Improvement of eucalyptus bleached kraft pulp effluent treatment through combined ozone-biological treatment

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ABSTRACT: The aim of this work is to evaluate the combination of ozonation and biological treatment for reducing recalcitrant organic matter and enhancing the biological treatment efficiency of kraft pulp elemental chlorine-free (ECF) bleaching and similar effluents. Alkaline bleach filtrate was treated with ozone at doses of 100 and 250 mg/L, temperature of 70°C, and pH 10 (mill conditions). The ozone-treated and untreated effluents were mixed with acid bleaching filtrate to prepare the combined effluents, which were then treated in a bench-scale activated sludge system. The constant biological treatment conditions used were: temperature = 35±2°C, hydraulic retention time = 12 hours, and sludge retention time = 10 days. Effluents were also fractionated by molecular mass using a membrane with a 500-Da molecular-weight cutoff to characterize the high- and low-molecular-mass organic matter. Ozonation combined with biological treatment resulted in statistically significant increases in removal of COD, BOD_5, TOC, lignin, and AOX compared to the effluent without ozone treatment. The high-molecular-mass fractions represented the majority of all components analyzed, and their contribution to total effluent load increased after biological treatment because of the greater removal efficiency of low-molecular-mass organic matter.

Application: This study reinforces the need to develop wastewater treatment technologies capable of degrading high-molecular-mass organic components, which continue to represent a potential risk to aquatic environments.

Brazilian water resources legislation has imposed charging for water use and effluent pollutant discharge, which may represent a considerable increase in the cost of manufacturing products that rely on intensive water use, such as pulp and paper. Therefore, the pulp and paper industry has shown considerable interest in developing technologies that minimize effluent pollutant loads. Although most of the biodegradable compounds in pulp mill effluents are removed during biological treatment, these effluents still contain considerable quantities of recalcitrant compounds that are not removed by conventional treatment processes. The effective removal of this recalcitrant organic matter is currently a primary objective of mill effluent treatment system managers. One way to achieve this removal is through chemical oxidation, for example by advanced oxidation processes based on generation of hydroxyl radicals (·OH), powerful oxidizers that are capable of oxidizing virtually all organic compounds [1]. Chemical oxidation can be used alone or as a pretreatment; in the latter case, the aim is to oxidize compounds partially to more biodegradable products which can then be removed more economically in a subsequent biological treatment [2].

Researchers have already shown that it is possible to increase COD removal by combined ozone-biological treatment of kraft pulp bleaching effluent [3,4]. However, alkaline bleaching filtrates typically contain more recalcitrant high-molecular-mass chlorinated compounds than do acid filtrates [5,6] and ozonation at high pH favors formation of ·OH radicals that react much more rapidly and nonselectively with organic compounds present in effluents than does ozone itself [7]. Therefore, the objective of the present study was to evaluate the use of ozone for pretreatment of alkaline bleaching filtrate alone, followed by biological treatment of combined acid + alkaline filtrates to increase the overall treatment efficiency of bleached kraft pulp effluent. The choice of alkaline filtrate for ozone treatment stems from its high pH (>10) and high recalcitrant organic load, plus the fact that treating it separately would decrease the effluent volume to be treated later, thus reducing the cost of ozone treatment. The effects of chemical and biological treatments and their combination on the high- (>500 Daltons) and low- (<500 Daltons) molecular-mass organic matter were also investigated to provide a better understanding of the effects of the treatments.

