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THE EFFECTS OF ALKALINE FILTRATE RECIRCULATION TOWARDS THE PROPERTIES OF LONG FIBER PULPS WITH OD(EPO)DED BLEACHING SEQUENCE

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HIGHLIGHTS

The alkaline filtrate recirculation increased the reagents load during bleaching.

The alkaline filtrate recirculation did not alter the quality of the refined pulp.

The alkaline filtrate recirculation increased calcium, chloride, sodium and sulfate levels in the D stage and sodium and sulfate levels after the (EPO) stage

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ABSTRACT

Circuit closure can reduce water consumption, but negative effects on pulp quality and equipment wear may make it unfeasible. This study aimed to evaluate how the alkaline filtrate of the recirculation stage (EP) affects bleaching, pulp quality and characteristics of the filtrates produced. Pre-delignified cellulose pulp from a mixture of three coniferous woods was used. Bleaching followed the D(EPO)DED sequence, with the addition of 5, 10 and 15 kg·odt^{-1} of the alkaline filtrate (EP) to the pre-O_2 pulp. The physical and mechanical cellulosic pulp properties were evaluated in the control and with 10 kg·odt^{-1} of the alkaline filtrate. The inorganic compound accumulation in the system was evaluated in the control pulp and with 15 kg·odt^{-1} of the alkaline filtrate. The filtrate use increased the ClO_2 consumption for bleaching and sulfuric acid and NaOH to adjust the pH of the stages. The pulp tensile index was higher and the tear index lower with the filtrate use in pulp without refinement, however the properties of refined pulp were similar between treatments. The filtrate increased the calcium, chloride, sodium and sulfate levels in the D stage and that of sodium and sulfate after the (EPO) stage. Filtrate recirculation can reduce water use, but it increases bleaching costs and metal accumulation in the system.

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INTRODUCTION

Circuit closure can recycle water and chemical reagents, reducing pollutant loads (Frigieriet et al., 2015; Frigieri et al., 2016). Bleaching is the industrial stage with the highest water consumption and reversing this scenario would result in economic and environmental gains (Kansalet al., 2008; Kamali and Khodaparast, 2015). The alkaline bleaching filtrate has characteristics similar to those of the black liquor normally sent to the recovery boiler and could therefore be recirculated (Kamali and Khodaparast, 2015).

The chloride and metal concentrations are lower in the alkaline than in the acid filtrate, making its reuse more feasible (Huberet al., 2014). In addition, sodium can be recovered from alkaline filtrates, which have high color concentrations and biochemical (BOD) and chemical (COD) oxygen demands (Blackwell, 1992; Martin et al., 1996; Kamali and Khodaparast, 2015).

Effluent recirculation accumulates contaminants during the process, increasing chemical reagent consumption during bleaching and causing equipment corrosion (Amaral et al., 2012; Amaral et al., 2014). The brown pulp must be washed to minimize these effects, which increases water use (Towerset al., 2002;Toczyłowska-Mamińska, 2017).

The objective of this study was to evaluate how recirculation of part of the alkaline extraction filtrate (EPO) affected bleaching, final pulp quality and the characteristics of the filtrates generated. The results obtained could help in the implantation of the closed circuits in the bleaching of cellulose pulp mills.

MATERIAL AND METHODS

Sample collection and preparation

Pre-delignified cellulose pulp and bleaching filtrates were collected in a Kraft pulp mill in western Canada that uses a softwood mixture (*Picea* spp., *Pinus* spp. and *Douglas fir*). The pulp sample was washed with excess water to remove the maximum of impregnated carryover material and maintain its characteristics constant during the experiment (Table 1).

Recirculation of the alkaline filtrate of the D(EPO) DED sequence

Organic loads of five, 10 and 15 kg·odt^{-1} of the alkaline filtrate (EP) were added to the pulp after the pre-O_2 stage to simulate filtrate recirculation. These values are commonly found in effluents from pulp mills.

TABLE 1 Pulp characteristics from softwoods (*Picea* spp., *Pinus* spp. and Douglas fir) after excessively washed.

