

Impact of high chlorine dioxide substitution on oxygen delignified pulp

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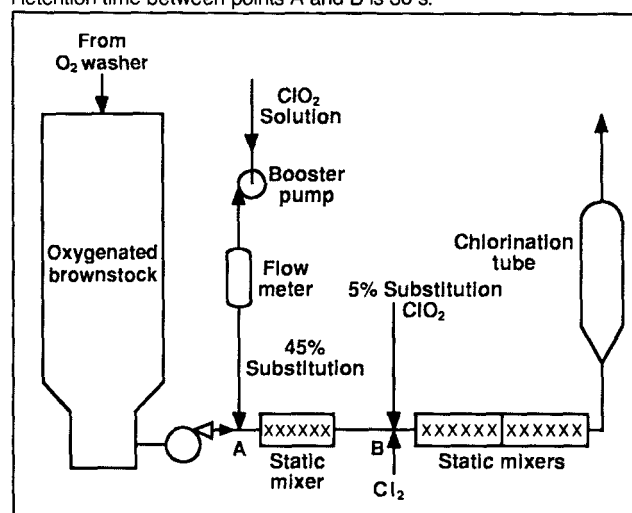
Mill trials at the E. B. Eddy Forest Products mill at Espanola led to the adoption of 50% chlorine dioxide substitution as the normal procedure on its softwood bleaching line.

Concerns about chlorinated dioxins and furans being generated as an unwanted by-product of pulp bleaching prompted us to evaluate our bleached pulps and effluents for these compounds. Results showed trace concentrations in the bleached pulps and untreated effluents. The measured levels were significantly lower than those found in conventionally bleached pulps; we attribute that to the reduced chlorine bleaching requirements of oxygen delignification. Due to a combination of several factors such as oxygen delignification, primary and secondary treatment, and pulping of both softwood and hardwood, Espanola's discharge of chlorinated organic compounds as absorbable organic halogens (AOX) was extremely low, around 1.4 kg/air-dried metric ton. The main objective, therefore, was to eliminate the trace levels of chlorinated dioxins and furans. Recent findings from the Pulp and Paper Research Institute of Canada (Paprican) and Sweden indicated that among several measures substantial replacement of chlorine with chlorine dioxide in the chlorination stage had the potential to decrease or eliminate measurable chlorinated dioxins and furans (1, 2). In addition, the formation of chlorinated dioxins and furans due to the presence of precursor compounds (such as unsubstituted dioxin and furan) can be minimized by improving brownstock washing and by using water based defoamers or oil based defoamers that are free from aromatics. The precursor level in our brownstock was low and acceptable (Table I). Suppliers were required to produce oil based defoamers from pure and aromatic-free oil. Brownstock washing was improved by running the second oxygen washer (after the oxygen stage) as an open system. Salt-cake carryover to the chlorination stage decreased from 20-30 lb/air-dried ton to 10-15 lb/air-dried ton. Having implemented all these measures, what remained was to increase the chlorine dioxide substitution. As the mill was fully committed to decreasing the chlorinated dioxins and furans below detection, the necessary equipment to allow high ClO_2 substitution was

I. Dibenzofurans in brownstock before chlorination

Date	Sample	Dibenzofuran concentration, ppt	Analytical lab
Nov. 14, 1988	Oxygenated brownstock	1450	Paprican
Nov. 15, 1988	Oxygenated brownstock	1540	
Nov. 16, 1988	Oxygenated brownstock	1970	
Nov. 17, 1988	Oxygenated brownstock	830	
Nov. 18, 1988	Oxygenated brownstock	1100	
	Water blank	≈1000	

1. Process schematic of the high chlorine dioxide substitution trial. Retention time between points A and B is 30 s.



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installed. Fortunately, the R-3 chlorine dioxide generator had significant surplus capacity for producing the additional ClO_2 . Mill evaluation of increased chlorine dioxide substitution began in late November 1988.

II. Effluent chlorinated dioxin and furan results (2,3,7,8 isomers)

	Substitution, %							
	10	28	38	44	47	48	52	57
C-filtrate TCDD, ppq	ND(25)	ND(28)	ND(46)	ND(1)	ND(26)	ND(25)	ND(19)	ND(18)
C-filtrate TCDF, ppq	ND(18)	ND(21)	ND(33)	ND(44)	43	120	ND(15)	ND(14)
E-filtrate TCDD, ppq	170	65	190	180	110	ND(26)	68	ND(20)
E-filtrate TCDF, ppq	490	320	1000	770	220	470	ND(12)	ND(15)
Main pumping station TCDD, ppq	ND(23)	ND(15)	Spoiled	...	...	ND(27)	...	...
Main pumping station TCDF, ppq	63	52	Spoiled	...	...	ND(23)	...	...
Q-zone TCDD, ppq	ND(34)	Not tested	Not tested	...	...	ND(60)	...	...
Q-zone TCDF, ppq	45	Not tested	Not tested	...	...	ND(45)	...	...