MATERIAL AND METHODS

Effluents

Researchers collected two effluent samples from a 1 million tpy Brazilian kraft pulp mill $D_0(EOP)D_1P$ bleaching line. Samples were collected from the D_0 and D_1 chlorine dioxide stages (acid filtrate) and the EOP and P alkaline stages (alkaline filtrate). The effluents were characterized and stored at 4°C until used. Characterization of the effluent included analysis of pH, chemical (COD) and biochemical (BOD_5) oxygen demands, total organic carbon (TOC), adsorbable organic halides (AOX), and lignin (reported as phenol) according to the

Component	Acid Filtrate	Alkaline Filtrate	Combined Effluent
pH	3.4	10.0	7.0
COD, mg O_2 L^{-1}	1,274 ± 18	1,146 ± 21	1,133 ± 4
BOD_5, mg O_2 L^{-1}	362 ± 4	307 ± 5	315 ± 13
BOD_5/COD	0.28	0.27	0.28
TOC, mg C L^{-1}	522 ± 4	453 ± 5	530 ± 2
Color, mg Pt.L^{-1}	940 ± 16	487 ± 13	658 ± 17
Lignin, mg phenol L^{-1}	22.0 ± 0.7	6.7 ± 0.3	14.3 ± 0.7
Carbohydrates, mg L^{-1}	187 ± 8	143 ± 4	139 ± 5
AOX, mg Cl^- .L^{-1}	16.0	8.3	13.9
Acute toxicity, % mortality	0	0	0

I. Initial characteristics (average ± one standard deviation) of the acid and alkaline filtrates and combined effluent (filtrate mixture).

APHA *Standard Methods* [8], of color by the Canadian Pulp and Paper Association method [9], and of carbohydrates according to the method described in [10]. Acute toxicity was evaluated after adjusting effluent pH to 7±0.2, using the microcrustacean *Artemia salina* as the test organism, following the method described in [11]. TOC was quantified in a Shimadzu TOC 5000 (Tokyo, Japan) automatic analyzer and AOX in a Euroglas 1600 (Delft, Holland) automatic analyzer. All analyses were performed in duplicate, and average values are reported. **Table I** presents the initial characteristics of the effluents used in this study.

Ozonation

Ozonation was performed only on alkaline filtrate without pH adjustment (pH 10.7–10.9). Ozone was produced from pure oxygen in a laboratory generator (Sumitomo Precision Products, Model SG01A, Japan). The ozone flow rate was adjusted to 10–12 mg/min and doses of 100 and 250 mg/L were applied. Ozonation was carried out in a one-liter-capacity vertical glass reactor immersed in a 70ºC water bath. The reactor was coupled to a second reactor containing a potassium iodide solution (100 mL of 1N KI, 500 mL of 4N H_2SO_4, and 2500 mL of distilled water) to quantify residual ozone by titration with sodium thiosulfate.

Biological treatment

Untreated and ozone-treated (100 and 250 mg/L) effluents were mixed with equivalent volumes of acid filtrate, and their pH was adjusted to 7±0.2 for biological treatment. Treatment was carried out in continuous bench-scale activated-sludge reactors (500mL working volumes). Before biological treatment, nitrogen (NH_4Cl) and phosphorus (H_3PO_4) were added to reach a final DBO_5:N:P ratio of 100:5:1. Temperature was maintained at 35±2ºC, and aeration was performed through porous stones attached to an air pump. Dissolved oxygen (DO) was kept above 3 mg/L. The reactors were inoculated with activated sludge from the treatment system of the same mill where the effluents were collected. Effluent feed and sludge recirculation from the secondary clarifier were carried out using a peristaltic pump (Gilson, Minipuls, France). Hydraulic retention time was 12 hours. Mean cell residence time was kept at 10 days by removal of appropriate volumes of sludge directly from the biological reactor.

The COD, volatile suspended solids (VSS), pH, and dissolved oxygen (DO) components were monitored daily. The system was considered to have stabilized when the effluent COD varied less than 10% in three successive measurements [12]. After stabilization, the treated effluent was collected once a day on six consecutive days for measurement of BOD_5, COD, lignin, carbohydrates, color, TOC, toxicity, and AOX.

