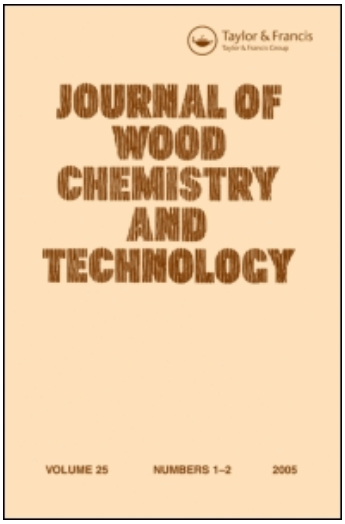

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Separate Bleaching of Pulp Fractions Enriched in Earlywood and Latewood Fibers

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Abstract: Unbleached softwood kraft pulp was fractionated in hydrocyclones into a fine fraction, enriched in earlywood fibers, and a coarse fraction, enriched in latewood fibers. The response to individual bleaching chemicals and the bleachability in bleaching sequences of the pulp fractions was studied.

The light absorption coefficient, light scattering coeffient, and brightness were higher in the unbleached fine fraction than in the coarse fraction. Hydrogen peroxide managed to reduce the light absorption coefficient of the earlywood enriched fine fraction more efficiently than that of the latewood enriched fraction.

In the TCF-sequence the light absorption coefficient of the fine fraction was reduced to the level of the coarse fraction at a given consumption of bleaching chemicals. In the ECF-sequence the difference in light absorption coefficient remained between the fractions. At a given consumption of bleaching chemicals, the fine fraction had

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higher brightness than the coarse fraction, 2%- and 1%-units on the ISO-scale in the TCF and ECF bleaching sequence, respectively.

Keywords: Softwood, bleaching response, latewood fibers, earlywood fibers, hydrocyclone, fractionation

INTRODUCTION

The fiber morphology varies between species as well as within individual trees. The thin-walled earlywood fibers and thick-walled latewood fibers in softwoods, for example, differ considerably in characteristics. The fiber morphology determines the final paper properties to a large extent.[1,2]

To use the full potential of the wood raw material, the differences in the morphology of latewood and earlywood fibers as well as chemical differences between different morphological parts is needed to be taken into account. For instance, the earlywood fibers have a higher proportion of the lignin located in the middle lamella compared to the latewood fibers with a more ligninfied fibre wall.[3]

In a previous study, handcut spruce veneer was chipped into nearly ideal fractions of earlywood and latewood fibers and the separate fractions delignified by kraft cooking and subsequently oxygen delignified.[4] The oxygen delignified earlywood kraft fibers had a higher light absorption coefficient at a given kappa number than the latewood oxygen delignified kraft fibers. That difference in light absoption coefficient was maintained when bleaching with chlorine dioxide, ozone, and peracetic acid, whereas hydrogen peroxide decreased the light-absorbing structures in the earlywood to the same level as for latewood. Earlywood fibers had a higher brightness due to a larger number of fibers in the sheet even though the colored material in the pulp, measured as light absorption coefficient, was higher. [4] Dai et al.[5] studied the bleachability of earlywood and latewood fibers from handcut chips of loblolly pine in a ECF-bleaching sequence D(E_{OP})DED. They found that latewood kraft pulp had lower bleachability than the earlywood kraft pulp. Nevertheless, their study was based on evaluating the bleachability as ISO-brightness at given bleaching chemical consumption. ISO-brightness, however, does not distinguish between the contributions from light scattering and light absorption ability. Light scattering depends more on the morphology of the fibers whereas the chemical composition, in particular colored substances, influences the light absorption coefficient.

In the present study, the objective was to investigate the bleachability of pulp fractions of more realistic raw material than spruce veneer. Hydrocyclones were used for fractionating an unbleached softwood kraft pulp. Hydrocyclones are known to fractionate unrefined fibers, primarily according to cell wall thickness. This will produce a fine fraction enriched in earlywood fibers and a coarse fraction enriched in latewood fibers.[6–8]

MATERIAL AND METHODS

Raw Material

The starting material was an industrially produced kraft pulp from spruce and pine chips, composed of 70% sawmill chips and 30% roundwood chips. The kappa number of the pulp was 24.