ND = not detectable. Q-zone = secondary treated effluent. Method detection limit is 25.

III. Chlorinated phenolics in effluents, ppb

	Phenols			Guaiacols			Catechols			Vanillins			Total chlorinated phenolics		
	C ₁	E ₁	MPS	C ₁	E ₁	MPS	C ₁	E ₁	MPS	C ₁	E ₁	MPS	C ₁	E ₁	MPS
10% ClO ₂ substitution November 16	81	209	40	134	2270	309	3067	585	201	15	484	31	3297	3548	581
30% ClO ₂ substitution November 28	140	304	37	554	2433	322	2405	437	114	40	942	86	3139	4116	559
40% ClO ₂ substitution December 2	83	270	30	119	2499	252	2722	530	106	16	879	111	2940	4178	499
50% ClO ₂ substitution	117	229	27	452	2085	278	1797	256	102	160	916	143	2526	3486	550

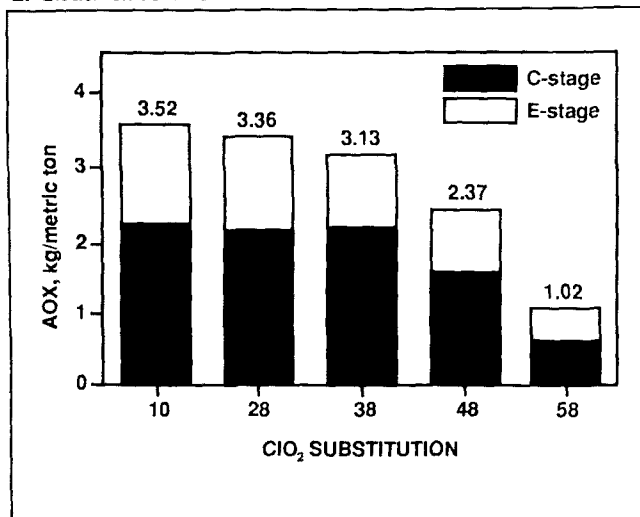
C₁ = softwood chlorination filtrate. E₁ = softwood extraction filtrate. MPS = main pumping station (total untreated effluent).

Mill description

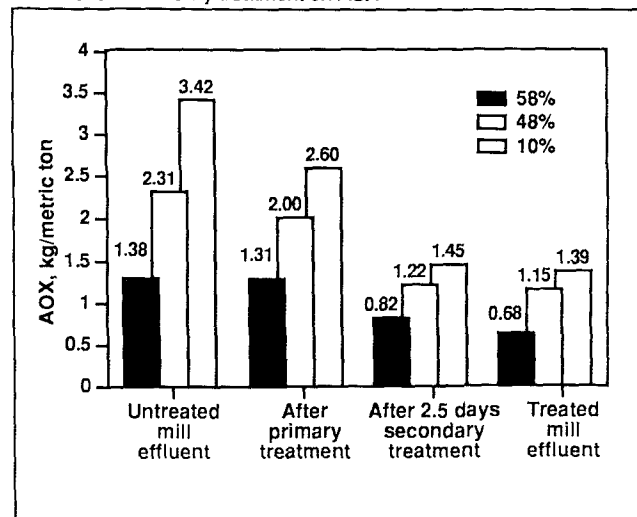
At Espanola, the 975-ton/day pulp mill consists of two separate pulping and bleaching lines, one producing softwood and the other hardwood. The softwood is predominantly jackpine, and 80% is received in chip form from sawmill residues. Hardwood chips are segregated into two piles: dense hardwood (birch and maple) and light hardwood (poplar). Approximately 80% of the hardwood is received as roundwood and then debarked and chipped in our woodroom. Softwood chips are thickness screened in Rader disc screens to remove overthick chips (>22 mm), pins (1.5–3 mm), and fines (<1.5 mm). Pins are reclaimed on a controlled basis, fines are burned in the Biomaster bark boiler, and overthick chips are land filled. Hardwood chips are not screened. The softwood pulping line consists of a 510-air-dried-ton/day Kamyr continuous digester, two-stage atmospheric diffuser, pressure knotters and pressure screens, and a screen-room decker. The screened pulp is further delignified 45% in a high-consistency oxygen stage followed by two stages of washing. The bleaching sequence is (C+D)(EO)HD to an 88+ brightness. Bleached pulp is dried on a Kamyr cylinder pulp machine or used as slush in two specialty packaging and fine paper machines.