Molecular mass separation

Effluents were first filtered using glass-fiber filters (Millipore AP40, Billerica, United States) after adjusting their pH to 7±0.5, and 100 mL aliquots were then filtered through cellulose acetate membranes with a molecular-weight cutoff of 500 Daltons (Da) (Amicon YC05, Millipore, United States) in an ultrafiltration cell (Amicon 8400, Millipore, United States). Filtration was terminated after passage of 75% of the effluent volume. After filtration, the membrane was immersed in 40 mL of distilled water at 30ºC and placed in an ultrasonic bath to release high-molecular-mass compounds that were retained on the membrane. Filtrate and retentate were adjusted to their original volume by addition of distilled water and characterized as previously described. Recovery of organic matter in each effluent after fractionation was calculated using the equation:

$$\%\text{recovery} = \frac{(HM + LM) \times 100}{T} \quad ,$$

where:
HM = value of component in high-molecular-mass fraction
LM = value of component in low-molecular-mass fraction
T = value of component in original effluent.

Statistical analysis

Researchers collected six samples of each effluent after biological treatment. Analysis of variance was performed on all components analyzed ($\alpha = 0.05$), followed by comparison of component means by the Tukey test ($p<0.05$) to identify significant differences in effluent quality resulting from ozone use.

Component	100 mgO_3/L		250 mgO_3/L	
	mg/L	Removal, %	mg/L	Removal, %
COD	1,102	2.7	1,083	4.4
BOD_5	361	-14.6	380	-20.7
TOC	494	6.9	491	7.4
Color	616	6.3	602	8.5
Lignin	11.8	17.9	11.3	21.3
Carbohydrates	135	2.8	135	2.9
AOX	13.0	6.6	12.5	10.2

II. Effect of ozonation on organic matter removal from eucalyptus alkaline ECF bleaching filtrate (negative values indicate an increase).

RESULTS AND DISCUSSION

Effect of ozone

Table II presents the effect of ozonation on the different components analyzed.

Results are reported relative to the dose applied, although residual ozone was 9% at a dose of 100 mg/L and 15% at a dose of 250mg/L. As shown in Table I, alkaline filtrate exhibited low biodegradability (BOD_5/COD = 0.27), making it a good candidate for ozonation. It is known that ozone is able to transform certain poorly degradable compounds to more easily degradable products without appreciable reduction in COD or TOC, consequently increasing the effluent BOD_5/COD ratio [13]. In the present study, an increase in BOD_5 of approximately 15% at a dose of 100 mgO_3/L and 21% at a dose of 250 mg O_3/L was observed. COD reductions were only 3% and 5%, resulting in biodegradability increases of 19% and 27% at doses of 100 and 250 mgO_3/L, respectively. Partial oxidation of dissolved organic matter by ozone was the desired effect of the chemical pretreatment and indicates that the oxidizing power of ozone was not wasted on mineralization of organic compounds (COD removal), which can be done at lower cost in a subsequent biological treatment. Bijan and Mohseni [6] reported 10%–15% increases in the biodegradability of a softwood alkaline bleaching filtrate after ozonation with doses ≤ 250 mg/L.

Percent removal of all components except BOD and carbohydrates increased with increasing ozone doses, demonstrating the preferred reaction of ozone/hydroxyl radicals with more recalcitrant compounds such as lignin, as already reported in other studies [3,4]. Among the components analyzed, lignin showed the highest percent removal (18% to 21%), followed by AOX (6.6% to 10.2%) and color (6.4% to 8.5%), with an increase from 100 to 250 mgO_3/L. Lignin degradation is a desirable effect of ozonation, given its recalcitrance in biological treatment [14]. Color and AOX removals confirm literature reports of ozone's reactivity with color-causing compounds [13,14,15] and organochlorine compounds [15,16,17].

