PAPER II

Effect of birch kraft pulp primary fines on bleaching and sheet properties

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EFFECT OF BIRCH KRAFT PULP PRIMARY FINES ON BLEACHING AND SHEET PROPERTIES

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By removing the primary fines from an oxygen-delignified mill birch pulp, a fiber fraction was obtained having low metals content and no extractives. After DEDeD bleaching the fiber fraction had somewhat higher brightness and better brightness stability than the birch pulp containing the primary fines. The fines fraction was enriched with lignin, extractives, xylan, and metals. Bleaching the fines fraction in a QQP sequence did not affect the extractives, whereas a ZeQP sequence clearly reduced the extractives content. In a biorefinery concept, the fines fraction could be utilized as a source of xylan, fatty acids, sterols, and betulinol. Another possibility is to use the fines fraction unbleached or separately bleached as a bonding material in various fiber furnishes.

Keywords: Betula; Kraft pulp; Fines; Ozone bleaching; Hydrogen peroxide; Chlorine dioxide; Metal ion; Bonding; Brightness stability; Extractive content

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INTRODUCTION

The use of fractionation techniques is increasing in the pulp and paper industry. One reason is the possibility to remove and separately treat fiber fractions that may contribute negatively to pulp and papermaking. For example, by removing the primary fines before bleaching, closure of the bleaching loop at the pulp mill may be facilitated, since the metals ion content is reduced before bleaching. In papermaking, problems related to stickies, smell and odor caused by extractives could be avoided by removal of the primary fines, since most of the extractives are found in primary fines. If the primary fines are treated separately, and the extractives and metal ions content can be reduced, then the primary fines can be utilized in papermaking.

Primary fines consist of ray cells, some broken fibers, and thin sheets from the fiber surface. Primary fines usually represent between 1 and 3 percent of the o.d. mass of pulp, depending on the wood species. The fines fraction differs from the fiber fraction in that it has higher contents of lignin, metal ions, and extractives (Bäcström and Brännvall 1999; Liitiä et al. 2001; Hinck and Wallendahl 1999; Treimanis et al. 2009; Treimanis 2009).

In birch the majority of the extractives are located inside the parenchyma cells. Birch pulp extractives cause severe problems in pulp and papermaking. Of the birch extractives, betulinol is usually the main component in precipitations or stickies found in both pulp and paper mills. Its melting point is 261 °C; thus it is crystalline through the all stages of the pulp making. Sitosterol can be oxidized to a form that results in a bad smell;

sitostanol is again saturated and stable. Both are found in stickies, although they are not sticky themselves (Holmbom 2003; Back and Allen 2000). Fatty acids are sticky, especially saturated fatty acids in the form of metal soaps, and they have been found to impair the degree of sizing (Lidén and Tollander 2004). Both the fatty acids and sitosterol components can be oxidized, which can result in taste and odor problems. Especially, the unsaturated fatty acids are easily oxidized, leading to volatile bad smelling aldehydes, such as hexanal and nonal (Oyaas 2000). All lipophilic substances, which are enriched on the fiber surfaces, tend to decrease the fiber-fiber bonding ability (Kokkonen et al. 2002).

The objective of the study was to clarify the changes in chemical composition of the pulp by removal of the primary fines from an oxygen-delignified mill birch kraft pulp before bleaching, and how the bleaching chemical consumption and pulp properties are affected using a DEDeD sequence. Also the effects of separate bleaching of the fines fraction using QQP and ZeQP sequences were investigated, especially in order to reduce the extractives content. Finally, the possibilities of utilizing the fines fraction, unbleached or bleached, as a bonding agent for, e.g., chemimechanical pulp, were evaluated.

EXPERIMENTAL

Primary fines (4%) were removed from an oxygen-delignified mill birch kraft pulp (before refining) using KCL's Super DDJ (Dynamic Drainage Jar) equipment, which is composed of a tank with a 200-mesh wire and a mixer. This separation method was chosen since it is easy way in the laboratory scale to separate fibers and fines. In an industrial setup the fines separation would probably consist of pressure screens equipped with small aperture size hole-screen, or with rotating units with augmented action, e.g. VarioSplit (Hinck and Wallendahl 1999).

In this paper the original birch pulp containing primary fines will be called birch pulp, primary fines-free birch pulp will be called fiber fraction, and birch primary fines will be called fines fraction.

Bleaching

The birch pulp and the fiber fraction were bleached in the laboratory using a DEDeD sequence. Bleaching experiments were performed in a sealed plastic jar. The brightness target for the pulps was 88% ISO. The bleaching conditions are shown in Table 1.