Fractionation

A multistage hydrocyclone fractionation system was used to fractionate the pulp into a fine and a coarse fiber fraction, Figure 1. Furthermore, two different types of hydrocyclones were used to reach a high degree of earlywood and latewood enrichment of the two respective fractions. Fractionation step F1 and F2 used a hydrocyclone type particularly capable of concentrating earlywood in the fine fraction. The highly diluted fine fraction was dewatered on a bow screen. The coarse fraction was collected using a 76 μm wire mesh. Approximately 55% of the pulp originally fed into the fractionation system was discarded. Tap water was used for dilution.

The process conditions in the different fractionation steps are given in Table 1.

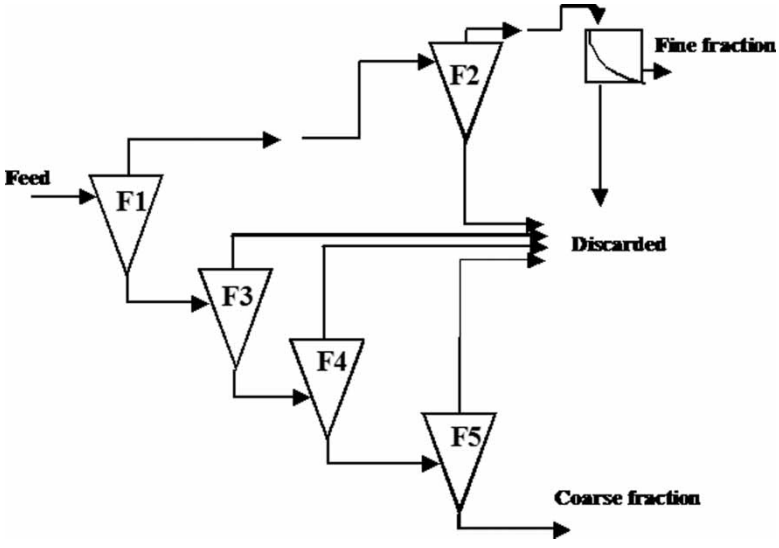

Figure 1. Fractionation scheme. The fine fraction becomes enriched in earlywood fibers and the coarse fraction in latewood fibers.

Table 1. Process conditions of the hydrocyclone fractionation

	F1	F2	F3	F4	F5
Feed flow [l/min]	181	156	339	339	340
Feed consistency [g/l]	3,2	0,7	2,4	2,0	1,8
Feed pressure [kPa]	248	180	251	249	250
Pressure drop (Feeds-Fine) [kPa]	200	140	204	204	202
Mass reject rate [%]	76	40	74	76	77

Bleaching

Bleaching response: Steel autoclaves were used for oxygen delignification, a rotating glass reactor for ozone treatments and sealed polyethylene bags in thermostatic water baths for complexing agent (Q), chlorine dioxide (D), hydrogen peroxide (P), and peracetic acid (Paa) stages. Deionized water was used in all bleaching experiments. The pulp samples were treated according to sequences OQ, DQ, ODQ, OZQ, OQPQ, OQPaaQ. The conditions in the bleaching stages are given in Table 2.

Bleachability: The two fractions were bleached in a TCF-sequence, OQ*(OP)PaaQ*(PO) and an ECF-sequence: OD(E + P)DED Table 3. Q*

Table 2. Bleaching conditions in the bleaching response study

Bleaching stage	Charge, kg/t	Temp, °C	Pressure, MPa	Time, min	End pH	Pulp consistency, %
O-stage	12–24 (NaOH) 5 ($MgSO_4$)	100	0.7	35–90	10.2–11.4	12
Q-stage (1 and 2)	2,7 (DTPA)	90	—	60	ca 5.5	10
D-stage (no O_2 prior to D)	24–55 (act. Cl)	50	—	45	2.4–3.0	8
D-stage	10–28 (act. Cl)	50	—	45	2.9–4.5	8
Z-stage	2–8,7 (ozone)	20	—	3	2.9–3.4	40
P-stage	5–50 (peroxide)	80	—	22 h	10.1–11.0	10
Paa-stage	4,3–28 (Paa)	70	—	60	5.4–5.9	10