The hardwood pulping line consists of five batch digesters, pressure knotting, and three-stage drum brownstock washing. The brownstock is delignified in a high-consistency oxygen stage followed by one stage of washing, and then screened in atmospheric Cowan screens and washed on the screen-room decker. The filtrate cycle is closed from the screen-room decker through the oxygen washer and the brownstock washers. The bleaching sequence is (CD)EHD to an 88+ brightness. Bleached pulp is dried on a fourdrinier Black-Clawson pulp machine. Approximately 65% of the total pulp production is marketed. Black liquor from both lines is combined, evaporated in a five-effect HPD evaporator with concentrator, and burned in a 4-million-lb/day CE low-odor recovery boiler. Recausticizing is conventional, using two unit-type clarifiers. Evaporator and digester condensates are steam stripped, the stripper gases being burned in the two lime kilns along with the gases from the odorous gas collection system. Soap skimmed from the black liquor is processed into tall oil, as well as being sold as-is. Chlorine dioxide is generated in a 15-ton/day Erco R-3 generator. The effluent treatment system handles the entire mill effluent and consists of a 12-h settling basin (primary treatment) and a 6-day aerated lagoon (secondary treatment).

2. Bleach effluent AOX content



3. Effect of secondary treatment on AOX



IV. Effluent toxicity* and BOD₅

Sample	ClO ₂ substitution, %			
	10	30	40	50
Final effluent BOD ₅ , tons/day	2.2, November 23-30	1.8, December 1-6	1.7, December 7-9	2.1, December 10-16
Total untreated mill effluent	16-35	26	...	18-35
Total treated mill effluent	100	100	...	100

*Results based on bioassay results on rainbow trout fingerlings, 96-h L.C.₅₀ lethal concentration 50 expressed as percent.

V. Chlorate concentration in mill effluent

Sample	ClO ₂ substitution, % ^a			
	10	30	40	50
Chlorination effluent (softwood line)	82	94 ^b	112 ^c	129 ^d
Chlorination effluent (hardwood line)	108	...	...	102
Total mill untreated effluent (lagoon splitter box)	33	37	31	37
Total mill treated effluent (Q-zone)	ND	ND	ND	ND

^aResults in mg/L. ^b3 samples. ^c3 samples. ^d11 samples.
ND = not detectable.

Normal chlorine dioxide substitution

Our normal operation is 10% ClO₂ substitution added simultaneously with chlorine. Chlorine, chlorine dioxide, and stock are mixed in static mixers. The normal residence time in the C-stage is around 5 min at a temperature of 55°C. Chlorination is controlled with a Measurex Versabrite sensor located after the tower. Manual residual tests done once an hour refine the control further. Chlorine dioxide is ratioed to chlorine to maintain a constant 10% substitution.

High chlorine dioxide substitution

The chlorination system was modified to allow high chlorine dioxide substitution, added sequentially as shown in Fig. 1. The bulk of the chlorine dioxide is added about 30 s before chlorine injection to achieve economical delignification. The remaining chlorine dioxide at 10% substitution on molecular chlorine is added simultaneously as before to ensure pulp viscosity protection. Approximately \$150,000 was spent to install additional chlorine dioxide piping, a magnetic flow meter for chlorine dioxide, a booster pump for chlorine dioxide, and an additional static mixer (Fig. 1).

Evaluation program for the trial

The high substitution trial was run in two parts. In part 1, the substitution was increased from 10% through 28% and 38% to 48%. The objective was to operate at 50% for 6 months, then run part 2, increasing substitution from 50% to 55%, 58%, 61%, and 66%. However, an error in the chlorine flowmeter, which was discovered after the part 2 trial, resulted in substitutions varying between 40% and 50% for the 6 month period, and part 2 being run at 44%, 47%, 52%, and 57% substitution.

In part 1, each level of substitution was evaluated for the following parameters:

- Brownstock—precursors such as dibenzofurans (DBF)
- Bleached pulp—viscosity, strength, brightness, cleanliness, chlorinated dioxin and furan, AOX, and extractives concentrations
- General—bleaching conditions, chemical consumption, and bleaching cost
- Effluents—chlorinated dioxin and furan, AOX, chlorinated phenolics, toxicity, and chlorate.

