density with PFI mill beating was examined for unbleached, oxygen-delignified, and fully bleached alkaline pulps from *P. radiata* (Fig. 1). Unbleached kraft, kraft-AQ, and soda-AQ pulps required similar amounts of PFI beating to develop a given level of handsheet density. Both oxygen delignification and ozone and peroxide bleaching reduced the amount of PFI beating required for a given

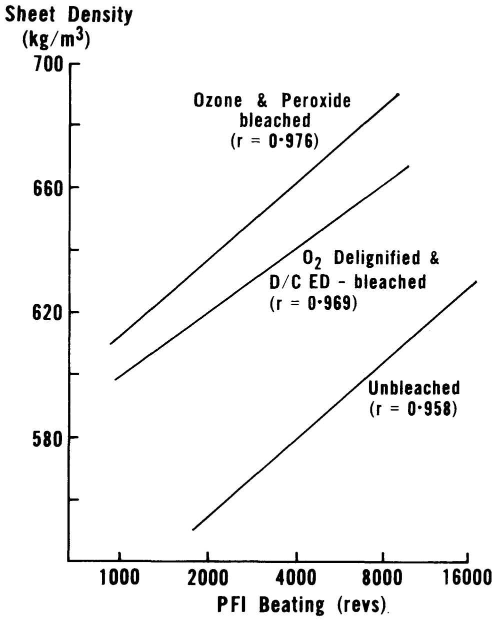


FIG. 1—Development of sheet density with PFI beating for kraft, kraft-AQ, and soda-AQ pulps.

level of handsheet density. For example, at 4000 PFI revs handsheet densities of unbleached, oxygen-delignified, and ozone- and peroxide-bleached pulps were 580, 640, and 660 kg/m^3 respectively. Chlorine-based bleaching had no effect on beating requirements. Similar trends were evident in the development of freeness with PFI mill beating.

As indicated by the slopes of the regressions in Fig. 1, the rate of handsheet density development was similar for all pulps. Thus oxygen delignification and ozone and peroxide bleaching reduced beating requirements by making the unbeaten fibres more flexible and collapsible.

Handsheet Strengths

Tear/burst relations are considered a valid basis for comparing over-all levels of pulp strength, especially for pulps prepared by different methods (MacLeod 1980). In Fig. 2 tear/burst relations for unbleached, oxygen-delignified, ZPZP-bleached, ZEP-bleached, and D/CED-bleached pulps are presented for each of the pulps. From analysis of variance among individual tear/burst relations, single regressions are presented where differences were not significant at the 5% level.

There were no statistical differences between the tear/burst relations of unbleached kraft, kraft-AQ, and soda-AQ pulps (Fig. 2). These results differed from those of previous studies which have shown soda-AQ pulps to have lower tear/burst relations than kraft and kraft-AQ pulps from *P. radiata* (Allison 1981). Though the unbleached soda-AQ pulp in the present study possessed exceptional strength, its tear and burst properties were within the limits of accepted variability for handsheet evaluations of a given type of pulp.

