PEER REVIEWED

Effect of tertiary coagulation and flocculation treatment on effluent quality from a bleached kraft mill

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ABSTRACT

Since January 1995, the tertiary color-removal process at Crestbrook Forest Industries mill (Skookumchuck, BC) has removed an average of 60±7% of effluent color. The tertiary treatment system is a dual-polymer process that uses coagulation and flocculation technologies, which are relatively nonspecific. Consequently, besides removing the chromophores of concern, tertiary treatment of kraft effluent can also significantly reduce the levels of other major parameters such as chemical oxygen demand, chlorinated organic compounds, and target contaminants such as TCDF. A study of the system's performance showed that it functioned effectively over a broad range of temperature, pH, polymer dose, and influent BOD. The tertiary treatment system reduced COD, AOX, 2,3,7,8-TCDF, and mixed-function oxygenase induction by 24%, 23%, 80%, and 18%, respectively. Treatment of segregated D- and E-stage effluents did not reduce polymer consumption, but this approach shows promise as a means of reducing equipment size and thus capital cost.

Application:

Shows that tertiary treatment to remove color from bleach-plant effluent has a beneficial effect on a broad range of parameters that are used to gauge effluent quality.

FEDERAL LEGISLATION GOVERNING Canadian pulp and paper manufacturers regulates the discharge of biochemical oxygen demand (BOD), total suspended solids (TSS), acute toxicity, and dioxin/furans. Provincial governments in four Canadian provinces have also applied limits to the discharge of adsorbable organic halides (AOX). In addition to these conventional pollutants, other parameters can be regulated on a site-by-site basis, depending on the particular characteristics of the receiving waters. In response to these regulations and to forces in the marketplace, the industry has made dramatic strides in reducing the discharge of these conventional pollutants.

Effluent discharge regulations have continued to evolve in the major pulp-producing countries of the world. In 1992, the Canadian government enacted legislation requiring mills to carry out environmental effects monitoring (EEM) programs to determine the effectiveness of discharge permits in protecting ecosystems in the receiving environments *(1, 2)*. These programs represented a shift from traditional regulatory strategies that relied on effluent quality parameters. In an effort to establish a feedback loop between the receiving environment's response and regulation development, mills were required to measure a host of sublethal toxic and population effects using a number of specified biological indicators. Over the same time period, the U.S. Environmental Protection Agency (EPA) examined the possibility of regulating several effluent quality parameters not previously controlled. Included in the EPA list of candidate parameters under consideration were effluent chemical oxygen demand (COD), AOX, and color *(3)*.

In the past, effluent color has been regulated only on a site-specific basis, primarily because of the belief that high-molecular-weight chromophores did not exert an appreciable environmental impact. Despite this, some mills have installed tertiary color-removal systems (TCRS) in response to particular sensitivities or characteristics of the local receiving environment. One such operation is the Crestbrook Forest Industries (CFI) pulp mill in Skookumchuck, BC. Prior to implementation of color-removal technologies at CFI, limited dilution combined with the aqua-blue color of the receiving water resulted in a noticeable color increase downstream from the mill *(4)*.

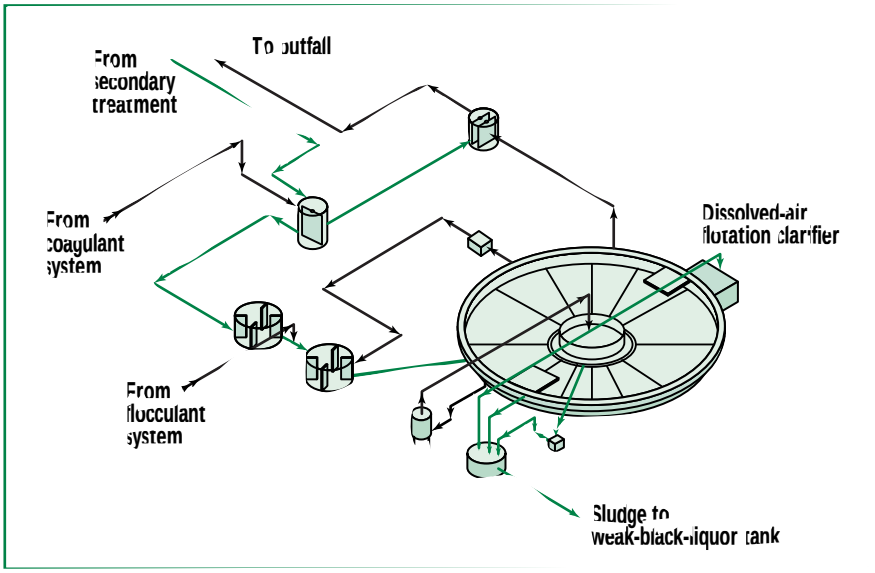
The primary goal of tertiary coagulation and precipitation systems installed at operations such as CFI is to reduce effluent color. However, in light of new legislative initiatives such as the proposed U.S. Cluster Rules and Canadian environmental effects monitoring (EEM) program, these technologies may bear reexamination as a means of removing target compounds and producing a higher-quality effluent.

