

Formation of AOX and OCl in ECF bleaching of birch pulp

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ABSTRACT: How does the hexenuronic acid (HexA) in birch kraft pulp influence the formation of chlorinated organic substances in the effluent (AOX) and in the pulp (OCl)? Industrial birch chips cooked to different kappa numbers were oxygen delignified, treated in a hot acid stage, or not pretreated at all before D(EP)D bleaching. The AOX and OCl contents were determined and the filtrate from the first D stage was also stored for 10 days at pH 7 and 35°C, and the AOX levels were determined again. The amount of HexA removed in the first D stage correlated with the unstable part of the AOX formed in this stage, with this unstable part consisting of the AOX that is eliminated during 10 days of storage. This linear relationship is strong evidence that the unstable AOX originates from HexA. The levels of stable AOX and OCl, on the other hand, correlated with the Klason lignin content of the pulp.

Application: Although a hot acid stage reduced the AOX analyzed in the subsequent chlorine dioxide stage, it was not effective in reducing the total stable AOX in the bleaching sequence.

Although mills have substantially reduced the levels of adsorbable organic halogens (AOX) in pulp bleaching effluents during the last three decades, these halogens are still an important environmental concern. For a number of paper products, it is also important to keep a low level of organically bound chlorine (OCl) in the pulp. In elemental-chlorine-free (ECF) bleaching, AOX and OCl are formed when elemental chlorine (Cl_2) or hypochlorous acid (HOCl) reacts and chlorinates organic structures in the pulp [1-3].

In a previous study on softwood kraft pulp, we showed that the AOX formation is highly dependent on the amount of hexenuronic acid or HexA (4-deoxy- β -L-threo-4-enopyranosyluronic acid) in the pulp [4]. The explanation, based on earlier mechanistic studies [5, 6], was that HexA reacts with the elemental chlorine in the chlorine dioxide bleaching stage and that chlorinated structures of low molecular weight are released to the filtrate. We also showed that this part of the AOX was easy to reduce chemically under mild neutral conditions.

Recently, a new low-molecular-weight chlorinated structure originating from HexA was proposed, and previously proposed structures were confirmed in the filtrates from ECF bleaching of eucalyptus and birch [7]. The same study confirmed that the low-molecular-weight AOX was easily eliminated when the filtrates were neutralized. Studies on eucalyptus kraft pulp have also indicated that HexA in the

pulp causes more AOX to form than does lignin [8].

The level of OCl in the pulp seems to depend mainly on the total kappa number and the chlorine dioxide charge and is not as strongly influenced by the HexA content as is the level of AOX [3, 4, 8]. The fact that lignin is the main source of the OCl was shown in a study in which all the OCl was found in the lignin or lignin-carbohydrate complexes of the pulp [3]. However, assuming that the same chlorine species react to form both OCl and AOX, the OCl might be indirectly influenced by the factors affecting AOX formation. Therefore, the chlorination reactions responsible for the AOX and OCl formation are preferably studied together. Another interesting aspect of these chlorination reactions is that they seem to have a positive effect on the bleaching efficiency of chlorine dioxide [3, 9, 10].

These recent studies looking at the formation of chlorinated organic substances from different angles have given us a clearer picture of the influence of pulp components on AOX and OCl formation in the bleaching of both hardwood and softwood kraft pulps. However, in the case of birch pulp cooked to different kappa numbers and submitted to different prebleaching stages (A^* treatment, O_2 delignification), the direct influence of HexA on the formation of organically bound chlorine (AOX and OCl) has not been studied.

Therefore, we have tested birch pulps cooked to different kappa numbers as a continuation of our earlier work [4]. It is also interesting to compare the results from birch pulp with our earlier results from spruce pulp because the formation and the levels of HexA in the pulp during kraft cooking are very different in hardwood and softwood [11-13]. We monitored the pulp composition throughout the bleaching sequence to gain insight into the bleaching and chlorination reactions.

EXPERIMENTAL

Birch chips were taken from a mill and air-dried and screened in the laboratory. The chips were sorted by hand to remove bark. The fraction used in the experiments consisted only of chips that passed through a hole screen of 45 mm and a slot width of 8 mm but not a hole screen of 7 mm. The chips were cooked in a laboratory circulation digester with 36.6% sulfidity to produce two pulps with kappa numbers of 14 and 20. The cooking time was held constant, but the effective alkali and the temperature were adjusted to reach the two kappa numbers.