Effects of biological treatment

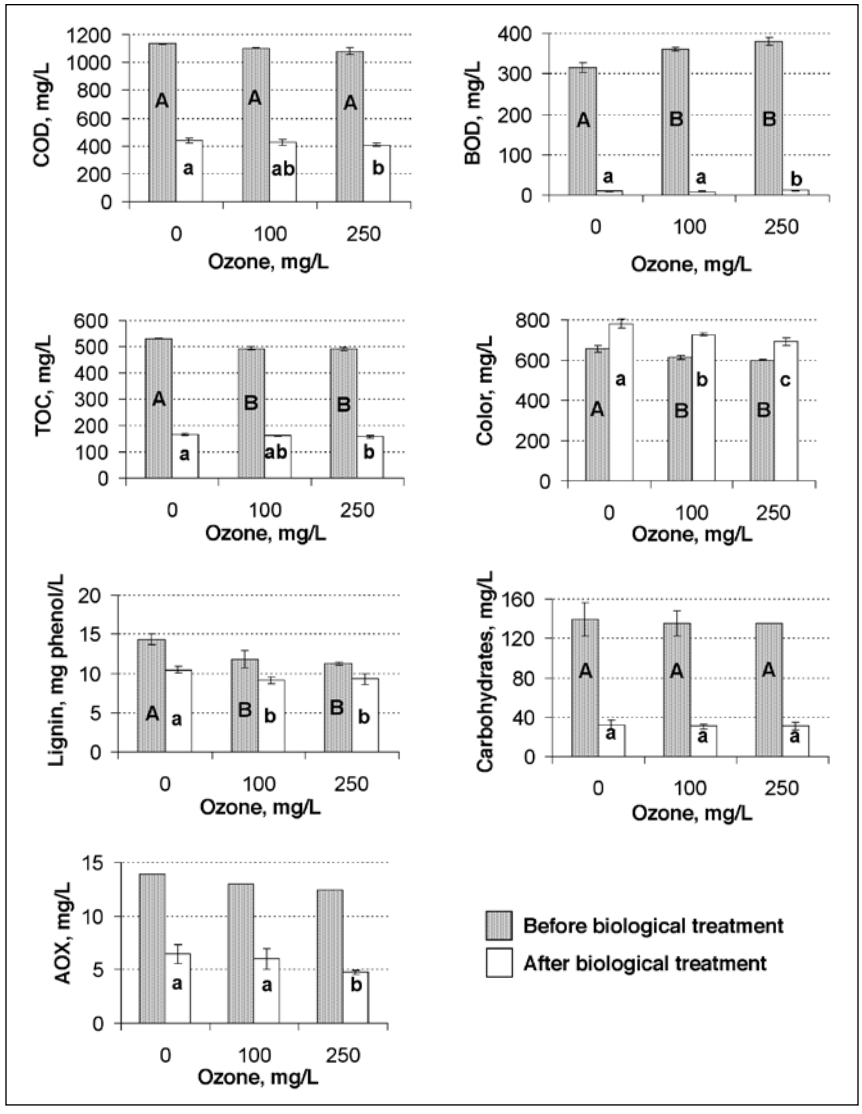
Component values before and after biological treatment are shown in **Fig. 1**. Pretreatment with 250 mgO_3/L resulted in significantly higher removal of COD, BOD, TOC, and AOX, while pretreatment with 100 and 250 mgO_3/L resulted in significantly greater removal of color and lignin after biological treatment.

Even with the increase in BOD after ozonation (Table II), BOD removal was greater after the combined treatment, demonstrating the advantage of transforming organic matter from non-biodegradable to biodegradable form during the oxidative treatment. The increase in COD removal in the combined treatment, although significant, was still small (from 61% to 64% at 250 mgO_3/L). This level of total removal is similar to that obtained when a combined ECF bleaching effluent was treated with ozone and then biologically treated [3,4].

Most bleaching-effluent ozone pretreatment studies have concentrated on treating alkaline filtrates because these normally contain the greater part of the organic load [5,6,7,14,18]. However, for the bleaching effluent used in this study, the acid filtrate presented a greater organic load than the alkaline filtrate (Table I). The relatively high concentration of high-molecular-mass organic matter in modern kraft pulp acid-bleaching filtrates has been reported recently [19] and is due to the adoption of new bleaching technologies such as the hot chlorine dioxide stage (DHT). For eucalypt bleaching effluents, therefore, the main advantages of performing ozonation on alkaline filtrate alone are the chance to generate highly reactive hydroxyl radicals and the reduction of the total ozone charge applied per volume of effluent treated.