Table 3. Bleaching conditions in stages in bleaching sequences

Bleaching stage	Charge, kg/t	Temp, °C	Pressure, MPa	Time, min	End pH	Pulp consistency, %
Q*	2.7 (DTPA) 1.0 (Mg^{2+})	90	—	60	5.5	10
(OP)	5 (H_2O_2) 2 (NaOH)	100	0.5	120	10.5	10
Paa	5 (H_3C-COOOH) 2 (NaOH)	70	—	60	5	10
Q*	2.7 (DTPA) 0.5 (Mg^{2+})	90	—	60	5.5	10
(PO)	5–60 (H_2O_2) 9–18 (NaOH)	110	0.5	120	10.5–10.9	10
D_0	24 (a Cl)	50	—	45	2.5	8
(E + P)	3 (H_2O_2) 8.4 (NaOH)	60	—	60	11.5	10
D_1	8.5–32 (a Cl) 1–3 (NaOH)	70	—	120	3.8–4.1	10
E	2.5	70	—	60	11.4	10
D_2	4–16 (a Cl) 0–0.3 (NaOH)	70	—	240	3–3.7	10

denotes that magnesium sulphate was added. The bleachability was determined as the consumption of OXE in the bleaching sequences. The concept of OXE, oxidation equivalents, was used to be able to compare the different bleaching chemicals. It is defined as the amount of oxidation chemical that consumes 1 mol of electrons when it is reduced.[9]

Analyses

Wood, dry content SCAN-CM 39:88; pulp, dry content ISO 638; kappa number SCAN-C 1:77R; viscosity ISO 5351; brightness ISO 2470; metal analysis ICP-AES; amount of fines, BDDJ (Britt Dynamic Drainage Jar) Tappi T 261 cm-90 with a sieve of 76 μm; carbohydrate analysis according to Theander and Westerlund.[10]

The amount of earlywood and latewood fibers in pulp was counted microscopically. A fiber is classified as latewood when the lumen was less or equal with two fiber wall thickness. Everything else is classified as earlywood fibers.

The fiber dimensions were analyzed with STFI FiberMaster.[11] The bendability value was obtained by measuring the shape factor at two velocity rates.

The total fiber charge was determined by conductometric titration. The surface charge was determined by electrolyte titration using polyDADMAC (Mw $>$ 920 000 and charge density 5.8×10^{-3} ekv/g)[12]

Light absorption coefficient was determined at 457 nm on a single sheet toward two different backgrounds. The light-scattering coefficient at 557 nm according to ISO 9416:1998.

RESULT AND DISCUSSION

Pulp Fractionation

With the purpose to determine the potential of separate bleaching of earlywood and latewood fibers, the goal of the fractionation was to achieve pulp samples consisting of as pure separate fractions of earlywood or latewood fibers as possible. Fractionation by hydrocyclones does not give a perfect separation; each fractionation step gives a coarse fraction enriched in latewood fibers and a fine fraction enriched in earlywood fibers compared to the feed pulp.

Table 4 shows the amounts of earlywood and latewood fibers in the fractions chosen for the bleaching study. The fine fraction had as much as 81% earlywood fibers. In the coarse fraction, the amount of latewood fibers had been raised from 39% in the original pulp to 62%. Primary fines have been shown to affect the bleachability as their content of lignin is higher and the lignin structures less reactive.[13,14] In order to eliminate the influence of fines, the pulps were screened prior to the bleaching study. Nevertheless, a minor fraction of the fines remained in the pulps.

Fiber Dimensions

Table 5 presents some characteristics of the fractions. According to FiberMaster analysis, the fibers in the fine fraction were shorter than the fibers of

Table 4. Data from microscopic counting of earlywood and latewood fibers in the three categories of pulps. The pulps were screened prior to analysis and further experiments. The fines content, determined as BDDJ, after screening is also shown.

	Earlywood fibers, %	Latewood fibers, %	Fines, %
Fine fraction	81	19	1.5
Coarse fraction	38	62	0
Original pulp	61	39	3.0

Table 5. FiberMaster analyses of fiber dimensions and shape factors

	Length, L_w mm	Width, μm	Shape factor, %	Bendability
Fine fraction	2.66	36.0	85.7	7.41
Coarse fraction	2.85	35.7	85.3	3.34

the coarse fraction, in accordance with previous studies.[15,16] The fine fraction fibers were also much more flexible as seen from the bendability values. The shape factor was practically the same for the two fractions and the level is typical for industrially pulped fibers.