The purpose of this study was to examine the impact of the tertiary coagulation and flocculation system at CFI on other effluent quality parameters. Included in the overall objective are three specific goals:

1. Determine the impact of common operating parameters (pH, temperature, BOD, and coagulant dose) on color removal
2. Determine the impact of tertiary treatment on removal of COD, AOX, chronic toxicity, tetrachlorodibenzofuran (TCDF), and mixed-function oxygenase (MFO) induction
3. Determine the potential for treatment of segregated D- and E-stage filtrates.

	After primary treatment	After secondary treatment	After tertiary treatment
BOD_5, mg/L	198	21.2	15.7
COD, mg/L	874	480	364
TSS, mg/L	41.9	21.5	40.2
AOX, mg/L	8.3	4.8	3.7
Color, c.u.	635	655	257

I. Wastewater characteristics across full-scale treatment system



1. Tertiary color-removal system

MATERIALS AND METHODS

The mill

The study was conducted at CFI's single-line kraft pulp mill in Skookumchuck, BC. The mill started up in 1968 and has since converted a Kamyr continuous hydraulic digester to vapor-phase operation, added oxygen delignification, and implemented 100% ClO_2 substitution to produce 650 metric tons/day of fully bleached market pulp.

The mill discharges into the pristine, aqua-blue Kootenay River. Concerns over color and odor downstream of the mill discharge emerged shortly after the mill startup in 1968. A rapid soil-infiltration system was used to remove color between 1981 and 1994. However, declining performance led to a search for alternative technology for color removal. As part of a CAD 300 (US$ 216) million rebuild in 1993–94, a CAD 10 (US$ 7) million (capital cost in 1993 dollars) dissolved-air flotation (DAF), second-generation, tertiary effluent-treatment system was installed for color removal *(4)*. The system was designed by Sandwell Inc. based on patents owned by Stone Container Inc.

Tertiary color-removal system

The CFI mill sends approximately 38,000 m^3 of effluent per day to the wastewater treatment system. Mill effluent is subjected to primary (settling basin) and secondary [5-day hydraulic retention time (HRT) in an aerated stabilization basin (ASB)] treatment before entering the TCRS. The average characteristics of the combined mill effluent at various points across the treatment system are given in **Table I**.

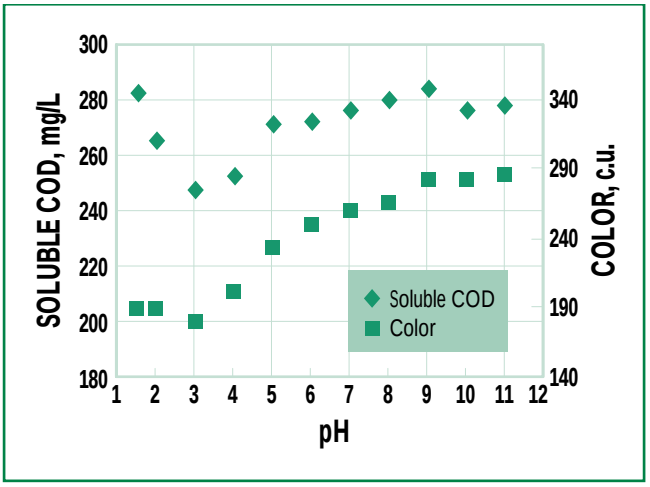
Figure 1 is a schematic of the tertiary color-removal system. Upon entering the tertiary treatment plant, effluent is pumped into two 150-m^3 (5-min HRT) mixing tanks that are connected in series. In the first tank, coagulant (quaternary polyamine) is added to form small flocs. As the effluent flows into the second tank, flocculant (polyacrylamide) is added to form larger flocs from the coagulated chromophores. The effluent then flows into a specially designed, low-shear DAF system (110-min HRT) for removal of color flocs. The color flocs are skimmed from the surface of the DAF, combined with weak black liquor, and pumped back to the mill for incineration in the recovery boiler. Effluent flows from the bottom of the clarifier through an external launder and into a pipeline, which transports it to a submerged diffuser outfall.