These two pulps were either oxygen delignified, treated in a hot acid stage, or left untreated prior to ECF bleaching. Here, the pulps with kappa nos. 14 and 20 are denoted "B14" and "B20". Pulps subjected to oxygen delignification are denoted as " O_2 ", and those subjected to a hot acid stage are denoted as " A^* ". A total of six different pulps entered the D(EP)D sequence.

ENVIRONMENTAL CONTROL

Pulp	D		Final pH	(EP)	D		
	Time, min.	aCl, %		Final pH	Time, min.	aCl, %	Final pH
B14	40	2.8	2.9	11.6	140	2.9	3.1
B14-O ₂	45	1.9	2.7	11.3	60	0.3	2.9
B14-A*	45	2.0	2.8	11.2	155	2.1	3.1
B20	40	4.0	2.9	11.2	155	2.9	3.4
B20-O ₂	45	2.5	2.8	11.2	60	0.6	3.0
B20-A*	45	3.3	2.8	11.3	135	2.0	3.5

I. Bleaching conditions in the D(EP)D sequence.

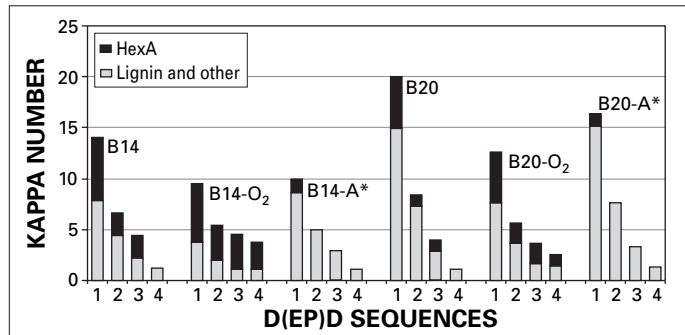
All of the oxygen delignification, hot acid, and bleaching stages were carried out at 10% consistency. The oxygen delignification stages were performed with pulp in plastic bags inside stainless steel autoclaves, and the final pH was between 11.2 and 11.6. All other bleaching stages were conducted with plastic bags. In the hot acid stages, with sulfuric acid (H₂SO₄) at 90°C for 240 min, the final pH levels were between 2.9 and 3.2.

Since our main focus was on evaluating the first D and the (EP) stages of the D(EP)D sequence, we used a constant kappa factor in the first D stage instead of optimizing the distribution of chlorine dioxide between the first D stage and the second D stage. We used a kappa factor of 0.2 (% aCl/kappa no.) and a temperature of 50°C in the first D stage. In the (EP) stage, the temperature was 90°C, the hydrogen peroxide charge was 0.1%, and the time was 60 min. In the second D stage, the temperature was 70°C, and the chemical charge was adjusted to reach a brightness of about 87% ISO. The other conditions in each stage of the D(EP)D sequence are given in **Table I**.

The filtrates from the first D stage were analyzed for AOX before and after storage for 10 days in sealed containers at 35°C and pH 7, an approach we used for the first D(EO) effluent in our previous study [4]. In the study reported here, however, only the effluent from the first D stage was stored, since the AOX in the extraction filtrate remains stable under these conditions [14].

The kappa numbers were determined according to SCAN-C 1:00. The standard deviation of the kappa number for a reference sample with a mean value of 6.2 was 0.08, as determined over a long period of time with this equipment. The ISO 95-62 method was used to determine the amounts of AOX and OCl.

The Klason lignin was determined at STFI, Sweden, and the carbohydrate composition was determined according to AH 83-13 (STFI, carbohydrate composition including uronic acid, enzy-



1. Sets of kappa numbers for the six pulps, with numbers in each set indicating (1) unbleached, (2) the first D stage, (3) D(EP) bleaching, and (4) D(EP)D bleaching.

matic hydrolysis, and capillary electrophoresis). The Klason lignin was not adjusted for the acid-soluble lignin. Dichloromethane extracts of the pulps were analyzed, including a pretreatment with sodium chloride (NaCl) and acetic acid (CH₃COOH) and a subsequent dichloromethane extraction.