For part 2, the evaluation procedure was similar but not as extensive.

VI. Chlorinated dioxin and furan in bleached softwood pulp

Date	Operating conditions	Results, ppt		Analytical lab
		Dioxin 2,3,7,8 TCDD	Furan 2,3,7,8 TCDF	
October 1987	10% ClO ₂ substitution High Cl ₂ residual, 0.5% Normal brownstock washing	7.3	41	Enseco, Calif.
July 1988	10% ClO ₂ substitution High Cl ₂ residual, 0.5% Improved brownstock washing	4.1	11	Enseco, Calif.
Nov. 14-18, 1988	10% ClO ₂ substitution Lower Cl ₂ residual, 0.35% Improved brownstock washing	2.2-3.6 (5 samples)	7.1-11	Seakem, B.C.
Nov. 28, 1988	28% ClO ₂ substitution Cl ₂ residual, 0.1%	1.8	6.4	Seakem, B.C.
Dec. 15-21, 1988	48% ClO ₂ substitution, Cl ₂ residual, 0.03%	ND (1.2-2.3) (5 samples)	ND-6.2(1-1.9)	Seakem, B.C.
Jan. 19, 1989	44% ClO ₂ substitution Cl ₂ residual 0.03%	ND(1.5)	ND(1.3)	Seakem, B.C.
March 20, 1989	42% ClO ₂ substitution Cl ₂ residual 0.03%	ND(1.1)	4.2	Seakem, B.C.
April 27, 1989	44% ClO ₂ substitution	1.6	5.6	Seakem, B.C.
May 8, 1989	47% ClO ₂ substitution	1.6	5	Seakem, B.C.
May 19, 1989	52% ClO ₂ substitution	ND(1)	ND(0.9)	Seakem, B.C.
May 30, 1989	57% ClO ₂ substitution	ND(0.8)	ND(0.7)	Seakem, B.C.

Analysis

We conducted evaluations for bleached-pulp viscosity, strength, brightness, cleanliness, effluent toxicity, and chlorate. An outside laboratory in Seakem, B.C. analyzed for bleached-pulp chlorinated dioxin and furan, and for effluent chlorinated dioxin and furan. The University of Toronto conducted tests for bleached-pulp AOX and extractives. Paprican studied effluent AOX, brownstock precursors such as DBF, and chlorinated phenolics.

Sampling frequency

Dioxins and furans. Two samples for brownstock precursors such as DBF were taken before high chlorine dioxide substitution began, and two more were taken after 48% substitution was established. No precursor testing was done during the part 2 substitution trials. In testing for tetrachlorodibenzo-p-dioxin (TCDD) and tetrachlorodibenzofuran (TCDF) in bleached pulp, five consecutive daily samples were taken before high chlorine dioxide substitution began. Five consecutive daily samples were also taken after 48% substitution was established; one sample was taken at all other substitution levels. In testing for TCDD and TCDF in effluents, including the chlorination stage, extraction stage, main pump station (untreated mill effluent), and Q-zone (treated mill effluent), one sample was taken from each location before high substitution began and one sample at each level of substitution.

Chlorinated organic compounds as AOX. To test bleached pulp, we took five samples before high substitution began, one sample at each level of substitution up to 48%, and five samples at 50% substitution. No samples were taken during the part 2 evaluation. We took one sample of chlorination effluent, extraction effluent, total untreated effluent, and primary and secondary effluent

from five locations before high substitution began. One sample was taken from the same locations at each level of substitution up to 48%. Above 48%, only the 58% level was sampled as part of an on-going routine.

Effluent toxicity. Acute toxicity as 96-h lethal concentration 50 (L.C. 50) is tested once a week and is an on-going mill routine.

Effluent chlorinated phenolics. To establish background level, 10 samples from various parts of the mill effluent system were sampled once at the following locations:

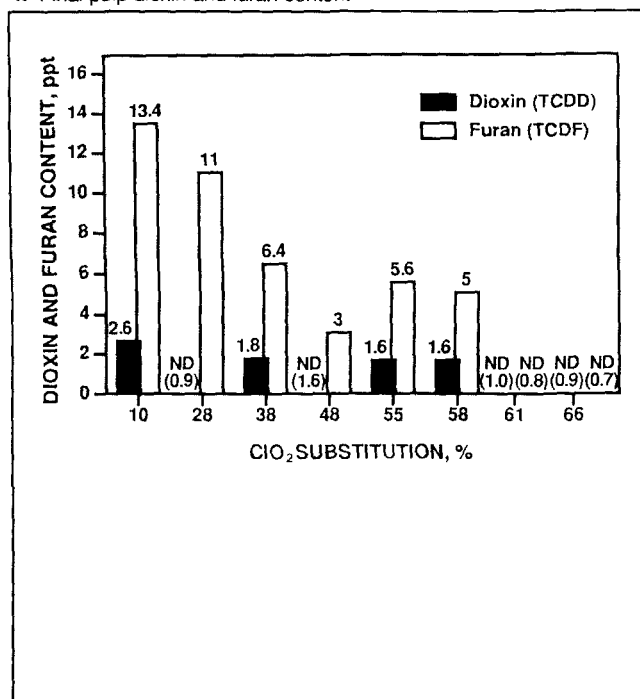
- No. 1 bleach plant C-stage filtrate
- No. 1 bleach plant EO-stage filtrate
- No. 2 bleach plant C-stage filtrate
- No. 2 bleach plant E-stage filtrate
- Main pump station (total untreated mill effluent)
- Settling basin inlet (main pump station plus woodroom effluent)
- Settling basin outlet (after primary treatment)
- Cell 1 outlet (after 2-5 days of secondary treatment)
- Cell 2 outlet (after 6 days of secondary treatment)
- Quiescent zone outlet (final treated effluent to the river).

These same samples were also collected at 48% substitution. Three samples of untreated effluents at 28% substitution and 38% substitution were taken:

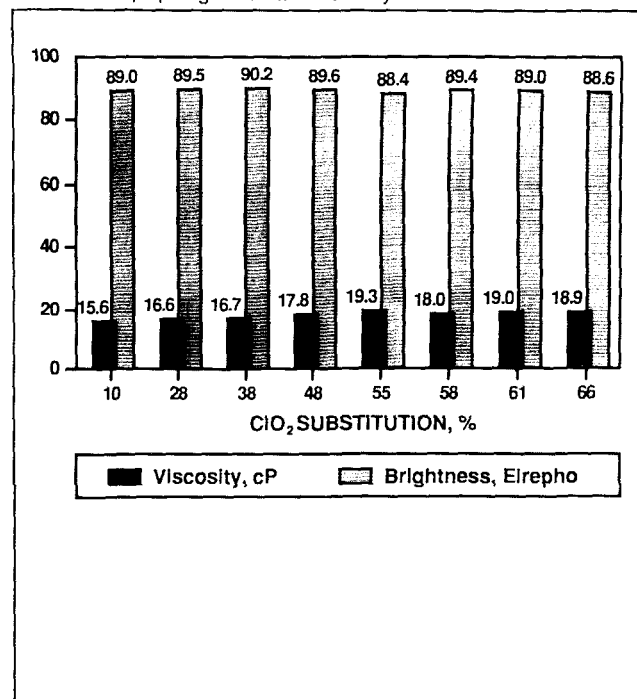
- No. 1 C-stage filtrate
- No. 1 EO-stage filtrate
- Main pump station.

No chlorinated phenolics testing was done during the part 2 trials.

4. Final pulp dioxin and furan content



5. Bleached pulp brightness and viscosity.



Results

Effluent quality

Chlorinated dioxins and furans. Results of these tests are reported in **Table II**. There was no detectable 2,3,7,8 TCDD in untreated or treated mill effluent or in C-stage filtrate at any level of substitution. The 2,3,7,8 TCDD was nondetectable in E-stage filtrate at 48% substitution (0.14 chlorine multiple) and at 57% substitution (0.11 chlorine multiple). There was no detectable 2,3,7,8 TCDF in treated or untreated mill effluent at 48% substitution. A substitution of 52% (0.14 chlorine multiple) was required to reduce the 2,3,7,8 TCDF in C-stage and E-stage filtrate below detectable levels.

Chlorinated organics as AOX. AOX from softwood C- and E-stages decreased approximately 33%, from 3.52 kg/metric ton to 2.37 kg/metric ton, as a result of increasing substitution from 10% to 50% (**Fig. 2**). Treated effluent decreased approximately 17%, from 1.39 kg/metric ton to 1.15 kg/metric ton AOX by increasing substitution from 10% to 50% (**Fig. 3**). August AOX results at 58% substitution show a further decrease to 1.02 kg/metric ton in the C- and E-stage effluents, 1.38 kg/metric ton in the untreated effluent, and 0.68 kg/metric ton in the treated effluent. Effluent treatment removes 50–60% of the AOX, with the majority being removed in the first 2.5 days of aerated treatment.