The color-removal system was designed to treat 45,000 m^3/day of effluent containing an average of 0.88 kg color/m^3. The system produces approximately 110 m^3 wet sludge/day. Over the first 18 months of operation, color removal averaged 55–80%, depending on effluent characteristics and polymer type and dose.

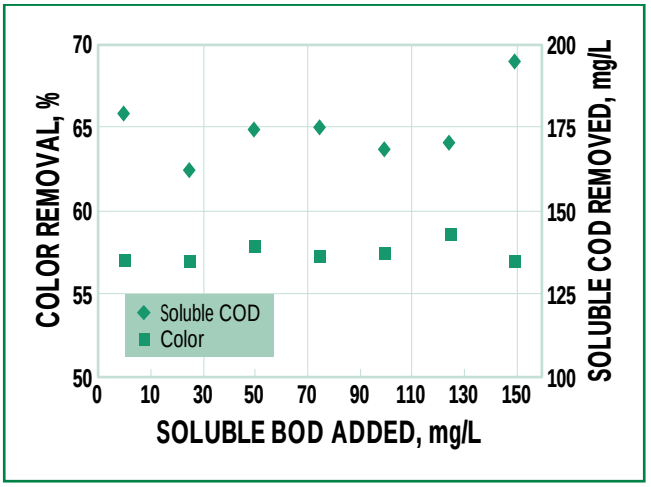
Effect of operating parameters on performance of dual-polymer system (jar tests)

Jar tests were carried out to determine the effect of a range of operating parameters on the performance of the dual-polymer system currently in use at the mill. Temperatures during the jar tests were maintained within ±0.5°C by immersing the test beakers in a temperature-controlled water bath. Jar tests were conducted using 500 mL of effluent in a 1-L beaker, stirring at a rate of 200 rpm in a jar stirrer (Phillips and Bird Model 7790-400, Richmond, VA). Coagulant makedown solution (1 mL = 20 ppm) was added using a syringe. After 3 min, the stirring speed was reduced to 100 rpm, and the appropriate dose of flocculant makedown solution (1 mL = 2 ppm) was added using a syringe. After an additional 3 min, the stirring speed was reduced to 50 rpm. After 6 min of stirring at 50 rpm, the sample was removed from the jar stirrer and allowed to settle for 10 min, at which time supernatant was decanted into a sample jar and assayed.

The influence of coagulant concentration (0–120 ppm), pH (1.5–11),



2. Effect of pH on color removal and COD in jar tests. Initial values of color and COD were 623 color units (c.u.) and 435 mg/L, respectively



3. Effect of soluble BOD on color removal and COD (jar tests)

Effluent stream	Initial color, c.u.	Initial COD, mg/L	Flow, m^3/h
D_1	870	1660	386
E_1	1636	3205	295

II. Characteristics of D_1- and E_1-stage filtrates, as sampled for the segregated-filtrate jar tests

temperature (7–40°C), and BOD_5 (5-day BOD) concentration (10–120 mg/L) on performance of the TCRS system was examined. Unless otherwise specified, tests were conducted on combined mill effluent after secondary treatment, sampled at the inlet to the TCRS.