The fines fraction was bleached using QQP and ZeQP sequences. Hydrogen peroxide and ozone were charged in such a way that both the sequences had about the same bleaching chemical consumption calculated as OXE (oxidizing equivalents), 1780 OXE/kg. The conditions were as follows:

- Chelation (Q): 70°C, 2 % consistency, 20-30 min, EDTA 0.4-0.5% calculated on dry pulp, initial pH ca. 4.0.
- Hydrogen peroxide stage (P) in QQP sequence: 80°C, 15% consistency, 180 min, NaOH 2%, $MgSO_4$ 0.1%, H_2O_2 4% calculated on dry pulp.
- Ozone stage (Z) in ZeQP sequence: approx. 50°C, 1.4% consistency, initial pH ca. 6.

- P-stage: NaOH 0.88%, H_2O_2 1%, other conditions the same as in QQP.
- e-stage (neutralizing washing stage) in ZeQP sequence: 70°C, 2 % consistency, 10 min, initial pH 7.5-8.0.

Table 1. Bleaching Conditions for the DEDeD sequence

Stage	D0	E1	D1	e*	D2
Consistency, %	9	10	9	3	9
Temperature, °C	50	65	65		70
Reaction time, min	60	60	120	2	180
Final pH	<2.5	10.5-11.0	~4	~9-9.5	~4.5
ClO_2 charge, %	0.2 times incoming kappa number	-			According to the brightness after the D1
NaOH charge, %	-	0.4*ClO_2 charge in D0	0.085*ClO_2 charge in D1	0.4	-

* e-stage, i.e. neutralizing washing stage was performed straight away after the D1-stage.

Analysis Methods

The following analyses were conducted on the birch pulp, fiber fraction and fines fraction:

- Total residual lignin, gravimetric and acid soluble lignin. (KCL internal method TAPPI T222 modif.).
- Uronic acids were measured using an enzymatic hydrolysis followed by HPLC measurement (Tenkanen et al. 1995; Hausalo 1995).
- Acetone extracts (SCAN-CM 49).
- Wood extractives– free fatty acids, resin acids, lignans, sterols, steryl esters and triglycerides. Pulp sample was freeze-dried and extracted with acetone. The silyl derivative of the wood extractives was analyzed using gas chromatograph with flame ionization detector (GC-FID). The amounts of free fatty acids, resin acids, lignans, sterols, steryl esters, and triglycerides were determined as group sums.
- Carbohydrate composition (TAPPI T249, modif.).
- Polysaccharide composition (Janson 1974).
- Carboxyl group content was determined with the method based on magnesium ion exchange. In principle, the bound magnesium ions are eluted and determined by quantitative analysis.
- Carbonyl group content was determined according to the oxime method. The carbonyl content is related to the nitrogen content as determined by Kjeldahl procedure or elemental analysis.
- Metal content was measured using Inductively Coupled Plasma Atomic Emission spectroscopy (ICP-AES). The samples were dissolved in nitric acid in a microwave oven before the analysis.
- Fines content using a Dynamic Drainage Jar (DDJ) equipped with a 200-mesh (76 μm) wire. Conducted on the birch pulp and the fiber fraction.

- Acetone soluble matter (SCAN-CM 49:03), uronic acid composition (Tenkanen et al. 1995, Hausalo 1995), metal ion content and post color (PC)-number (80°C, 65% RH, 48h according to ISO 5630-3 by UV-Vis reflectance spectroscopy, KCL Internal method, described in (Liitiä et al. 2004) was determined for DEDeD bleached birch pulp and fiber fraction.

RESULTS

Contents of Organic Compounds in the Birch Pulp, Fiber, and Fines Fractions

The fiber fraction and birch pulp had higher cellulose content than the fines fraction, and the fiber fraction was extractives-free (Table 2). The fines fraction had a substantially higher content of lignin, xylan, extractives, metals, and also hexenuronic acid. Also, the content of carbonyl and carboxyl groups was higher in the fines fraction. Higher content of xylan, lignin, and carbonyl groups has also been reported earlier in the fines (Treimanis 2009; Treimanis et al. 2009; Bäckström and Brännvall 1999; Liitiä et al. 2001; Hinck and Wallendahl 1999; Heijnesson-Hulten et al. 1997; Westermark and Capretti 1988). Ray cells are known to be a main source of extractives, and that is the reason for the higher content of extractives in the fines fraction (Heijnesson-Hulten et al. 1997).