Properties	Methodology	Pre-O_2 pulp
Kappa number	TAPPI – 236	21.1
pH	pH meter	8.7
Viscosity (mPa.s)	TAPPI 230 om 94	28.7
Brightness (%ISO)	ISO – 2469	30.64
Total solids (mg·kg^{-1})	(TAPPI T 650 om-05) (TAPPI, 2007)	448
K+ (mg·kg^{-1})	TAPPI – 266	226
Na+ (mg·kg^{-1})	TAPPI – 266	2063
Cl- (mg·kg^{-1})	TAPPI – 266	64
Ca2+ (mg·kg^{-1})	TAPPI – 266	2968

Pulp bleaching

The pulp was bleached with the D(EPO)DED sequence (Table 2). The "D" represents stages with chlorine dioxide, "E" with sodium hydroxide, "P" with sodium peroxide with alkali and "O" oxygen with alkali. The "()" represents the absence of washing between the stages. Bleaching was done in a pilot bleaching plant developed by Paprican (Berry, 2000).

The first bleaching was adjusted to achieve brightness of 89.5 ± 0.5% ISO in the pulp without organic load. This fixed dosage was used in the pulp bleaching with organic loads of five, 10 and 15 kg·odt^{-1} of the alkaline filtrate (EP) (Table 2). The pH at the end of stage D, ClO_2 consumption, kappa number, brightness after (EPO) stage and final brightness were evaluated (Table 2).

The second bleaching was carried out until the pulps of all treatments reached 89.5 ± 0.5% ISO. The ClO_2 consumption in the bleaching and consumption of H_2SO_4 and NaOH to adjust the final pH were evaluated.

Physical and mechanical pulp characterization

After bleaching, the physical and mechanical pulp properties without carryover and with the addition of 10 kg·odt^{-1} were evaluated. The number of rotations for refinement was chosen to facilitate the development of pulp properties. Each test was replicated five times (Table 3).

The tear and tensile indexes, as well as pulp viscosity without refinement were compared using the t test at 5% significance. The statistical analysis of the identity model was performed to evaluate if the refinement level and pulp

TABLE 2 D(EPO)DED bleaching sequence using fixed dosage of the reagents in all treatments.

	D	(EPO)	D	E	D
Kappa factor	0.16	-	-	-	-
ClO_2 (kg·odt^{-1})	-	-	*10*	-	*5*
NaOH (kg·odt^{-1})	-	*12.8*	-	*5*	-
O_2 (psi)	-	*45*	-	-	-
H_2O_2 (kg·odt^{-1})	-	*5*	-	*2.5*	-
Retention time (min)	45	*30+60*	*120*	*60*	*180*
Consistency (%)	4	*10*	*10*	*10*	*10*
Temperature (°C)	60	*75*	*70*	*75*	*75*
Final pH	2.8-3.2	*10-10.5*	*3,0-3,5*	*9.5-10*	*3.5-4.5*

TABLE 3 Methodology for physical and mechanical properties evaluation of pulps after bleaching.

Analysis	Referência
Consistency	TAPPI – 240
Sheets confection	TAPPI – 218
Viscosity	TAPPI 230 om 94
Tensile index	TAPPI – 494
Tear index	TAPPI – 414
Drainability (Freeness)	TAPPI - 227 om 94

properties without carryover and with 10 kg·odt^{-1} could be adjusted in a model at 5% of significance.

Characterization of inorganic compounds in the system

The acetate, carbonate, chloride, chlorate, formate and sulfate content in the effluent were verified according to TAPPI 669 and the metal and total oxalate content according to TAPPI - 266.

RESULTS AND DISCUSSION

Potassium permanganate consumption pulp with the presence of carryover from (EPO) filtrate

Addition of the alkaline filtrate increased the pulp kappa number. The filtrate has large quantities of lignin and extractives (Huber et al., 2014), which increases chemical reagent consumption such as chlorine dioxide and ozone (Brogdon et al., 2015, Sarto et al., 2015) (Figure 1).

The carryover presence from the (EPO) stage increased the potassium permanganate consumption linearly by 17% with an organic load at 15 kg·odt^{-1}. Similar behavior was reported for *Eucalyptus globulus* pulp

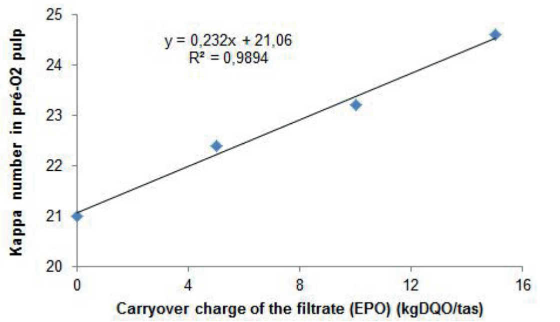

FIGURE 1 Relation between the carryover charge of the filtrate and the kappa number of the pre-O_2 pulp from softwoods.