The impact of BOD on performance of the dual-polymer system was tested in two ways. First, to determine the impact of soluble, readily biodegradable BOD on performance, a series of tests was carried out using secondary-treated effluent spiked with a BOD stock solution. The BOD stock solution consisted of acetic acid (0.5% v/v), formic acid (0.5% v/v, 88% HCOOH), and methanol (0.31% v/v). The COD and BOD_5 of the stock solution were 10,500 mg/L and 7500 mg/L, respectively. In a second series of tests, primary clarified effluent was seeded with a mixed microbial inoculum from the ASB and aerated in a batch reactor. At three different times over five days of reaction, samples were withdrawn, assayed for BOD_5, and treated using the dual-polymer system for color removal.

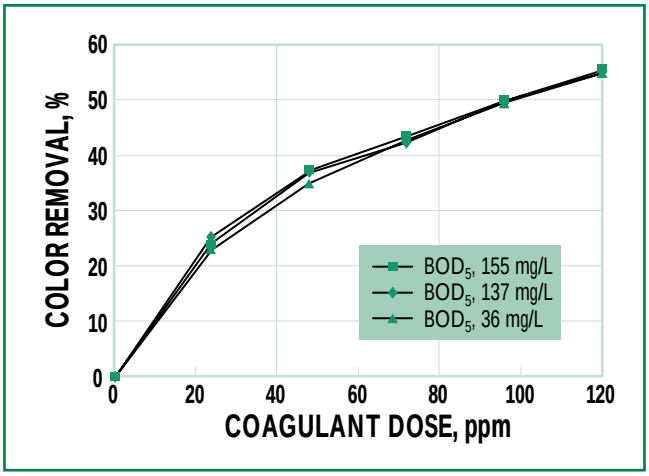
Treatment of segregated high-strength filtrates

In addition to tests carried out on secondary-treated combined mill effluent, color-removal tests were also carried out on the major color-bearing filtrates. For these tests, filtrates from the first bleaching (D_1) and extraction (E_1) stages of a five-stage bleach sequence—OD(EOP)D(EP)D—were used alone and in combination. (Note that the E-stage filtrate tested was combined EOP and EP. The overflow from the EP seal tank flows to the EOP seal tank, although the EP flow is typically low compared with the overflow from the EOP seal tank.) For combined tests, D_1- and E_1-stage filtrates were mixed in proportion to their relative flow rates. Filtrate pH was adjusted to pH 5 using either 50% (v/v) NaOH or 6*M* HCl. The initial characteristics of these streams are given in **Table II**.

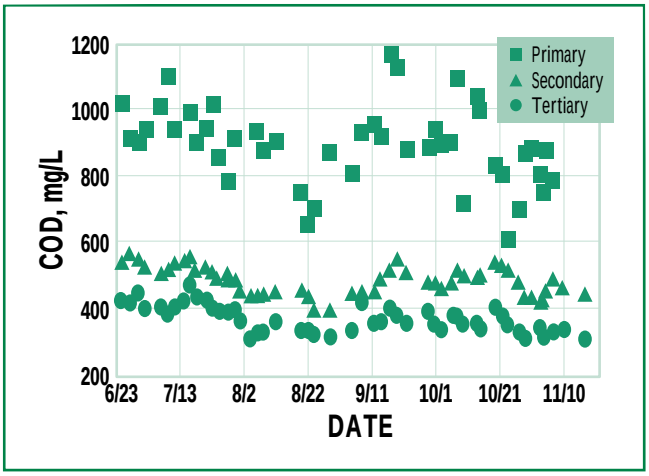
Analytical methods

Makedown solutions of coagulant (1% v/v) and flocculant (0.1% v/v) were prepared for use in the jar tests.