Table 2. Chemical Composition of Birch Pulp, Fiber Fraction, and Fines

	Birch pulp	Fiber fraction	Fines fraction
Cellulose, %	71.7	73.8	43.4
Lignin, %			
Gravimetric	<2.0	<2.0	5.6
Soluble	0.6	0.5	0.6
Total			6.2
Xylan, %	26.1	24.6	48.3
Uronic acid composition			
Methyl glucuronic acid, mmol/kg	31	27	27
Hexenuronic acid, mmol/kg	78	69	91
Acetone extract, %	0.31	<0.05	1.55
Carbonyl groups, mmol/100 g	1.6	1.6	3.2
Carboxyl groups, mmol/kg	153.7	148.8	218.2

The fines fraction had a clearly higher content of various extractives components than the birch pulp or the fiber fraction (Table 3). Also, the content of the various extractives components of the fiber fraction, containing in practice no fines (0.4% of DDJ fines), was substantially lower than that of the birch pulp containing 4.6% of DDJ fines. In particular, the content of harmful betulinol, a main component in deposits or stickies found at both pulp and paper mills, was substantially lower in the fines-free fiber fraction.

Also, the contents of fatty acids and sterols were substantially lower, when the fines were removed.

Table 3. Extractives Content of the Birch Pulp, Fiber Fraction, and Fines Fraction, Analyzed from Freeze-Dried Pulps

mg/kg	Birch pulp	Fiber fraction	Fines fraction
Fatty acids	170	26	1100
Betulinol	72	8	460
Lignan	39	10	540
Sterols	190	38	3000
Sterylesters	1200	310	25000
Triglyserides	72	42	760
Total	1700	430	31000

Metal Ion Contents of the Pulps

The fines fraction had a clearly higher metal content than the birch pulp and the fiber fraction (Table 4). A high metal ion content of the fines fraction has also been revealed earlier (Westermark and Capretti 1988; Treimanis 2009). As expected, the fiber fraction had a lower content of metal ions than the birch pulp.

Table 4. Metal Ion Content

mg/kg	Birch pulp	Fiber fraction	Fines fraction
Copper	<0.5	<0.5	5.3
Iron	<3	<3	100
Magnesium	150	140	250
Manganese	100	69	350
Silica	65	42	220
Calcium	1500	1200	3700

Particularly, the content of manganese, silica and calcium was lower in the fiber fraction than in the birch pulp. However, the positive thing was that the content of magnesium, a protector in hydrogen peroxide bleaching, was not much lower in the fiber fraction than in the original birch pulp.

Effect of Fines Removal on the DEDeD Bleaching Efficiency

Higher final brightness at 6% lower active chlorine consumption was obtained for the fines-free fiber fraction compared to the birch pulp. Calculated as active chlorine consumption per kappa unit reduction or brightness unit increase, there were no differences between the pulps, i.e. no difference in bleachability (Table 5).

A slight difference was seen in the brightness stability of the birch pulp and that of the fiber fraction. Brightness values before the aging treatment was about the same for both pulps (Table 6). However, after the aging treatment the birch pulp had a somewhat lower brightness value than the fiber fraction.

Table 5. Kappa Number and Brightness of the Bleached Pulps and Bleaching Chemical Consumption

	Birch pulp	Fiber fraction
Final kappa number	1.0	0.88
Final brightness, %	88.2	88.6
Active chlorine consumption, kg/BDt	46.37	43.60
Active chlorine consumption/Δkappa number, kg/BDt	3.93	3.96
Active chlorine consumption/Δbrightness, kg/BDt	1.07	1.09

Table 6. Brightness Before and After the Aging Treatment, and PC Number (465nm) for 48h, 80°C, 65% Relative Humidity

	Birch pulp	Fiber fraction
Brightness before treatment, %	88.61	88.46
Brightness after treatment, %	75.81	76.50
PC (Post Color) number	3.13	2.86

The PC number was affected by the content of lignin, hemicellulose component/ uronic acids, extractives, and also the metal ions. The birch pulp had higher extractives content, and also higher hexenuronic acid content (although below the determination limit) than the fiber fraction. Also the UV-Vis spectra revealed that the fiber fraction had a lower content of hexenuronic acids and lignin, and also less C=O structures than the birch pulp (Fig. 1).