RESULTS AND DISCUSSION

Pulp composition and bleachability

During kraft cooking, the xylan side-group 4-O-methylglucuronic acid (MeGlcA) is converted to hexenuronic acid (HexA) [15]. **Table II** shows the amounts of HexA, MeGlcA, and Klason lignin for the birch pulps with different kappa numbers and different pretreatments. As the table shows, the total amount of uronic acid (HexA + MeGlcA) was lower when the cooking was extended to a lower kappa number, but the amount of HexA was higher. This outcome is different from results obtained from softwood pulps, but it confirms recently published results for hardwood pulps [12, 13].

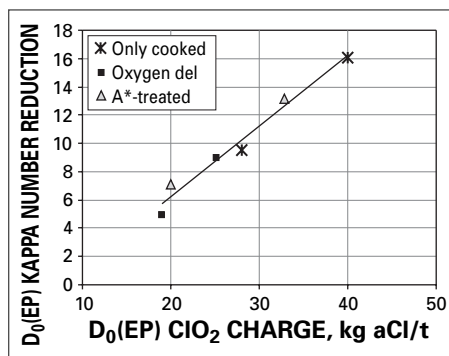
As expected, oxygen delignification reduced the lignin content without affecting the HexA acid content. The A* stage, on the other hand, reduced the HexA selectively, while the lignin content was reduced to a much lesser extent. The MeGlcA level was unaffected by the pretreatments and by the ECF bleaching sequence, but it was significantly reduced by prolonged cooking.

We estimated the HexA contribution to the kappa number by applying the contribution factor presented by Li and Gellerstedt [16]. Lignin and other structures contribute to the other part of the kappa number [17, 18]. Throughout the D(EP)D sequence for the six pulps, we monitored the total kappa number, the contribution of HexA, and the contribution of lignin and other structures. The results are shown in **Fig. 1**.

Prior to bleaching, the absolute amount of HexA was about the same in both oxygen-delignified pulps and pulps that were only cooked and not oxygen delignified. Because of their higher kappa number and lower brightness, the pulps that were only cooked required a higher chlorine dioxide charge than the oxygen-delignified pulps. Therefore, all the HexA

Kappa no.	Bright-ness, % ISO	HexA, mmol/kg	MeGlcA, mmol/kg	Uronic acid, mmol/kg		Klason lignin, %	ClO ₂ , kg aCl/ton	Final bright-ness, % ISO	Final kappa no.
				mmol/kg	mmol/kg				
B14	14.0	41.6	71	17	88	0.9	57	87.3	1.2
B14-O ₂	9.5	54.6	67	15	82	0.3	22	86.1	3.7
B14-A*	10.0	44.2	16	18	34	0.8	41	87.1	1.1
B-20	20.0	35.4	59	47	106	2.0	69	87.5	1.1
B20-O ₂	12.6	47.0	58	50	108	1.0	31	85.9	2.6
B20-A*	16.4	40.4	15	45	60	1.8	53	86.4	1.3

II. Pulp characteristics and bleaching results.



2. Kappa number reduction in the first two stages, or D(EP). The chlorine dioxide charge (kg/ton) was 2 x kappa no.

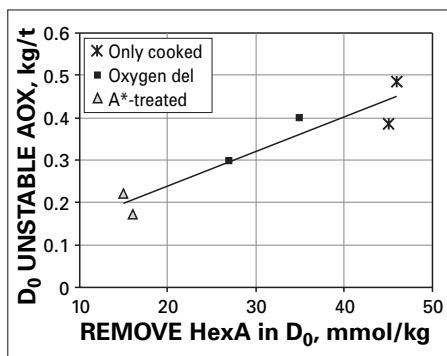
in the pulps that were only cooked was removed in the D(EP)D sequence. On the other hand, the chlorine dioxide charge required for the oxygen-delignified pulps was lower, and part of the HexA still remained after bleaching. The A*-treated pulps had a low HexA content prior to bleaching, and all the HexA was already removed in the first D stage.

It was not possible to find any relationship between beachability and the HexA and lignin contents given in Table II. The kappa number reduction in the first D(EP) stages seemed to depend mainly on the chlorine dioxide charge, regardless of the lignin and HexA levels, as Fig. 2 shows.

The D(EP)D-bleaching sequence was not optimized for the bleachability studies, but the results indicated that the brightness prior to bleaching was one of the most important variables for the bleaching chemical consumption to reach about 87% ISO brightness (Table II). Thus, the bleachability of these pulps depended primarily on factors other than the HexA and lignin levels.