BOD was assayed according to standard methods *(5)*. TSS was analyzed according to CPPA H.1 using a set sample volume of 250 mL. COD was assayed according to CPPA H.3 using premade COD vials (Hach Ltd., Loveland, CO). To obtain soluble COD, 150 mL of sample was filtered through a 1.5-µm glass-fiber filter (Whatman, 934-AH). Color was assayed using CPPA H.5. Dioxins and furans were assayed according to Environment Canada Reference Method EPS 1/RM/19 by a commercial laboratory (Axys Analytical Services Inc., Sidney, BC) using a mass spectrometer (VG Ultima Autospec) equipped with a gas chromatograph (Hewlett Packard 5890). Toxicity equivalent quotient (TEQ) was calculated using only the 2,3,7,8 isomer and by assigning a value of zero to nondetectable levels. AOX was assayed according to CPPA H.6P by a commercial laboratory (Econotech Services Ltd., New Westminister, BC) using an AOX analyzer (Mitsubishi TOX-10). Chronic toxicity was assayed by a commercial laboratory (BC Research Inc., Vancouver, BC) using *Ceriodaphnia dubia*, as outlined in Environment Canada Reference Method EPS 1/RM/21. MFO induction was assayed using an H4IIE rat hepatoma cell line, following the methods outlined by van den Heuvel *(6)*. Because whole-mill effluent was toxic to cells in culture, extracts only were tested. Whole-dried effluent; particulates sedimented by centrifugation (10,000 *g*, 2.5 hours); humic substances removed by flocculation with diethylaminoethyl (DEAE) cellulose; and hydrophobic compounds



4. Color removal from effluent that had been biotreated to varying final BOD_5 values (jar tests)



5. COD concentration after primary, secondary, and tertiary treatment in full-scale treatment system

stripped by C-18 filtration were extracted in methanol and solvent exchanged to dimethylsulfoxide (DMSO) for bioassay.

RESULTS AND DISCUSSION

Effect of operating parameters on TCRS performance

Performance of the TCRS was evaluated as a function of a range of operating parameters, including temperature, pH, coagulant dose, and BOD addition. Response variables included color and COD.

Effect of pH. The dual-polymer system functioned effectively over a wide range of pH values, as seen in **Fig. 2**. Color removal varied from a high of 71.1% at pH 3 to a low of 54.3% at pH 11, a less dramatic variation than has been previously reported *(7)*. This observed difference in the effect of pH may be a result of using different coagulants (for example, tertiary amines are more pH sensitive than the quaternary amine used in this study) or to differences in effluent character.

Color and COD removal improved with decreasing pH down to a value of pH 3. Below this value, the high concentration of hydrogen ions may interfere with bonding between the flocculant and the chromophore. Alternatively, the charge of the chromophores may be decreased because of protonation of ionizable groups, which occurs at low pH values. Operating at lower pH values can significantly affect the ionic nature of many constituents of kraft pulping effluents.

Despite improved color and COD removal at pH values as low as 3, operating at such low values of pH impedes floc formation and therefore can result in substantial carryover of particulate material in the full-scale DAF system.

Effect of coagulant dose and temperature. Color and COD removal improved with increasing dose of coagulant and decreased slightly with temperature, as seen in **Table III**. Each column of values in Table III illustrates the typical impact of increasing coagulant dose on removal of color and COD. Although the removal improves with increasing coagulant dose, the marginal improvement decreases.

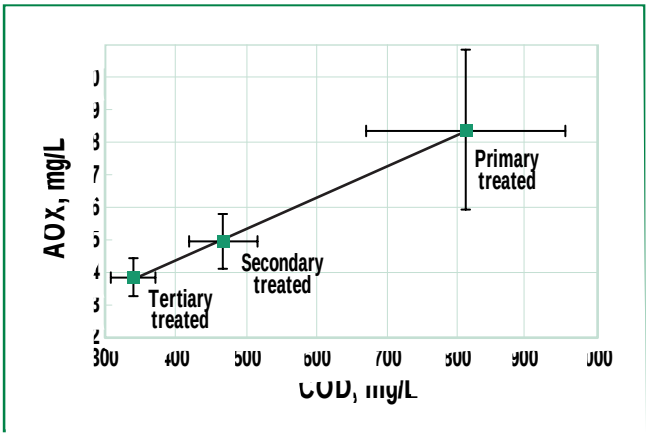
It is interesting to note that while performance deteriorated slightly during operation at elevated temperatures, floc characteristics improved. Large, rapidly settling flocs were more readily produced when operating at higher temperatures. Enhanced floc formation at higher temperatures can reduce costs by allowing the application of lower coagulant doses. The resultant cost savings have the potential of more than offsetting the small loss of performance.