Chemical Composition

The analysis of the carbohydrate composition, presented in Table 6 shows that earlywood fibers had more xylan and less glucomannan than the latewood fibers. In studies of earlywood and latewood fibers separated by hydrocyclones, similar results have been observed[15,18] although it is contradictory to the study on veneer, were it was found that the earlywood pulp had less xylan and more glucomannan than the latewood pulp.[4] The different results may be attributed to different cooking conditions. In the veneer study, the separation into earlywood and latewood fibers was made prior to pulping and the latewood fibres required a higher alkali charge than the earlywood fibers to reach the same kappa number. In the case of fractionation by hydrocyclones, earlywood and latewood fibers were cooked under the same conditions and separated after pulping.

The hexenuronic acid content was about the same in fine and coarse fraction, 25 and 23 μmole/g, respectively, in accordance with Tormund et al.[4] whereas Nordborg[18] found that the earlywood fraction had a higher amount of hexenuronic acids than the latewood fraction.

The earlywood fibers had a higher content of barium, magnesium, and manganese Table 7. The extremely higher amount of copper and iron in the

Table 6. Carbohydrate composition, (calculated according to Jansson.[17]) The cellulose content of the fibers is practically the same, whereas the proportion of hemicelluloses differs

	Carbohydrate composition in pulp, %		
Pulp Kappa no. 22–24	Xylan	Glucomannan	Cellulose
Fine fraction	7.6	8.8	80.4
Coarse fraction	6.8	9.9	80.2
Original pulp	7.2	9.3	80.2

Table 7. The metal content of the fractions and the original pulp

Metal content, mg/kg	Al	Ba	Ca	K	Mg	Mn	Cu	Fe
Fine fraction	15	4.1	1600	16	290	30	48	33
Coarse fraction	18	2.2	1500	12	230	17	100	130
Original pulp	11	2.9	1300	19	220	28	28	53

coarse fraction should be attributed to the fractionation process. The fibers in this fraction have gone through several cyclones and most likely these metal ions have been picked up from the equipment and process water by the pulp.

The earlywood fibers have more accessible surface area per weight, due to lower coarseness, which probably explains the higher surface charge compared to latewood fibers Table 8. The total charge is higher as well for the earlywood fibers, possibly related to the higher xylan content.

Bleaching Response Study

The bleachability of the two fractions was determined first by a bleaching response study and then by bleaching to full brightness in TCF- and ECF-sequences. In the bleaching response study, the fractions were subjected to treatment with one bleaching agent at a time and the increase in brightness and decrease in light absorption coefficient was monitored. Table 9 gives the characteristics of the pulp samples used for bleaching. Noticeable is the large difference in light scattering between the fine and coarse fraction. The high light scattering coefficient of the earlywood fibres is due to a larger number of fibers, providing more light scattering surface, than sheets made of latewood at equal grammage.

Oxygen delignified pulp samples were treated with different bleaching agents. The response, as an increase in brightness, is shown in Figure 2. To reach certain brightness, the coarse fraction generally required a higher charge of bleaching agent. The most pronounced difference was achieved by hydrogen peroxide, in accordance with our previous study.[4]

Different bleaching chemicals favor different lignin structures.[19] Chlorine gas and ozone react mainly with aromatic structures, chlorine dioxide, and

Table 8. Charge density in pulp determined as total and surface charges

	Total charge, μmole/g	Surface charge, μmole/g
Fine fraction	76.1	4.2
Coarse fraction	72.6	2.4
Original pulp	74.0	3.2

Table 9. The characteristics of pulp used for the bleaching study. The low viscosity after the O-stage was due to the high metal ion content. Prior to the bleaching sequence study, the pulps were subjected to two Q-stages in order to remove metals