After biological treatment, a higher percent removal of TOC (68% to 70%) than AOX (53% to 62%) was observed, reflecting the greater removal during biological treatment of organic compounds containing little or no chlorine than of more highly chlorinated organic compounds, as previously reported [20,21]. However, at an ozone dose of 250 mg/L, AOX removal (62%) approached TOC removal efficiency (68%), demonstrating ozone's reactivity toward organochlorine compounds. Because the degree of chlorination (AOX/TOC ratio) is commonly used to estimate a compound's recalcitrance and potential environmental impact [14,22], this increased AOX removal is an important advantage of using ozone.

Biological treatment increased color by 19% in effluent without ozone pretreatment, by 18% in effluent pretreated with 100 mgO_3/L, and by only 11% in effluent pretreated



1. Concentrations of components analyzed before and after biological treatment of bleaching effluent pretreated with 0 (control), 100, and 250 mg/L of ozone (error bars represent one standard deviation). For each data series, columns identified with the same letter do not differ statistically (Tukey test, p<0.05). Upper-case letters refer to effluent after ozonation and lower-case letters to effluent after ozonation and biological treatment.

with 250 mgO_3/L. Color increases after aerobic biological treatment of bleaching effluents have been widely reported [19,20,23]. The lesser color increases after the combined treatment than after biological treatment alone reflect the destruction of effluent chromophores and leucochromophores by ozone. The high efficiency of ozone as a post-treatment for color removal is well known [15], but recent studies have shown that pretreatment with ozone can be more efficient than post-treatment for COD removal [3,4], and therefore the point of ozone application will depend upon the desired effluent quality improvement.

Lignin removal increased from 26% after biological treatment to 35%–36% after combined treatment (ozone + biological) because of oxidative degradation of this recalcitrant matter during ozone pretreatment. There was no increase in carbohydrate removal in the combined treatment, reflecting the poor reactivity of this class of compounds with ozone, as previously mentioned. On the other hand, carbohydrates are readily removed during biological treatment, and their removal efficiency reached 77%–78% for the three biologically treated effluents. None of the effluents presented acute toxicity toward *Artemia salina*.

Effect of treatments on molecular mass of dissolved organic matter in effluents

The distribution of organic matter in high- and low-molecular-mass fractions

Component	0 mgO_3/L		100 mgO_3/L		250 mgO_3/L	
	Before biological (%)	After biological (%)	Before biological (%)	After biological (%)	Before biological (%)	After biological (%)
COD	96	104	87	100	86	106
BOD_5	110	na*	88	na	90	na
TOC	83	76	88	69	84	78
Color	96	89	90	87	93	82
Lignin	91	76	85	78	85	82
Carbohydrates	95	89	95	96	97	106
AOX	100	97	104	102	101	120

* na = not analyzed

III. Recovery percentages of organic matter in bleaching effluent pretreated with 0 (control), 100, and 250 mg/L of ozone. Molecular mass separation was performed on samples before and after biological treatment.

before and after biological treatment is presented in **Fig. 2**.