Coagulant dose, ppm	COD REMOVAL, %			COLOR REMOVAL, %		
	6.7°C	23°C	40°C	6.7°C	23°C	40°C
24	22	23	14	38	40	38
48	32	32	31	51	50	49
72	36	35	32	58	56	55
96	40	39	34	63	61	60
120	41	40	37	67	66	65

III. Effect of coagulant dose and temperature on percent removal of soluble COD and color in jar tests

Effect of BOD. Color removal was not affected by soluble, readily biodegradable BOD over the range tested, as seen in **Fig. 3**. However, while readily biodegradable BOD represents the majority of BOD in clarified primary effluent, a considerable portion of the BOD is present as slowly biodegradable and/or colloidal material. Since this BOD can exert a much stronger interfering effect on tertiary treatment than the soluble materials tested—and since biological treatment is typically able to effect nearly complete removal of colloidal BOD—further tests were conducted to determine the influence of the complete spectrum of BOD-exerting compounds found in effluent.

Figure 4 shows the results of tests that were conducted on effluent that had been biologically treated to vary-



6. Correlation between AOX and COD values across the full-scale TCRS. Each data point is an average of five determinations of COD and AOX, and error bars represent ±1 standard deviation from the average value.

ing degrees. Again, the results indicate that BOD exerted little impact on performance of the dual-polymer color-removal system. Previous work has shown that, under certain operating conditions, it is advantageous to carry out biological treatment prior to color removal *(7)*. However, our study indicates that biodegradable material normally removed during biological treatment of effluent does not interfere with tertiary color removal.

Performance of full-scale TCRS

Effect of tertiary treatment on COD concentration. Over the period of the study, 45% of effluent COD was removed during secondary treatment. The tertiary treatment system removed an additional 13% (24% of the residual) or 17.3% (31.5% of the residual) COD expressed as total COD or soluble COD, respectively. **Figure 5** shows the COD levels in the effluents from primary, secondary, and tertiary treatment during the course of this study. COD removal across the TCRS was considerably lower than the 60% color removal that was achieved. This indicates that the polymer system exhibited some degree of selectivity toward the chromophores, perhaps a result of higher molecular weight or greater charge density than the bulk of the COD.

Governments worldwide are moving toward COD regulations *(8)*. In Australia, new regulations limit monthly average COD discharge to 25 kg/metric ton. In Sweden, recent permits have limited annual average COD discharges to 13–20 kg/metric ton. In the United States, new EPA guidelines limiting effluent COD and AOX (among other parameters) were released in 1997. The COD limit originally proposed by the U.S. EPA in 1993 for bleached kraft mills was 25.4 kg/metric ton. Later proposals suggested modifying this to either 30.4 kg/metric ton or 45.6 kg/metric ton *(3)*. However, EPA has postponed setting limits on COD until Phase II implementation of the Cluster Rules.

At CFI, effluent COD concentrations measured after secondary treatment and after tertiary treatment are 28.1 kg/metric ton and 21.3 kg/metric ton, respectively. Thus the implementation of the TCRS at CFI would have allowed the mill to achieve the COD limit originally proposed by the EPA. While it would not be economically feasible to install such a tertiary treatment process for removal of COD, mills that have already invested in such technologies may be in a better position to meet new guidelines with minimal additional capital expenditures.

Effect of tertiary treatment on AOX concentration. At CFI, approximately 45% of AOX is removed during secondary treatment, a value in good agreement with recently published results *(9)*. The TCRS removes 23% of the residual (or 13% of the original) AOX. There is a strong correlation between average AOX and total COD values at each sample point, as seen in **Fig. 6**.

Since December 1995, BC pulp and paper mills have been limited to a liquid AOX discharge of less than 1.5 kg/air-dry (a.d.) metric ton *(10)*. Oxygen delignification along with 100% ClO_2 substitution allows the CFI mill to meet this level using conventional primary and secondary effluent treatment. However, it is noteworthy that the tertiary treatment system reduced the AOX discharge from 0.281 kg/a.d. metric ton to 0.216 kg/a.d. metric ton. For mills in other jurisdictions with impending AOX regulations, tertiary color-removal systems may prove invaluable in complying with stricter effluent limits.