Pulp for oxygen bleaching	Kappa number	Viscosity, ml/g	Light absorption coeff., m^2/kg	Light scattering coeff., m^2/kg	Bright-ness, % ISO
Fine fraction	24	1000	25	28	30
Coarse fraction	22	1060	21	22	27
Pulp for bleaching study after O-stage					
Fine fraction	14	810	17	27	38
Coarse fraction	13	800	14	23	37
Pulp for bleaching sequence after QQO-stage					
Fine fraction	15	860	14	28	42
Coarse fraction	13	900	12	24	39

oxygen degrade lignin with free phenols, whereas hydrogen peroxide reacts with carbonyls and aliphatic double bonds. Because structures with double bonds generally absorb light, hydrogen peroxide efficiently eliminates chromophores. This is illustrated in Figure 3, showing light absorption coefficient at

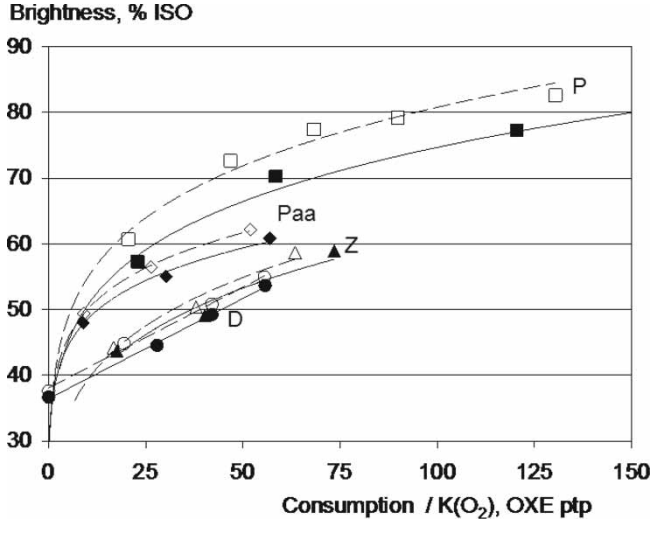

Figure 2. Earlywood fibers = unfilled symbols; latewood fibers = filled symbols. At a certain consumption of bleaching chemical, the fine fraction reached a higher brightness when treated with peroxide and peracetic acid. The kappa number of earlywood and latewood pulp varied slightly (see Table 9) so the consumption of OXE is given as OXE/Kappa number of oxygen delignified pulp.

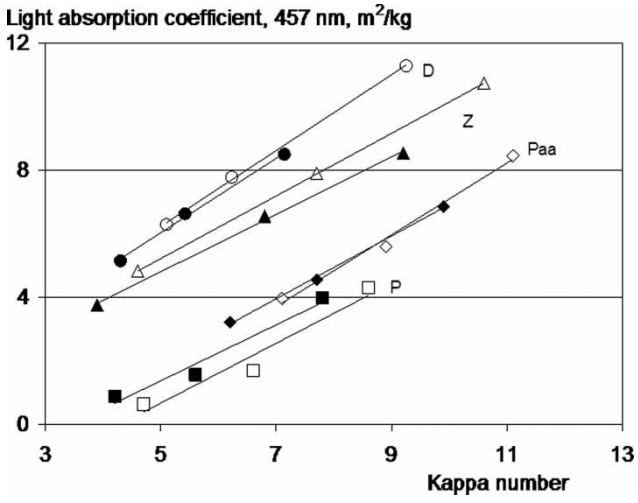

Figure 3. Llight absorbing coefficient at different kappa numbers for the studied bleaching chemicals. Earlywood fibers = unfilled symbols latewood fibers = filled symbols.

different kappa numbers for the studied bleaching chemicals. At a given amount of lignin, measured as kappa number, the chlorine dioxide treated pulps had the highest amount of light absorbing structures; that is, the highest light absorption coefficient. Hydrogen peroxide, on the other hand, had the lowest light absorption. This can be interpreted as hydrogen peroxide, both reduced light absorbing structures as well as by delignifying the pulp whereas chlorine mainly delignifies. These results are in accordance with our previous investigation.[4] The light absorption coefficient of the oxygen delignified fine fraction was higher than the light absorption coefficient of the coarse fraction, 14 and 12 m^2/kg, respectively. Of additional interest is that hydrogen peroxide managed to decrease the light absorption coefficient of the earlywood enriched fine fraction to a lower level compared to the coarse fraction. It has been shown earlier that hydrogen peroxide bleaches thinwalled fibers more efficiently and reduces their carbonyl content to a substantially higher extent compared to thick-walled fibers.[15]

Bleaching Sequences

The aim of the bleaching response study was to monitor the effect achieved by specific bleaching agents, but to reach full brightness a complete bleaching sequence has to be applied. The fractions were bleached in ECF- and TCF-sequences OD(E + P)DED and OQ*(OP)PaaQ*(PO), respectively.