AOX and OCI

We studied the stability of the AOX in the first D filtrate by storing the filtrates for 10 days under specified conditions. The AOX determined directly was denoted "total" AOX, while the AOX remaining after 10 days of storage was denoted as "stable" AOX and the part eliminated during the 10 days was denoted as "unstable" AOX. The AOX formed in the (EP) stage and second D stage was not tested for stability because unstable AOX is not present in the extraction filtrate [14], and it was assumed to be present in only very



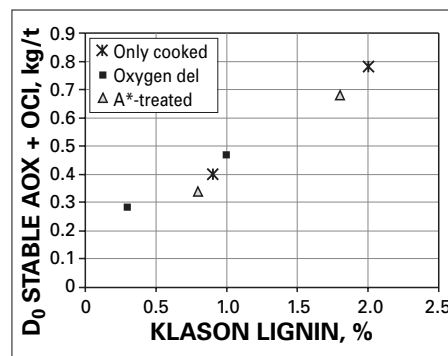
3. Unstable AOX vs. HexA removed in the first D stage.

small amounts in the second D stage.

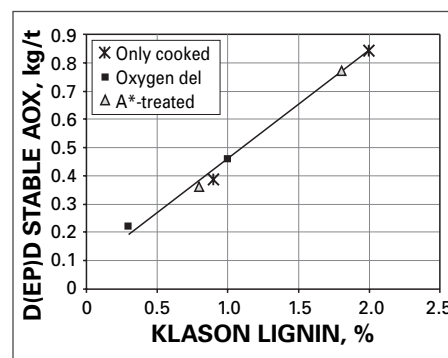
In the first D stage, 16–21 mol% of the chlorine dioxide charged was found as chlorine bound to organic substances, either as AOX or OCI (Table IIIA). At both kappa numbers, the oxygen-delignified pulp gave the highest value, and the A*-treated pulp gave the lowest value. This trend was also seen for the total AOX formation per chlorine dioxide charged in the first D stage, where the oxygen-delignified pulps had the highest values (10.4–11.8 mol%) followed by the pulps that were only cooked (9.7–9.8 mol%) and the A*-treated pulps (6.9–7.2 mol%). The same trends were seen in the case of softwood kraft pulps in our previous work [4].

The variations in the AOX and OCI levels cannot be explained only by the chlorine dioxide charges and the incoming kappa numbers, but a study of the stable and unstable parts of AOX reveals information about the factors behind the variations. The amount of unstable AOX in the first D filtrate was linearly dependent on the amount of HexA removed in the first D stage, as shown in Fig. 3. Thus, the unstable AOX clearly originated from HexA, as was shown in our similar study on spruce [4] and in other studies on eucalyptus and birch [7].

The stable AOX and OCI formed in the first D stage (Table III) was highly dependent on the amount of Klason lignin in the pulp. Figure 4 shows that there is a tendency for the oxygen-delignified pulps to give slightly more stable AOX and OCI at a given lignin content. This tendency probably arises because the oxygen-delignified pulps received a very high chlorine dioxide charge per unit of Klason lignin, and more of the



4. Klason lignin vs. the amount of stable AOX and OCI formed in the first D stage.



5. Klason lignin prior to bleaching vs. the amount of stable AOX formed in the D(EP)D sequence, when bleaching to 87% ISO brightness.

bleaching was done in the first D stage with the oxygen-delignified pulps than with the other pulps. The formation of stable AOX and OCI thus seems to be affected both by the Klason lignin content and by the chlorine dioxide charge.

There was also a relatively good correlation between the formation of OCI and AOX in the (EP) stage and the Klason lignin, in the same way as for the first D stage. The total amounts of stable AOX formed in the sequence when bleaching to approximately 87% ISO brightness are given in Table IIIB (i.e., values in the column for stable AOX in the rows under "After full D(EP)D bleaching"). These values are plotted in Fig. 5. If the comparison is made when most of the lignin has reacted, the oxygen-delignified pulps falls in line with the other pulps, and the total amount of stable AOX formed in the D(EP)D sequence shows a good linear relationship with the Klason lignin in the pulp prior to bleaching.