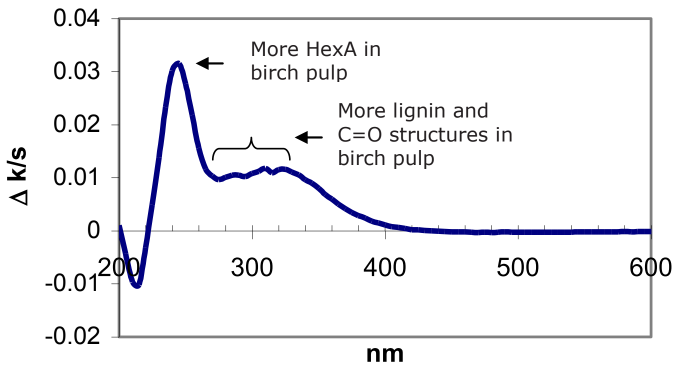

Fig. 1. Difference between the UV-Vis spectra of the birch pulp and the fiber fraction

Effect of the QQP and ZeQP Bleaching of the Fines Fraction on the Extractives

The hydrogen peroxide, QQP bleaching, was unable to remove the extractives from the birch fines fraction although it acts also as an alkaline extraction stage (Table 7).

It is known that the problems with birch extractives have anatomical and chemical explanations (Back and Allen 2000; Sjöström 1981, Laamanen 1984). In birch the majority of the extractives are located inside the parenchyma cells, which have rather small pits. This renders the birch pulp extractives more troublesome than in other hardwoods.

Table 7. Extractives Content of the Fines Fraction and QQP Bleached Fines Fraction

mg/kg	Fines fraction	QQP bleached fines fraction
Fatty acids	1100	2200
Betulinol	460	520
Lignan	540	680
Sterols	3000	3000
Sterylesters	25000	25000
Triglyserides	760	790
Total	31000	32000

Similar to what was earlier found by Laamanen (1984), hydrogen peroxide treatment of birch kraft pulp in the laboratory, with a large dose of 5%, did not change the composition of the extractives. The reason for this was said to be that hydrogen peroxide was unable to penetrate into the (extractives) parenchyma cells. Furthermore, in the alkaline hydrogen peroxide stage, in which there are significant concentrations of Ca^{2+}-and Mg^{2+}- ions, the fatty acids will form metal soaps rather than soluble fatty acids soaps, and due to this the extractives content will not decrease (Fernando and Daniel 2005). In addition, it has been observed that the sterols remaining in bleached pulps are present almost exclusively inside the parenchyma cells (Fernando and Daniel 2005). It is also mentioned in the literature (Fernando and Daniel 2005) that betulinol and saturated fatty acids are very resistant towards oxidation.

The total content of extractives was decreased by ZeQP bleaching from 31000 mg/kg to 19000 mg/kg (Table 8). The extractives content of the fines fraction was about 42% lower after the Ze bleaching than that of the unbleached fines fraction, and after the ZeQP sequence about 40% lower. However, the content of some extractives components after the hydrogen peroxide stage in ZeQP bleaching was about the same or even greater than in the unbleached fines due to the same reasons as in the case of the QQP bleaching.

Table 8. Extractives Content of the Unbleached Fines Fraction, the Fines Fraction after Ze-stage, and the ZeQP Bleached Fines Fraction

mg/kg	Unbleached fines fraction	Fines fraction after Ze	ZeQP bleached fines fraction
Fatty acids	1100	1100	2000
Betulinol	460	370	400
Lignan	540	450	660
Sterols	3000	2200	2500
Sterylesters	25000	13000	13000
Triglyserides	760	760	770
Total	31000	18000	19000

The content of sterylesters was substantially lower after the bleaching than before that. The content of sterols was slightly lower in ZeQP bleached fines than in the unbleached fines. Barbosa et al. (2008) also found ozone to be effective in removal sterols in the bleaching of Eucalyptus kraft pulp.

As in the QQP bleaching of the fines fraction, the content of betulinol, lignan and triglyserides did not either change as a consequence of the ZeQP bleaching.

Birch Pulp Compared to Fines-Free Fiber Fraction

There were no big differences in the sheet properties of the DEDeD bleached birch pulp and fines-free fiber fraction (Table 9).

The light absorption coefficient of the fiber fraction was lower than that of the birch pulp. This was due to the lower lignin content of the fiber fraction than that of the birch pulp. Tensile index and Scott bond of the unrefined birch pulp were higher than those of the unrefined fiber fraction due to the higher fines content of the birch pulp. However, this means that the fiber fraction could be refined to a higher tensile index and Scott bond at a given freeness or SR number.