The brightness of the fine fraction was higher at a given consumption of bleaching chemicals compared to the coarse fraction for both types of sequence (Figure 4) To reach brightness 89% ISO, the fine fraction required 20 kg OXE ptp less than the coarse fraction in the ECF bleaching. In the TCF sequence, the fine fraction required 35 kg OXE ptp less than the coarse fraction to reach brightness 86%. In an earlier study by Dai et al.[5] where bleaching was performed according to D(E_{OP})DED, they found that earlywood fibers required less chlorine dioxide to reach a given brightness.

The reduction of light-absorbing structures was monitored through the bleaching sequences. The coarse fraction had a lower light absorption coefficient to begin with and this remained through the ECF-sequence (Figure 5) Through the TCF-sequence, the light absorption coefficient was the same for the different pulp fractions. The explanation is probably an effect of the main bleaching agent, hydrogen peroxide, which, according to the bleaching response study, reduced the light absorption coefficient of the earlywood enriched fine fraction substantially more compared to the coarse fraction. The main bleaching agent in the ECF-sequence, chlorine dioxide, mainly delignifies and is not as capable as hydrogen peroxide to eliminate chromophores, thus the originally higher light absorption coefficient of the fine fractions remained.

The viscosity of the oxygen bleached pulp, that was the starting material for the TCF- and ECF-bleaching, was 40 units higher for the coarse fraction. In the ECF-sequence, this difference remained up to brightness levels of

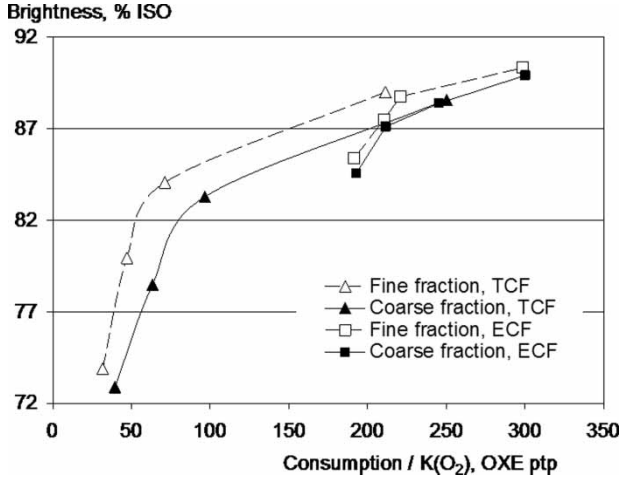

Figure 4. The brightness increased at a given consumption of OXE. Earlywood fibers = unfilled symbols; latewood fibers = filled symbols. The kappa number of earlywood and latewood pulp varied slightly (see Table 9) so the consumption of OXE is given as OXE/kappa number of oxygen delignified pulp.

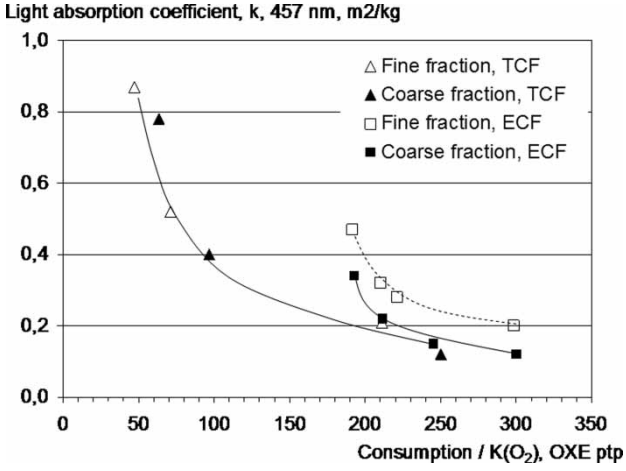

Figure 5. At a given chemical consumption, the amount of light absorbtion coefficient was lower in the coarse fraction. Earlywood fibers = unfilled symbols; latewood fibers = filled symbols. The kappa number of earlywood and latewood pulp varied slightly (see Table 9) so the consumption of OXE is given as OXE/kappa number of oxygen delignified pulp.