ENVIRONMENTAL CONTROL

	Total, AOX, mol%	Stable, AOX, mol%	Stable/total AOX, %	OCl, mol%	Total AOX + OCl, mol%
<i>After the first D stage</i>					
B14	9.8	2.9	29	7.1	16.9
B14-O ₂	10.4	2.6	25	7.4	17.8
B14-A*	7.2	2.9	40	8.5	15.7
B-20	9.7	3.6	37	9.8	19.5
B20-O ₂	11.8	3.9	33	9.3	21.1
B20-A*	6.9	3.5	51	10.4	17.3
<i>After the (EP) stage</i>					
B14	1.5	—	—	3.6	—
B14-O ₂	1.1	—	—	3.9	—
B14-A*	2.5	—	—	3.5	—
B-20	5	—	—	3.6	—
B20-O ₂	3.4	—	—	3.6	—
B20-A*	5.9	—	—	3.2	—
<i>After full D(EP)D bleaching</i>					
B14	6.8	3.4	—	—	—
B14-O ₂	11.7	5	—	—	—
B14-A*	6.5	4.4	—	—	—
B-20	9.6	6.1	—	—	—
B20-O ₂	13.7	7.3	—	—	—
B20-A*	9.4	7.3	—	—	—

IIIA. AOX and OCl concentrations in mol%.

	Total, AOX, kg/ton	Stable, AOX, kg/ton	Stable/total AOX, %	OCl, kg/ton	Total AOX + OCl, kg/ton
<i>After the first D stage</i>					
B14	0.55	0.16	29	0.4	0.95
B14-O ₂	0.4	0.1	25	0.28	0.68
B14-A*	0.29	0.12	40	0.34	0.63
B-20	0.77	0.29	37	0.78	1.55
B20-O ₂	0.59	0.2	33	0.47	1.06
B20-A*	0.45	0.23	51	0.68	1.13
<i>After the (EP) stage</i>					
B14	0.08	—	—	0.2	—
B14-O ₂	0.04	—	—	0.15	—
B14-A*	0.1	—	—	0.14	—
B-20	0.4	—	—	0.29	—
B20-O ₂	0.17	—	—	0.18	—
B20-A*	0.39	—	—	0.21	—
<i>After full D(EP)D bleaching</i>					
B14	0.78	0.39	—	—	—
B14-O ₂	0.52	0.22	—	—	—
B14-A*	0.53	0.36	—	—	—
B-20	1.33	0.84	—	—	—
B20-O ₂	0.86	0.46	—	—	—
B20-A*	0.99	0.77	—	—	—

IIIB. AOX and OCl concentrations in kg/ton.

	Only cooked ^a		A* treated ^b	
	Total	Stable	Total	Stable
<i>Kg/ton of pulp</i>				
Birch	1.2	0.7	0.8	0.6
Spruce	0.5	0.3	0.3	0.2
<i>Mol% of ClO₂ charged</i>				
Birch	14.6	8.6	12.8	9.4
Spruce	6.9	4.0	4.7	3.2

^aKappa no. 20. ^bKappa no. 16.

IV. Total and stable AOX from D(EP) or D(E) stages.

The results suggest that chlorinated lignin structures are the major contributors to the OCl in the pulp and to the stable AOX in the filtrate. Other components, such as extractives, have also been shown to be responsible for AOX in the filtrate and OCl in the pulp [8, 19]. In eucalyptus, 9–14% of the AOX and OCl was shown to originate from dichloromethane (DCM) extract [8]. In the study presented here, we determined the DCM extract but did not investigate its influence on the AOX and OCl formation.

The amount of DCM extract was 0.7% and 0.8% on pulp at kappa nos. 14 and 20, respectively. The oxygen delignification decreased the DCM extract by approximately 20% and the A* treatment by approximately 4%. Thus, the variations were not very large, and there was a partial covariation between the DCM extract and the Klason lignin. The DCM extract will also influence the formation of stable AOX and OCl, but the major trends and variations in this study were probably the result of variations in Klason lignin content.

The HexA removed from the pulp was apparently responsible for the unstable AOX formed in the first D stage, and the Klason lignin content was mainly responsible for the stable AOX and OCl formed, but what were the practical consequences of the different kappa numbers and different pretreatments?

The amounts of total AOX and stable AOX formed in the D(EP)D sequence after the different pretreatments and at the different kappa numbers are shown in the last six rows of Table IIIA and IIIB. The oxygen delignification and the A* treatment had about the same effect on

reducing the total AOX formation in the D(EP)D sequence (about 30%) compared with the pulps that were only cooked.

When we subtracted the unstable AOX and examined the levels of stable AOX, however, we discovered a large difference between the two pretreatments. Oxygen delignification reduced the stable AOX by about 45%, whereas the reduction attributable to A* treatment was only about 10%.