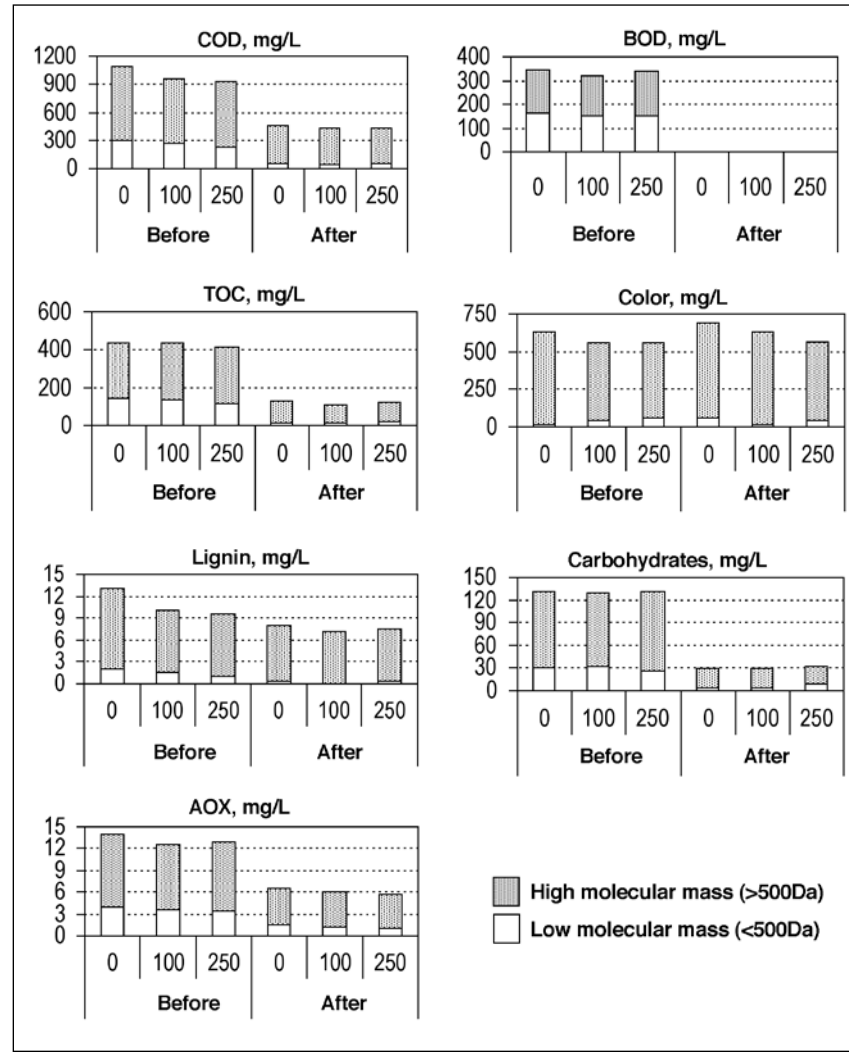
Recovery percentages of organic matter after molecular mass separation are presented in **Table III**.

For the majority of components, recovery varied from 80% to 110% of the amount in the original effluent; these values are typical of this type of analysis [3,21,22]. In general, recovery was greater from effluents before biological treatment than after biological treatment.

Ozonation of alkaline filtrate resulted in a greater increase in the biodegradability of the low-molecular-mass fraction (from 0.55 to 0.65, at a dose of 250 mgO_3/L) than in that of the high-molecular-mass fraction (from 0.23 to 0.27, at a dose of 250 mgO_3/L). These results are in contrast to those reported by Bijan and Mohseni in [6], who observed an increase of 30% to 40% in biodegradability of a softwood alkaline bleaching filtrate after ozonation with 700 mgO_3/L that was associated almost exclusively with an increase in the biodegradability of the high-molecular-mass fraction (>1000 Da).

Before biological treatment, the high-molecular-mass fractions represented 73%–75% of the COD, 68%–71% of the TOC, 53%–55% of the BOD, 90%–98% of the color, 85%–90% of the lignin, 75%–81% of the carbohydrates, and 71%–74% of the AOX in the effluents. After biological treatment, the high-molecular-mass fractions corresponded to 87%–90% of the COD, 86%–90% of the TOC, 92%–98% of the color, 78%–81% of the AOX, 96%–99% of the lignin, and 72%–91% of the carbohydrates. The high-molecular-mass fractions not only contributed the greatest part of the components analyzed, but they also presented the lowest biotreatability, with lower percent removal efficiencies than the low-molecular-mass fractions for all components (**Table IV**), with the sole exception of carbohydrates, after pretreatment with 250 mg O_3/L.