Chlorinated phenolics. Results of the tests for chlorinated phenolics are listed in **Table III**. Increasing substitution decreased the total chlorinated phenolics formed, with the decrease occurring in the C-stage effluent. It also changed the proportions of phenolics formed, increasing the vanillins and guaiacols and decreasing catechols. This proved beneficial since our lagoon effectively treats vanillins and guaiacols (82% and 76% removal, respectively) but is much less effective on phenols and catechols (44%

and 12% removal). Increased substitution also decreased the amount of highly chlorine substituted phenolics formed, producing more di- and tri-chlorinated compounds and less tetra-chlorinated compounds.

Acute toxicity. Results of the tests for acute toxicity are listed in **Table IV**. Fish bioassay tests showed no significant change in the untreated effluent toxicity. The 96-h L.C. 50 values remained in the normal range of 16% to 35%. Treated effluent consistently remained nontoxic, both before and after high substitution.

Chlorate. Results of the tests for chlorate are listed in **Table V**. Effluent chlorate measurements using Dionex showed no significant increase with high substitution in the total mill untreated effluent, although the C-stage effluent showed an increase of about 58%.

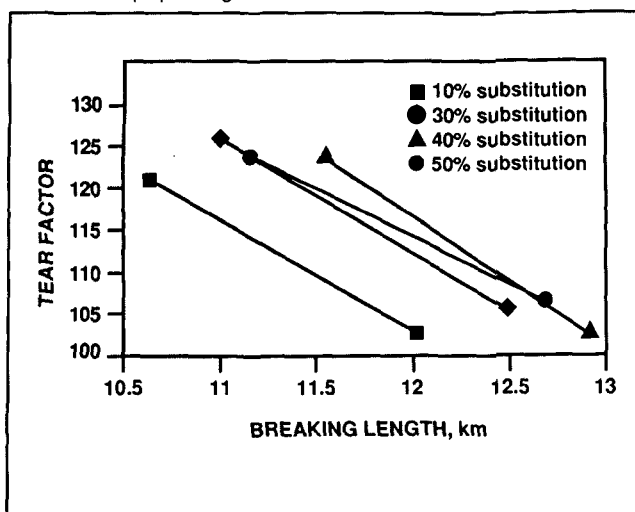
Pulp quality

A substitution of 52% (0.14 chlorine multiple) was required to reduce both 2,3,7,8 TCDD and 2,3,7,8 TCDF below detection level in the bleached softwood pulp (**Table VI**, **Fig. 4**). Pulp viscosity increased from 15.6 cp to 17.8 cp (**Fig. 5**) when we increased substitution from 10% to 48%. Pulp strength as measured by a tear-tensile relationship showed a significant increase (**Fig. 6**). Final pulp brightness was unchanged at 88+ Elrepho (**Fig. 5**). Pulp remained clean with very little dirt and very few shives. Bleached pulp AOX content showed a slight decrease as a result of increasing substitution from 10% to 48% (**Fig. 7**, **Table VII**).

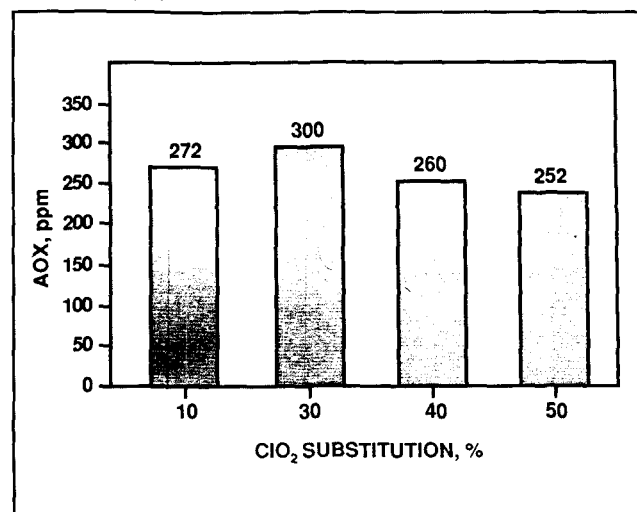
Bleaching cost

Total bleaching cost was minimized at 30% substitution, being about \$0.63/air-dried ton below the base case (10% substitution) at a higher final brightness. Cost and total