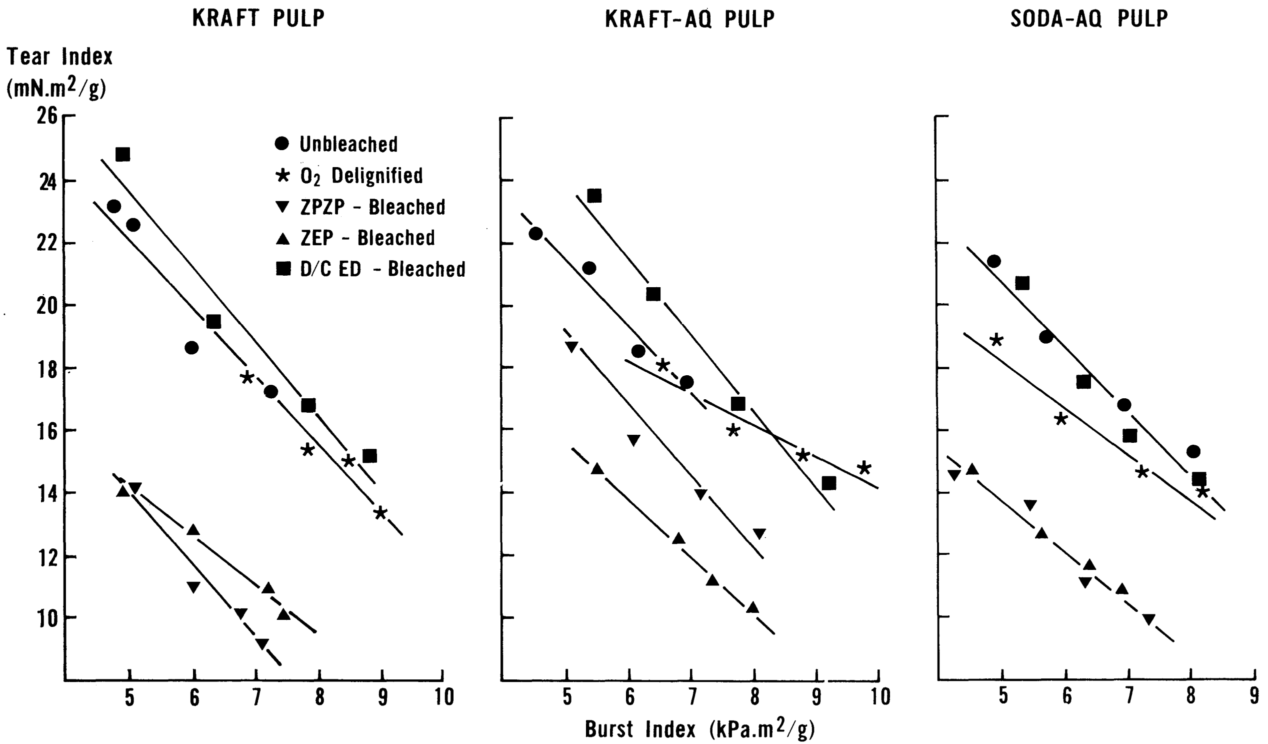


FIG. 2—Tear/burst relations for kraft, kraft-AQ, and soda-AQ pulps

Extended oxygen delignification of kraft and kraft-AQ pulps caused no significant changes in pulp strength (Fig. 2). However, the soda-AQ pulp was more sensitive to extended oxygen delignification and tear/burst relations were significantly reduced. At 7.0 $kPa.m^2/g$ burst index, tear indices after oxygen delignification were 17.1 $mN.m^2/g$ for the kraft and kraft-AQ pulps and 15.4 $mN.m^2/g$ for the soda-AQ pulp. These results are in agreement with those of earlier work on extended oxygen delignification of alkaline pulps from *P. radiata* (Allison 1981).

Both oxygen-based bleach sequences significantly reduced pulp strength (Fig. 2). The extent of strength loss was similar after either ZPZP or ZEP bleaching, but poor control of ozone treatments made comparisons between different alkaline pulps difficult. In general the strength of kraft and kraft-AQ pulps after ozone and peroxide bleaching were lowered to about the same level as those of the bleached soda-AQ pulps, i.e., ozone and peroxide bleaching had a negative levelling effect. At 7.0 $kPa.m^2/g$ burst index, tear indices normally ranged from 10.0 to 12.0 $mN.m^2/g$.

The effects of chlorine-based bleaching on alkaline pulps were consistent and positive – D/CED bleaching enhanced tear/burst relations to levels above those of the oxygen-delignified pulps (Fig. 2). For kraft and kraft-AQ pulps, this resulted in bleached pulps with strength properties superior to the initial unbleached pulps. For soda-AQ pulps, it brought bleached pulp strengths back up to the level of the initial unbleached pulps.

To gain more insight into the effects of different treatments on pulp properties, tear and burst indices *versus* sheet density were plotted to show effects at a given degree of sheet consolidation. A typical example is presented in Fig. 3 to illustrate the effects of oxygen delignification, oxygen-based bleaching, and chlorine-based bleaching on the soda-AQ pulp.

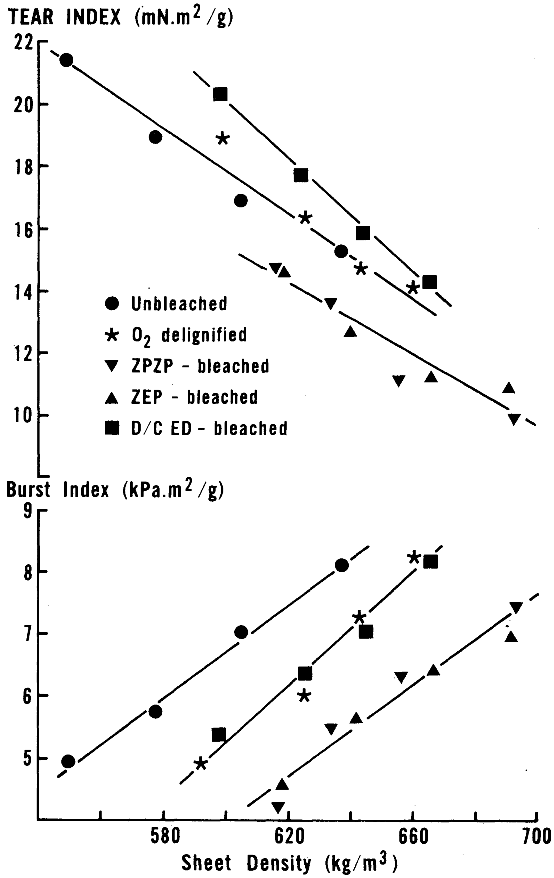
In agreement with previous work, extended oxygen delignification had no effect on tear index at a given sheet density though it reduced burst index by up to 18% (Allison 1981). Upon further bleaching with oxygen-based sequences, both tear and burst indices were greatly reduced at a given sheet density. Reductions in tear and burst indices at 620 kg/m^3 were 15% and 23% respectively. This trend was apparent for all alkaline pulps bleached with ozone and was further evidence of the severe degradative effect that ozone treatments can have on softwood pulps.