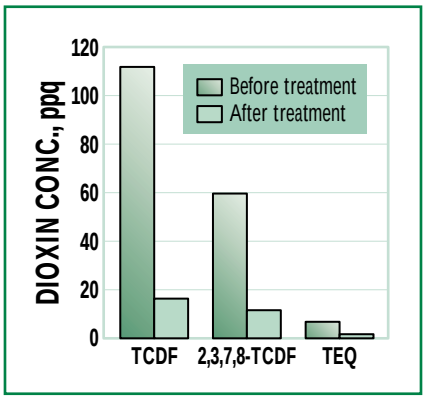
Effect of tertiary treatment on TCDF concentration. Because of their toxicity and persistence, the level of dioxins and furans in bleached kraft mill effluents has been regulated under the Canadian Environmental Protection Act since 1992 *(11)*. Final treated effluent from chemical pulp mills must contain nondetectable levels of 2,3,7,8-TCDD (tetrachlorodibenzo-*p*-dioxin) and 50 ppq or less of 2,3,7,8-TCDF. In response to this challenge, the Canadian pulp and paper industry has reduced the discharge of 2,3,7,8-TCDD in effluent from 450 g/year in 1988 to about 2 g/year in 1994 *(12)*. This dramatic reduction was achieved through a combination of technology changes, especially increased ClO_2 substitution and elimination of chemicals containing dioxin precursors.

Despite the virtual elimination of dioxin and furan production, benthal sediment deposits in ASBs can contain significant levels of these compounds, deposited over 20–30 years of operation with chlorine bleaching. Previous work at a different mill site showed that 2,3,7,8-TCDF—present in sediments at a mean concentration of

KEYWORDS
Coagulation, flocculation, tertiary treatment, decoloration, effluent treatment, parameters, filtrates, Canada, bleaching effluents, drainage jars

End point, % v/v	SECONDARY TREATMENT Survival	Reproduction		TERTIARY TREATMENT Survival	Reproduction	
IC_{25}	>100	12.0	(7.1, 17.6)	17.2	(11.5, 26.8)	3.7 (2.2, 6.4)
IC_{50}	>100	26.2	(21.5, 32.0)	25.0	(18.7, 40.0)	7.7 (4.9, 11.8)

IV. Survival and reproduction of **Ceriodaphnia dubia** *invertebrates in secondary and tertiary-treated effluent from full-scale treatment system (95% confidence limits are in parentheses)*



7. Furan concentration before and after full-scale tertiary treatment

44,200 ppt—could be transported into mill effluent, causing the mill to exceed permitted limits *(13)*. In that study, partial dredging of the lagoon was sufficient to control TCDF concentrations in the effluent.

Despite the fact that benthal sediments in the ASB at the CFI mill in Skookumchuck were found to be responsible for significant TCDF values in the effluent, the installation of the TCRS has forestalled the need for remediative measures. Removal of TCDF, 2,3,7,8-TCDF, and TEQ across the TCRS was 83.4%, 79.6%, and 81.9%, respectively, as seen in **Fig. 7**. Equally important, fluctuations in the TCDF loading to the TCRS were not reflected by variations in TCDF concentrations at the outfall (results not shown). Similar high removal efficiencies were observed in bench-scale tests of chemical precipitation technologies (alum, lime, and polymeric coagulants) in a multimill study *(14, 15)*.

Effect of tertiary treatment on chronic toxicity. The results in **Table IV** show that tertiary treatment has a negative impact on effluent chronic toxicity as measured by *Ceriodaphnia dubia*. IC_{25} and IC_{50} values (effluent concentrations that inhibit 25% and 50% of the test organisms, respectively) for survival and reproduction decreased following tertiary treatment. This increase in chronic toxicity across the tertiary treatment system is likely, to a degree, assay specific. *Ceriodaphnia* were physically entrapped in and immobilized by residual flocculant.

Effect of tertiary treatment on induction of mixed-function oxygenases. The capacity for effluent methanol extracts to induce mixed-function oxygenase activity decreased as the wastewater progressed through primary, secondary, and tertiary treatment, as seen in **Fig. 8**. Methanol extracts of whole-mill and nonparticulate-associated tertiary-treated effluent showed an approximate 18% decrease in capacity to induce mixed-function oxygenase activity compared with secondary-treated effluent (Fig. 8). When broken down into particulate-associated and nonparticulate-associated activity, most of the decrease was associated with the nonparticulate (soluble) effluent component. Particulate-associated inducing activity decreased only marginally (10.9%) across the tertiary treatment system. Note, however, that this assay measured total activity associated with the particulate matter. Therefore, the increase in particulate-associated activity reflects the fact that the TSS concentration nearly doubled across the tertiary treatment system (Table I).