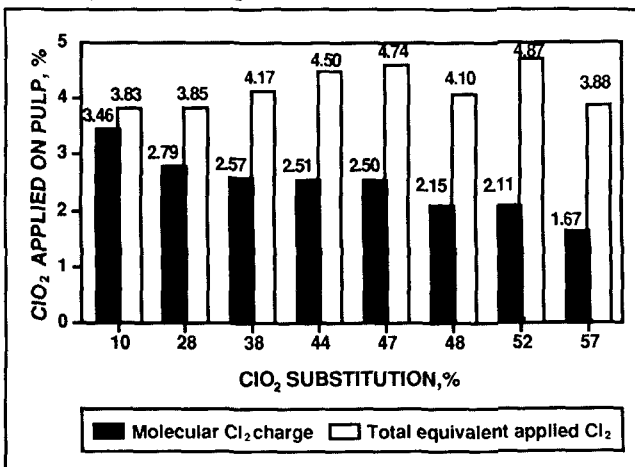
6. Softwood pulp strength



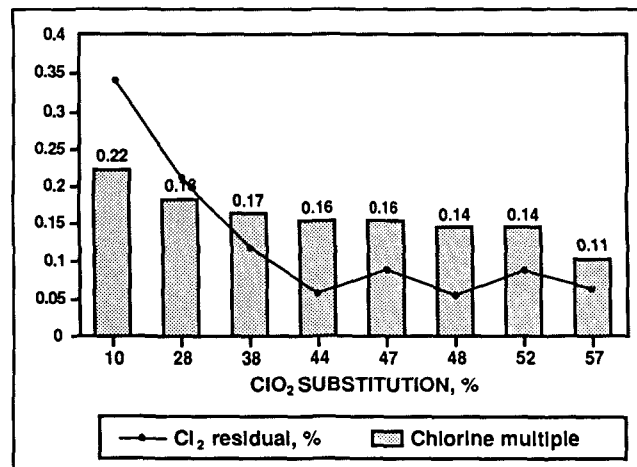
7. Bleached pulp AOX content



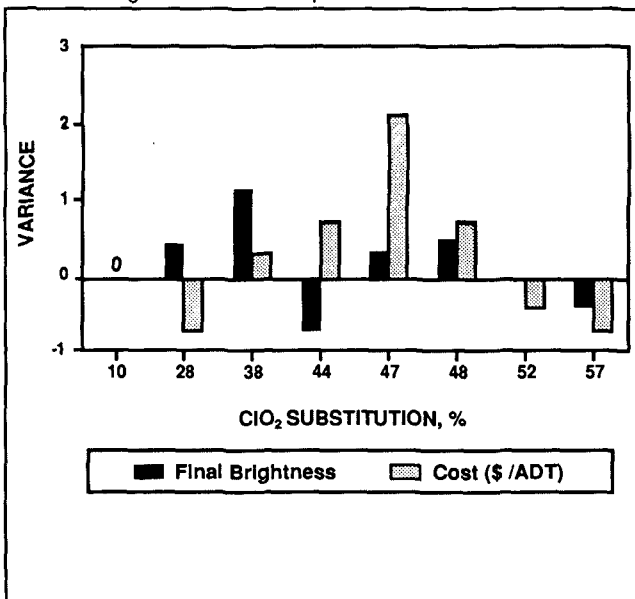
8. C-stage chlorine charge



9. C-stage residual and chlorine multiple



10. Bleaching cost baseline comparison



equivalent chlorine applied peaked between 44% and 48% substitution (Fig. 8 and 9). Cost decreased below the base case over 50% substitution.

Other observations

Chlorine multiple. Increasing substitution from 10% to 57% has reduced the chlorine multiple from 0.22 to 0.11. A reduction in chlorinated dioxin and furans is expected as a result of decreasing the chlorine multiple below 0.15, as evidenced from other work (3). Chlorine multiple here is defined as percent total molecular chlorine divided by the kappa number of unbleached pulp (Fig. 10).

Kappa factor. The kappa factor passed through a maximum at 47% substitution, increasing from 0.29 to 0.31. It decreased to 0.25 at 57% substitution (Table VIII). Kappa factor is defined as percent total equivalent chlorine divided by kappa number.

Chlorination residual. Increasing substitution from 10% to 48% allowed the chlorination residual to be decreased from 0.3% to 0.06% (Fig. 9). This did not have an adverse effect on shive bleaching in the C-stage.