The molecular size of wastewater constituents affects their biological treatability because compounds must be assimilated into bacterial cells. Pas-



2. Organic matter distribution in high- and low-molecular-mass fractions before and after biological treatment of bleaching effluent pretreated with 0 (control), 100, and 250 mg/L of ozone. BOD_5 was not analyzed after biological treatment because of insufficient sample volume.

Component	Biological		100 mgO_3/L + Biological		250 mgO_3/L+ Biological	
	HMM (%)	LMM (%)	HMM (%)	LMM (%)	HMM (%)	LMM (%)
COD	49	83	45	83	46	76
TOC	63	88	66	92	64	86
Color	-3	-320	-22	65	-5	32
Lignin	31	83	16	98	16	68
Carbohydrates	75	89	73	92	78	64
AOX	49	63	50	65	49	68

IV. Percentage removal of analyzed components of the high- (HMM, > 500 Da) and low- (LMM, < 500 Da) molecular-mass fractions of bleaching effluent after ozone (100 and 250 mgO_3/L) and biological treatment (a negative value indicates an increase).

sive transport of hydrophilic compounds through cell membranes is considered to be restricted to compounds with a molecular weight of less than 500 Da [21]. It has already been reported that COD, color, AOX, and TOC of high-molecular-mass fractions resist biological treatment and represent the principal source of pulp mill wastewater emissions [20,22,24]. From the results shown in Fig. 2, pretreatment of the alkaline filtrate with ozone had little effect on the molecular mass distribution of COD, which probably accounts for the small COD removal efficiency improvement observed.

The results obtained in the present study confirm the greater recalcitrance of high-molecular-mass organic matter toward biological treatment (Fig. 2) and indicate that the path toward greater treatment efficiency lies in the conversion of high-molecular-mass organic matter into low-molecular-mass matter. Apparently, pretreatment with ozone was not able to effect this transformation with high efficiency. This suggests that an intermediate ozone treatment after an initial biological treatment to remove rapidly biodegradable matter may be the best strategy for combined treatment of eucalypt kraft pulp bleaching effluents.

CONCLUSIONS AND RECOMMENDATIONS

The combination of ozonation and biological treatment proved to be an effective alternative to increase the final quality of eucalypt kraft pulp bleaching effluent. Researchers observed statistically significant increases in COD (3%), AOX (13%), lignin (9%), and TOC (1.5%) after biological treatment of effluent pretreated with 250 mgO_3/L. Ozone was also effective in limiting color increase after biological treatment, with an increase of only 5% for effluent pretreated with 250 mgO_3/L, compared to an increase of 19% in the effluent without ozone pretreatment.

More studies need to be carried out to identify the best configuration for combined ozone + biological treatments to reduce treatment costs and provide viable treatment alternatives for mills in the future.

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INSIGHTS FROM THE AUTHORS

This research was motivated by an interest in minimizing the environmental impact of bleached kraft pulp mill effluents, especially their recalcitrant high-molecular-mass fraction. Other studies have indicated that ozone can increase biodegradability of untreated bleached kraft pulp mill effluent, and its potential to improve overall treatment efficiency is certainly worth further exploration. Such research is particularly challenging to perform at a time when advances in bleaching technologies in Brazilian mills are significantly altering effluent composition.

Ozonation combined with biological treatment resulted in statistically significant increases in removal of COD, BOD_5, TOC, lignin, and AOX compared to the effluent without ozone treatment. The high-molecular-mass fractions represented the majority of all components analyzed, and their contribution to total effluent load increased after biological treatment because of the greater removal efficiency of low-molecular-mass organic matter. It was especially interesting to discover the beneficial effect of ozone pretreatment on final effluent color. This indicates that structural changes occur in effluent components during this treatment, leading to reduced chromophore production during biological oxidation.

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The next step would likely be further testing of combined ozone+biological treatment in mills, especially mills that already use ozone bleaching. The effectiveness of using residual ozone from the bleach plant in wastewater treatment should also be explored.

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