Treatment of concentrated D_1- and E_1-stage filtrates

As an alternative to treating combined mill effluent, it may be advantageous to treat the major color-bearing streams separately. In most mills, the major color load comes from the first extraction stage, with an additional significant contribution from the first bleaching (D_1) stage. As part of this study, we examined the potential for color removal from each of these streams separately and in combination.

Treatment of segregated D_1-stage filtrate produced large, rapidly settling flocs and resulted in a maximum removal of 55% color, as seen in **Table V**. This degree of treatment could be achieved only by using coagulant doses 3–4 times those used for treatment of combined secondary-treated effluent. COD removal varied from 4% (at the lowest coagulant dose tested) to 13% at a coagulant dose of 264 ppm (Table V).

In contrast to the treatment of D_1-stage filtrate, floc formation was poor, and coagulant consumption was excessive in E_1-stage filtrate. Despite the poor floc structure, removal of color and COD compared reasonably well with that achieved in combined mill effluent.

Up to 66% of color and 31% of COD could be removed from combined D_1- and E_1-stage filtrates. However, a coagulant dose of approximately 360 ppm was required to achieve these levels. Flow and color-load balances revealed that consumption of coagulant would be almost identical, regardless of whether the treatment was carried out on the segregated bleach-plant effluent or, as is practiced now, on the combined mill effluent. However, the reduced volumetric loading associated with treatment of segregated bleach-plant effluent could result in capital cost savings.

CONCLUSIONS

Tertiary treatment (coagulation and flocculation) can produce excellent color removal over a broad range of temperature, pH, and BOD. Moreover, tertiary treatment can have a beneficial impact on other wastewater parameters, including COD (24% removal), AOX (23% removal), 2,3,7,8-TCDF (80% removal), and mixed-function oxygenase-inducing activity (18% reduction). While treatment of segregated bleach-plant effluents may not reduce operating costs, future pilot-

scale studies may indicate that the installed cost for a system that treats segregated bleach-plant filtrates could be lower than a system designed to treat whole-mill effluent.

Tertiary coagulation and flocculation technologies have the potential to help mills without state-of-the-art process equipment to meet today's stringent regulations limiting effluent discharge. For mills that have already invested in technologies such as oxygen delignification, tertiary effluent treatment produces an effluent that is considerably improved over the industry average. TJ

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	COLOR REMOVAL			COD REMOVAL		
	Maximum, removal, %	Coagulant dose for color removal, ppm 50% of max.	100% of max.	Maximum removal, %	Coagulant dose for COD removal, ppm 50% of max.	100% of max.
D_1	55	44	264	13	70	264
E_1	69	318	744	41	248	744
$D_1 + E_1$	66	100	504	31	100	504

V. Color and COD removal in jar tests with concentrated D_1- and E_1-stage filtrates

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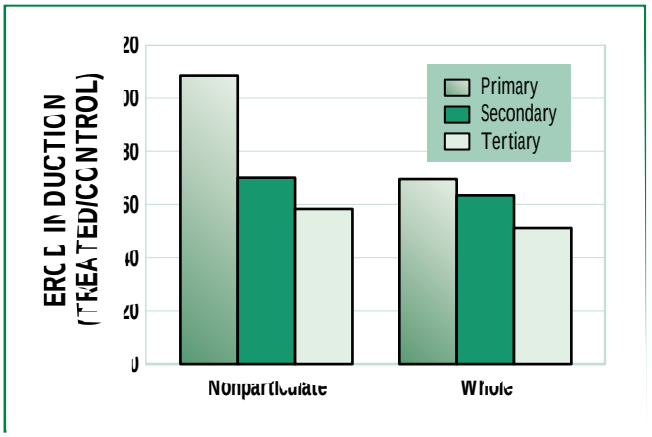

8. Ethoxyresorufin o-deethylase (EROD)-inducing capacity of methanol extracts of whole-mill effluent and nonparticulate fraction across full-scale primary, secondary, and tertiary treatment

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