(Barroca et al., 2002), but to a lesser extent. This happened because the alkaline filtrate compounds of the bleaching sequence from long fiber pulp consumed more potassium permanganate than the resultant of the bleaching sequence from hardwoods (Brogdonet al., 2015).

Carryover recirculation and consumption of chemical reagents

The carryover uses increased pH values, kappa number and reduced pulp brightness after the (EPO) stage and after complete bleaching sequence (Table 4).

Recirculation of 15 kg·odt^{-1} of carryover increased the kappa number by 29.9% and decreased brightness by 8.23% and 1.56% after (EPO) stage and after the entire bleaching sequence, respectively. Therefore, the kappa number increase was more significant, indicating the presence of permanganate oxidizable filtrate compounds, which do not affect pulp brightness (Sezgiet al., 2016). In addition, the greater reduction in brightness after the (EPO) stage indicates that those which followed partially corrected the negative impact of the carryover.

TABLE 4 Final pH (pH), ClO_2 consumption (ClO_2%), kappa number after EPO stage (Kappa EPO), brightness after EPO stage (EPO Bri.) and final brightness (F.B.) after OD(EPO) DED sequence using fixed dosages of chemical reagents.

Sequence	pH (D)	(ClO_2 %)	Kappa EPO	EPO Bri.	F.B.
Control	2.84a	100	5.21a	61.57a	89.43a
5 kg·odt^{-1}	3.25b	100	5.32b	61.36b	89.35b
10 kg·odt^{-1}	3.98c	99	5.67c	60.23c	88.31c
15 kg·odt^{-1}	4.5d	95	6.77d	56.5d	88.03c

Means followed by the same letter per column do not differ by the Tukey test at 5% probability.

The addition of the alkaline filtrate to the pre-O_2 pulp, mainly with 15 kg·odt^{-1} required pH adjustment in the D stage. Increasing the pH reduces the performance during this stage, which can be evidenced by the increased of chlorine dioxide consumption (Sevastyanova et al., 2012; Sezgi et al., 2016). Above pH 4.0, chlorite formation occurs (ClO_2^-), reducing the chlorine dioxide delignification rate. The chemical reagent loss in D stage is lower in pH from 3.0 to 4.0. However, formation of chlorate (ClO_3^-) at pH below 2.0 is also detrimental to the dioxidation stage (Dence e Reeve, 1996).

The increase in the chlorine dioxide (ClO_2), sulfuric acid (H_2SO_4) and sodium hydroxide (NaOH) load was required to reach 89.5 ± 0.5% ISO in pulps with carryover (Table 5). The increase in the chlorine dioxide (ClO_2) consumption is due to the organic load in the filtrate (Brogdon., 2015; Wilke et al., 2015). The higher pH of the D stage with 10 and 15 kg·odt^{-1} of carryover required the use of H_2SO_4 for its reduction (table 5). The NaOH consumption did not increase with 5 kg·odt^{-1} of carryover, however, the use of 10 and 15 kg·odt^{-1} of carryover increased consumption by 18 and 19%, respectively. The ClO_2 consumption generates organic acids, consuming the NaOH and, therefore, the load of the latter must be increased in the subsequent stages (Biermann, 1996; Ribeiro et al., 2014; Wilke et al., 2015).

Increasing reagent consumption in bleaching is detrimental to the economic viability of the sector, because this is the most expensive industrial step in the pulp and paper manufacturing process (Perng and Wang, 2016).

TABLE 5 ClO_2, H_2S_4O and NaOH consumption and final brightness (F.B.) of D(EPO)DED sequence with different chemical reagents dosages to reach 89.5 ± 0.5% ISO.

Sequence	ClO_2 (kg·odt^{-1})	H_2SO_4 (kg·odt^{-1})	NaOH (kg·odt^{-1})	F.B.
Control	26.1 a	0 a	18 a	89.57 a
5 kg·odt^{-1}	26.3 a	0 a	18 a	89.36 a
10 kg·odt^{-1}	27 b	3.4 b	21.3 b	89.3 a
15 kg·odt^{-1}	28 b	4.2 c	21.5 b	89.0 a

Means followed by the same letter per column do not differ by the Tukey test at 5% probability.