Excess hypochlorite generation at the R-3 plant. Due

VII. AOX in bleached pulp

Date	ClO ₂ %	Total	Chlorine, ppm				
			AOX, water extractable	AOX, nonwater extractable	EtoH/Tol. extractable	Non-EtoH/Tol. extractable	Extractives EtoH/ Toluene (1:1), %
Nov. 14/88	10	220	12	270	70	200	0.04
Nov. 15/88	10	300	13	280	30	250	0.03
Nov. 16/88	10	250	10	290	50	240	0.03
Nov. 17/88	10	340	8	240	40	200	0.02
Nov. 18/88	10	250	11	320	90	230	0.04
Average	10	272	11	280	56	224	0.03
Nov. 18	30	300	7	230	40	190	0.03
Dec. 2	40	260	9	240	40	200	0.04
Dec. 15	50	290	10	220	20	200	0.01
Dec. 16	50	220	10	220	40	180	0.00
Dec. 19	50	260	9	220	20	200	0.00
Dec. 20	50	230	9	230	40	190	0.04
Dec. 21	50	260	10	260	60	200	0.03
Average	50	252	10	230	36	194	0.02

VIII. Bleach plant operating conditions

	Substitution, %							
	10	28	38	44	47	48	52	57
Production, air-dried tons/day	488.6	506.2	498.6	510.2	509.6	527.5	515.4	503
O ₂ kappa number	11.4	11.1	11.1	11.4	11.3	11.2	11.2	11.3
Total equivalent Cl ₂ applied, %	3.83	3.85	4.17	3.6	3.87	4.10	3.68	3.37
Molecular Cl ₂ applied, %	3.46	2.79	2.57	1.61	1.63	2.15	1.42	1.16
ClO ₂ substitution with Cl ₂ , %	9.7	10.8	10.6	17.4	17.3	10.1	19.9	23.8
ClO ₂ substitution serial addition, %	0.0	19.0	31.5	46.9	50.4	42.1	53.3	56.6
Total ClO ₂ substitution, %	9.7	27.5	38.4	55.2	58	47.5	61.4	65.5
Molecular chlorine multiple	0.22	0.18	0.17	0.1	0.11	0.14	0.09	0.08
Kappa factor	0.24	0.25	0.27	0.23	0.25	0.27	0.24	0.22
Cl ₂ residual, %	0.34	0.21	0.12	0.05	0.08	0.03	0.08	0.06
E-stage NaOH applied, %	2.62	2.76	2.56	2.12	2.14	2.47	2.12	1.93
E-stage headbox pH	10.6	10.8	10.7	10.5	10.6	10.6	10.6	10.6
E-stage brightness, Elrepho	46.0	48.2	52.8	50.5	51.8	50.6	50.3	50.3
H-stage hypochlorite applied, % Cl ₂	0.47	0.47	0.33	0.35	0.38	0.37	0.4	0.42
H-stage brightness, Elrepho	62.7	64.5	65.8	63.2	65.5	63.2	65.2	64.6
D-stage ClO ₂ applied, %	1.63	1.47	1.56	1.58	1.61	1.58	1.59	1.56
D-stage brightness, Elrepho	89.0	89.5	89.6	88.8	89.3	89.3	89.2	88.9
Final brightness, No. 3 Kamy, Elrepho	89.0	89.5	90.2	88.4	89.4	89.6	89	88.6
Brownstock viscosity, cP	27.14	28.12	30.09	28.58	30.07	31.31	29.99	33.53
Hypochlorite viscosity, cP	17.83	18.76	20.16	20.85	20.96	20.32	21.79	21.04
Final viscosity, cP	15.56	16.61	16.73	19.25	18.03	17.76	18.96	18.89
Shive count, no. per lap	0.3	0.3	0.1	1.07	0.72	0.28	0.32	0.31

to the higher generator operating rate at high chlorine dioxide substitution, excess hypochlorite, which cannot always be used for bleaching, is being produced as a by-product.

Summary

In November 1988, the first part of a two-part chlorine dioxide substitution trial was run, increasing the substitution of chlorine dioxide for chlorine in the softwood chlorination stage from 10% to 28%, 38%, and 48%. Extensive monitoring was carried out at 10% and 48%, with limited monitoring at 28% and 38%. The second part of the trial was run in May 1989, increasing substitution from 40% to 44%, 47%, 52%, and 57%. Results indicate that the expected benefits of high substitution have been realized. At 52% substitution the chlorinated dioxins and furans in bleached pulp and in process and mill effluents are below detection levels. Other benefits were decreased effluent AOX and chlorinated phenolics content and increased pulp viscosity and strength. Bleached pulp brightness, cleanliness, AOX, and effluent toxicity remained unaffected.

Treated effluent AOX decreased to a very low level, approximately 0.7 kg/metric ton. Bleaching cost decreased approximately \$0.40/air-dried ton over 10% substitution operation. Fifty-two percent chlorine dioxide substitution is now the normal mode of operation in our softwood chlorination stage.□

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