After chlorine-based bleaching, tear index at a given sheet density was markedly improved while the burst index was unchanged in comparison to the properties of the oxygen-delignified pulp (Fig. 3). At 620 kg/m^3 sheet density, tear index rose by 9% to 18.2 $mN.m^2/g$.

Viscosity

In order to correlate handsheet strengths with pulp viscosity, tear index at 7.0 $kPa.m^2/g$ burst index was plotted *versus* pulp viscosity (Fig. 4). In general, correlations were inconsistent. Below 20 mPa.s, handsheet strengths decreased for most pulps except those bleached with a D/CED sequence. These pulps seemed to establish their own regression at a level higher than that of the unbleached and oxygen-treated pulps. Thus

FIG. 3—Tear and burst indices as a function of sheet density for soda-AQ pulps.



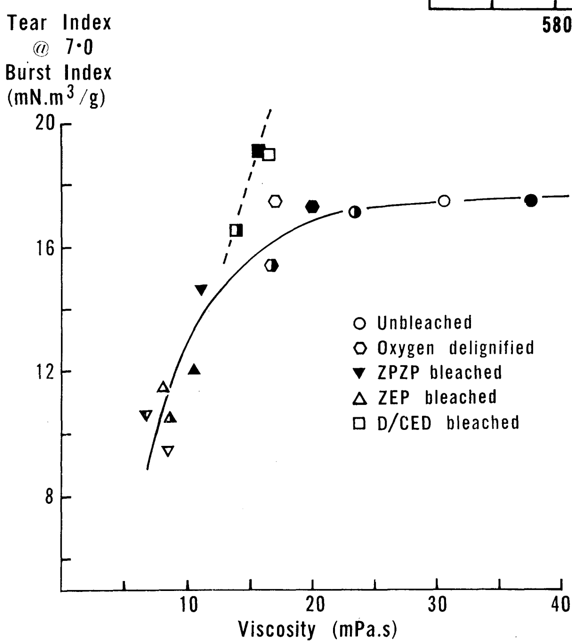


FIG. 4—Handsheet strength as a function of viscosity for alkaline pulps from **P. radiata** (open symbols for kraft pulps; filled symbols for kraft-AQ pulps; half-filled symbols for soda-AQ pulps).

pulp viscosity here was an unreliable indicator for strength comparisons between pulps with different treatment histories. Others have also made this observation (Allison 1981; Kubes *et al.* 1980).

General Discussion

One objective of this study was to determine the level of pulp strengths after pulping and bleaching with sulphur- and chlorine-free methods, e.g., with soda-AQ pulping followed by oxygen delignification and ozone and peroxide bleaching. Unfortunately this produced pulps significantly weaker than either unbleached kraft pulps or kraft pulps bleached with conventional chlorine-based sequences. Ozone and peroxide bleaching caused most of the losses in pulp strength. Even kraft and kraft-AQ pulps were severely affected by ozone and peroxide bleaching and losses in tear/burst relations were from 20% to 40%.

Though not proven directly, it was concluded that ozone treatments were the degradative cause of strength losses during oxygen-based bleaching. Before oxygen-based bleaching involving ozone treatments becomes viable, methods must be found to inhibit the attack of ozone on pulp carbohydrate fractions and thus improve the over-all selectivity of the treatment. Although dividing ozone applications into two stages in this study enhanced bleaching efficiency (i.e., reduced total ozone), it had no effect on selectivity when bleaching to a given brightness level. Recently, more promising methods to improve selectivity were reported, such as lowering pulp consistency (Kempf & Phillips 1978) and employing various organic additives and pretreatments (Mbachu & St. John Manley 1981).

CONCLUSIONS

The responses of oxygen-delignified kraft, kraft-AQ and soda-AQ pulps from *P. radiata* to ZEP, ZPZP, and D/CED bleaching were similar except that the soda-AQ pulp had slightly lower brightnesses, especially after bleaching with the more closely controlled D/CED sequence. The total yield advantages of AQ-additive pulping were preserved after full bleaching.

Except for lower chemical demands with the ZPZP sequence, there were no significant differences between the bleach response and pulp strength effects of ZEP *versus* ZPZP bleaching. Both sequences substantially reduced pulp viscosity and diminished handsheet tear and burst properties of oxygen-delignified alkaline pulps. On the other hand, oxygen-based bleaching further reduced pulp beating requirements.

In comparison, conventional chlorine-based bleaching with a D/CED sequence resulted in higher treatment yields and pulp viscosities plus increased pulp strength levels owing to increases in handsheet tear properties, but had no effect on beating requirements and resulted in less stable brightnesses.

Oxygen-based bleaching with ozone treatments caused unacceptable losses in pulp strength and further work is required to develop effective methods of improving the selectivity of ozone treatments.

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