Alkaline filtrate recirculation and cellulosic pulp quality

The tensile index of the control sequence was 23% higher than that with 10 kg/odt of carryover in the unrefined pulp. Increasing the chlorine dioxide consumption during bleaching in the sequence with carryover may cause greater fiber degradation (Feria et al., 2013; Ribeiro et al., 2014). This was not recorded in this research, because the pulp tear index was 16% higher and viscosity was similar in the sequence with 10 kg·odt^{-1} of carryover (Table 6). The paper resistance from softwoods depends on the tracheid quality, the tear index is better related to tracheid strength and tensile index with the number of connections between these structures (Biermann, 1996; Gharehkhaniaet al., 2015). Therefore, changes during bleaching with carryover addition might affect the interactions between them.

The use of carryover had damaged the tracheids connections, thereby reducing the tensile index. The refinement of the cellulosic pulp can reduce this problem by increasing the connection numbers between tracheids (Biermann, 1996; Bhardwajet al., 2007; Gharehkhania et al., 2015). Thus, in all evaluated parameters, the regression models generated for the two pulp types (with and without carryover) can be adjusted to one model (Figure 2).

TABLE 6 Tensile index, tear index and viscosity (Visc.) in unrefined pulps without the filtrate and with 10 kg·odt^{-1} of carryover.

Treatment	Tensile index (N·m·g^{-1})	Tear index (mN·m^{-2}·g^{-1})	Viscosity (mPa·s)
Control	64.61 a	21.26 a	23.2 a
10 kg·tas^{-1}	52.55 b	24.65 b	23.4 a

Means followed by the same letter per column do not differ by the Tukey test at 5% probability.

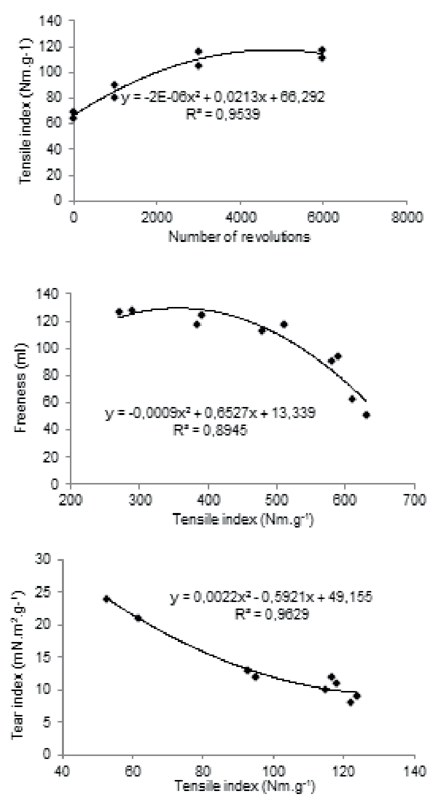

FIGURE 2 Tensile index, Drainability (freeness) and tear index behavior in refined bleached pulps and in the control with 10 kg·odt^{-1} of carryovers.

Refining increased the pulp tensile index, the parameter that depends mainly on the connection numbers between the tracheids. This also occurred with drainage, because the greater connection numbers hamper the water passing through the pulp (Gharehkhaniaet al., 2015). This behavior was reported for cellulose pulp from grasses (Andrade e Colodette, 2016), softwoods (Chen et al., 2016; Chen et al., 2017) and hardwoods (Zanuncio et al., 2016).

Refinement reduced the tear index, a parameter mainly dependent on the tracheid quality, which are degraded during refinement. The softwood tear index was reduced, even at low levels of refinement, as reported for *Pinus massonianae* and *China Fir* (Chen et al., 2016). In hardwoods, the tear index increased at the initial refinement levels due to the increase of connections and decreased at more intense refinement levels due to fiber degradation (Gharehkhania et al., 2015), as reported for *Corymbia citriodora* (Severo et al., 2013) and *Eucalyptus grandis* × *Eucalyptus urophylla* (Zanuncio et al., 2016).