As a comparison of the B14 and the B20-O₂ and B20-A* pulps showed, prolonged cooking to a lower kappa number was at least as effective in reducing the AOX (stable and unstable) as lowering the kappa number by oxygen delignification or A* treatment. Of course, there are many other economic and quality factors that we did not consider in comparing these three scenarios.

The fact that different results are obtained for total AOX and stable AOX emphasizes the importance of specifying when and how the AOX levels are determined before they are compared with other data. Furthermore, the benefit of a hot acid treatment stage is only minor in terms of reducing stable AOX.

Comparison with softwood results

The general conclusions about the formation of organically bound chlorine are basically the same in this birch study as in the earlier study on spruce. In birch, the unstable AOX was shown to depend on the HexA removed in the first D stage; in spruce, it was shown to depend on the HexA in the pulp prior to bleaching (HexA after the first D stage was not measured). However, in the spruce study, the first D charge per HexA unit was in all cases relatively high, probably leading to an effective removal of HexA, so the HexA removed and the HexA measured prior to bleaching were probably correlated. Therefore, the best way to describe the amount of unstable AOX formed in the first D stage for these two wood species is to say that it is dependent on the amount of HexA removed in the first D stage.

The formation of stable AOX and OCI depended on the lignin content in the birch study. In the spruce study, this formation was not studied so thoroughly, but it seemed to correlate with the chlorine dioxide charge and lignin content. It is probable that a combination of these factors determines the formation, with additional variations possible as a result of extractives and carry-over in the unbleached pulp.

Despite some experimental variations between the birch and spruce studies, it is interesting to compare the results to get an overview of the differences. **Table IV** shows data for the total and stable AOX levels from the first two stages for both birch and spruce pulps with and without an A* treatment stage at two different kappa numbers. Some extrapolations have been made to make comparisons at the same kappa number. Although the formation of AOX followed the same pattern for birch and spruce, the birch pulps seemed to yield more total AOX and stable AOX that could not be directly linked to the HexA or lignin

levels. In addition, differences in pulp composition and in the degradation of HexA and lignin during kraft cooking gave further variations in the AOX levels at given kappa numbers and after different pretreatments.

CONCLUSIONS

The unstable part of the AOX in the first D stage is linearly dependent on the amount of HexA removed from the pulp. This result is strong evidence that the unstable AOX originates from HexA. The amounts of stable AOX and OCI in the pulp were related to the Klason lignin content of the pulp.

The AOX formation per charge of chlorine dioxide was highest for oxygen-delignified pulps, followed by pulps that were only cooked and then by pulps that were treated with hot acid. These differences were attributable to the pulps having different HexA contents.

For achieving a low level of stable AOX in the effluent, the use of an oxygen delignification stage or cooking to a low kappa number was more efficient than a hot acid stage. The same factors influenced the AOX formation in both birch and spruce. However, the birch pulps gave higher levels of total AOX and stable AOX for a given charge of chlorine dioxide, and this difference could not be explained only by the different levels of HexA and lignin. **TJ**

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ENVIRONMENTAL CONTROL

INSIGHTS FROM THE AUTHORS

The discharges of chlorinated organic substances from pulp mills have been reduced substantially during the last two decades. However, as mills continue to improve ECF bleaching and strive to lower the levels of chlorinated organics in the most economically feasible way, it is important to understand the underlying mechanisms in the formation of AOX and OCI.

This research is a continuation of our previous work on AOX formation in softwood presented in TAPPI J. 1(7): 20(2002). Our research complements extensive work on chlorinated organics in effluent and pulp. To the best of our knowledge, the direct influence of the HexA content in birch pulp on AOX and OCI formation has not been studied at different kappa numbers. We plan to continue this research by studying pulps of other types of hardwood.

The most difficult aspect of this research was combining the cellulose technology results and wood chemistry into a theory that would explain the difference observed in the AOX and OCI levels. As it turned out, HexA is of vital importance to the AOX formation during ECF prebleaching of birch kraft pulp. The AOX

originating from HexA was easy to reduce through neutralization.

Although a hot acid stage reduced the AOX analyzed in the subsequent chlorine dioxide stage, it was not effective in reducing the total stable AOX in the bleaching sequence. The stability of the AOX should be carefully considered when AOX analyses are carried out and when different means of AOX reduction are evaluated.

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