Table 9. Birch Pulp vs. Fiber Fraction

	Birch pulp	Fiber fraction
Bulk, cm^3/g	1.45	1.48
Air resistance, Gurley, s	1.4	0.8
ISO brightness, %	87.3	87.9
Light scattering coefficient, m^2/kg	31.6	32.1
Tensile index, Nm/g	37.0	32.8
Scott bond, J/m^2	204	157

Utilization of the Birch Fines Fraction

A prerequisite for an industrial realization of removal of the fines fraction from the bleached kraft pulp is to find technically and economically feasible utilization possibilities. One option is to use the fines fraction, either unbleached or separately bleached, as a bonding agent in various fiber furnishes. Good results of using bleached birch fines as a bonding material in paperboard's middle layer has been obtained (Panula-Ontto and Fuhrmann 2007). In this study the possibility to mix the unbleached fines fraction or the QQP and ZeQP bleached fines with a softwood CTMP mill pulp was investigated (Table 10).

The unbleached fines fraction reduced the brightness of the CTMP blend sheet. This was due to the higher light absorption coefficient of the unbleached lignin rich primary fines. Higher bonding ability of fines fractions gave rise to increased tensile index and Scott bond of the CTMP blend sheet. Birch fines settled in the voids of the fiber network, which can be seen as clearly increased air resistance value and lower bulk of the CTMP blend sheet (Table 10).

Both the bleached fines fractions did not affect negatively the brightness of the CTMP. The brightness was even slightly improved in the case of QQP pulp. Hydrogen peroxide is effective bleaching agent in removing of chromophores, which was also seen as lower light absorption coefficient of QQP bleached fines compared to that of ZeQP bleached fines (Table 10).

Birch fines fraction, unbleached or bleached, can be used to increase Scott bond and tensile index values of CTMP-based paper. However, as expected, the bulk was decreased. The additions of the fines fraction were quite high, 10% and 20%, in this study. Lower amounts may be very well added without a negative effect on bulk. Alternatively, coarser mechanical pulp could be used, resulting in an increase of bulk but with still an acceptable bonding.

In addition to their use as a bonding material, birch fines could also be used in a biorefinery concept as a source of xylan, fatty acids, sterols, and betulinol. One possible application could be the adding of birch fines in the softwood kraft cooking. In softwood kraft cooking there is resin acids and also higher content of fatty acid soaps, which could facilitate the carrying the remaining pitch to the pulping liquor. At the same time, the xylan-rich birch fines could improve the strength (tensile stiffness) of the softwood pulp.

Table 10. Sheet Properties of CTMP + Birch Fines

	CTMP	CTMP:unbleached fines		CTMP:QQP fines		CTMP:ZeQP fines	
Furnish composition, %	100	90:10	80:20	90:10	80:20	90:10	80:20
Bulk, cm^3/g	2.80	2.58	2.22	2.47	2.22	2.59	2.40
Air resistance, Gurley, s	2.8	7.7	22.2	7.3	17	5.5	13
ISO brightness, %	68.5	64.5	60.5	69.1	69.7	68.5	68.6
Light absorption coefficient, m^2/kg	0.61	1.18	1.85	0.61	0.62	0.68	0.72
Tensile index, Nm/g	22.5	26.8	31.0	25.0	28.3	24.7	27.4
Standard deviation, Nm/g	0.8	1.2	0.9	1.3	1.1	0.9	0.6
Scott bond, J/m^2	67	98	121	85	112	85	103
Standard deviation,J/m^2	10	2	5	5	15	5	12

CONCLUSIONS

By removing 4% of the fines from a birch mill kraft pulp it was possible to obtain:

- An extractives-free pulp (<0.05% acetone extractives) with low metals ion content.
- A higher final brightness at 6% lower active chlorine consumption in DEDeD bleaching.
- Improved brightness stability after DEDeD bleaching.

RECOMMENDATIONS

Fines fraction could be used

- As a bonding agent, unbleached or bleached, in various fiber furnishes.
- In a biorefinery concept as a source of e.g. xylan, fatty acids, sterols, betulinol.

Fines fraction could be bleached

- To reduce the content of some of the extractives components. Using a ZeQP sequence, the extractives content of the fines fraction was reduced by 40%. However, the amount of extractives remained unaffected when using the QQP sequence. Hydrogen peroxide was more effective in brightening the fines fraction than ozone.
- But the problem is that some of the extractives components like betulinol cannot be removed by bleaching.

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