Inorganic compound accumulation in the system

Calcium, magnesium and sodium concentrations increased after the D stage with 15 kg·odt^{-1} of the filtrate (EPO) recirculation (Table 7). The increase of calcium and sodium ions concentrations generated precipitated salts, causing incrustation and increasing the carbonate, oxalate and sulfate concentrations (Frigieri et al., 2016).

The carbonate, chlorate, chloride and sulfate anion concentrations in filtrate after the D stage increased with the addition of the alkaline filtrate to the pre-O_2 pulp at 134; 5.7 and 15 and 267%, respectively. The higher chloride concentration may favor organochlorine (AOX) formation in the filtrates, impairing the treatability of the effluent and the aquatic biota, when released into rivers (Huber et al., 2014).

TABLE 7 Concentration (Conc. kg·odt^{-1}) of ions and salts in the pulp bleaching effluent after D stage.

Stage	Compound	Filtered	Conc. (kg·odt^{-1})	Compound	Filtered	Conc. (kg·odt^{-1})
D	Mg	Control	0.454	Sulfate	Control	0.589
		15 kg·odt^{-1}	0.522		15 kg·odt^{-1}	2.163
	K	Control	0.058	Calcium carbonate	Control	0.087
		15 kg·odt^{-1}	0.064		15 kg·odt^{-1}	0.205
	Ca	Control	2.34	Total Oxalate	Control	0.557
		15 kg·odt^{-1}	3.01		15 kg·odt^{-1}	0.570
	Na	Control	3.46	Solúvel Oxalate	Control	0.186
		15 kg·odt^{-1}	6.46		15 kg·odt^{-1}	0.389
	Chlorate	Control	3.872	Chloride	Control	6.59
		15 kg·odt^{-1}	4.10		15 kg·odt^{-1}	7.53

The ions and salt concentrations varied depending on carryover use after (EPO) stage (Table 8). Calcium and magnesium content was lower and sodium content increased 21% in the filtrate from the (EPO) stage due to the increase in NaOH consumption by 19% in the extraction alkaline stage (Frigieri et al., 2016). Partial circuit closure of the alkaline filtrate did not change the calcium or carbonate concentrations in the filtrate (EPO). Incrustation due to calcium carbonate causes more problems during the bleaching stages (Hermosilla et al., 2015). The potassium concentration was lower in the (EPO) filtrate, but that of the chloride increased by 2% with the addition of 15 kg·odt^{-1} of carryover. The sulfate content in the (EPO) filtrate increased 10 times with the recirculation of 15 kg·odt^{-1} of carryover, favoring the recovery cycle (Huber et al., 2014). The oxalate content of the (EPO) filtrate was similar and that of the formate increased.

TABLE 8 Ion and salt concentration (Conc.) in the bleaching effluent after (EPO) stage.

Stage	Compound	Filtered	Conc. (kg·odt^{-1})	Compound	Filtered	Conc. (kg·odt^{-1})
(EPO)	Mg	Control	0.054	Sulfate	Control	0.589
		15 kg·odt^{-1}	0.035		15 kg·odt^{-1}	2.52
	K	Control	0.150	Carbonate	Control	0.087
		15 kg·odt^{-1}	0.132		15 kg·odt^{-1}	0.018
	Ca	Control	0.520	Total oxalate	Control	0.557
		15 kg·odt^{-1}	0.520		15 kg·odt^{-1}	0.162
	Na	Control	2.47	Chloride	Control	1.95
		15 kg·odt^{-1}	2.94		15 kg·odt^{-1}	1.99
	Acetate	Control	0.628	Chlorate	Control	1.05
		15 kg·odt^{-1}	0.569		15 kg·odt^{-1}	1.029

CONCLUSIONS

The alkaline filtrate concentration in the pre-O_2 was found to hamper bleaching and increasing the ClO_2 consumption by 0.08 kg·odt^{-1} per unit of carryover. In addition, the filtrate carryover (EPO) increased the sulfuric acid and NaOH consumption for pH adjustment of the stages. The tensile index decreased and the tear index increased with filtrate use in the unrefined pulps.

However, the physical and mechanical pulp properties were similar after refinement. The calcium, sodium, sulfate and chloride contents were higher after "D" stage with the recirculation of 15 kg·odt^{-1} of the alkali filtrate and the sodium and sulfate contents were higher in the filtrate (EPO). Recirculation of the alkaline reduce the effluent generation and the usage of water in bleaching.

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