approximately 89% (Figure 6). In the TCF-bleaching, the greater reduction of light-absorbing structures in the coarse fraction corresponded to a higher reduction in viscosity, that is, the viscosity of the coarse fraction has dropped down to the level of the fine fraction.

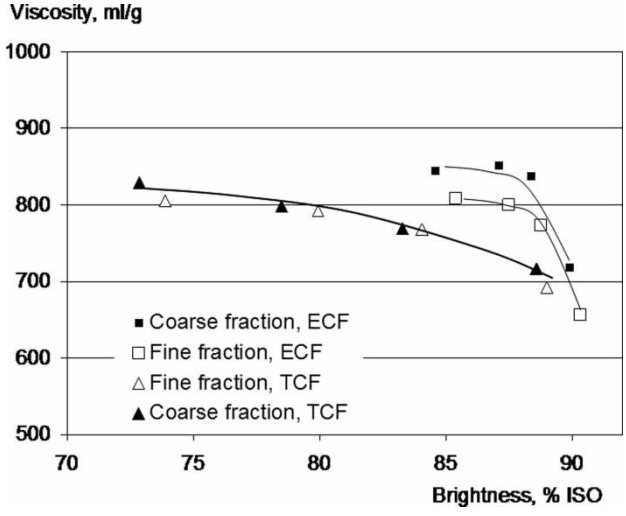

Figure 6. The selectivity in the ECF-sequence and TCF-sequence. Earlywood fibers= unfilled symbols; latewood fibers = filled symbols.

CONCLUSIONS

The results from this study on hydrocyclone-fractionated unbleached softwood pulp showed that although the earlywood enriched fine fraction had a higher light absorption coefficient, it nevertheless had higher brightness than the latewood enriched coarse fraction. This is a consequence of the higher light scattering coefficient of the fine fraction. These results stress the importance of considering the fiber morphology when studying the bleachability of fibers with different structural features or from different raw materials.

The different bleaching chemicals studied, chlorine dioxide, peractic acid, ozone and hydrogen peroxide, had different capabilities to eliminate light-absorbing structures in pulp, measured as light-absorption coefficient. Pulp treated with chlorine dioxide had the highest light absorption coefficient at a given kappa number, followed by ozone and peractic acid. The light absorption coefficient of the earlywood enriched fine fraction decreased to the same level as that of the latewood enriched coarse fraction when chlorine dioxide or peractic acid were employed. Ozonation of the pulps left the fine fraction with a higher light absorption coefficient at a given kappa number. Hydrogen peroxide–treated pulp had the lowest light absorption coefficient at a given kappa number. Additionally, hydrogen peroxide managed to decrease the originally higher light absorption coefficient of the earlywood enriched fine fraction to an even lower level than that of the coarse fraction.

When subjecting the fractions to a complete bleaching sequence, the fine fraction had a higher brightness at a certain consumption of bleaching chemicals, measured as oxidation equivalents, OXE. The fine fraction had approximately 1%-unit higher brightness than the coarse fraction when bleached by the ECF-sequence and 2%-units higher brightness when bleached by the TCF-sequence. The difference in light absorption coefficient between the two fractions remained in the ECF-bleaching sequence, where chlorine dioxide was the main bleaching agent. In the TCF-bleaching, the light absorption coefficient of the fine fraction was reduced down to the same level as that of the coarse fraction. This was a result of hydrogen peroxide's ability to reduce the light-absorbing structures in earlywood more effectively than in latewood.

To reach brightness 89% ISO, the fine fraction in the ECF-bleaching required 20 kg OXE ptp less than the coarse fraction. In the TCF sequence, the fine fraction required 35 kg OXE ptp less than the coarse fraction to reach brightness 86%.

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