Environmental impact

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Partial circuit closure of filtrate in an ECF bleaching plant

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Abstract: The bleaching sector of the wood pulp industry is its largest effluent generator. The aim of this study was to reuse the bleaching filtrate in order to reduce water consumption. The experiment was conducted by simulating the $D_0(EP)D_1$ bleaching sequence and recirculating different amounts of filtrate from the oxidation stage to control the pulp consistency of the delignification stage (pre-O). Physical, mechanical, chemical and optical properties of the pulp were studied. The accumulation of the non-process elements (NPE) and their effects were evaluated with the Aspen-Plus® computer simulator. The results of the computational modeling were satisfactory. The recirculation of filtrates increased the saturation index of the system by 19 %, but remained at sub-saturation levels. The pulp viscosity and elongation remained statistically stable. Recirculation of up to 50 % of the filtrate did not produce differences in pulp brightness; however, there were slight losses in the pulp resistances. In order to maintain 84 % ISO brightness, there was a higher consumption

of the bleaching reagents. Up to 50 % of recirculation of the filtrates was accomplished without jeopardizing the system and the pulp quality and resulted in a savings of $55 \text{ m}^3 \text{ h}^{-1}$ of water – 7 % of the consumption of the entire mill.

Keywords: bleaching filtrates; consistency control; non-process elements accumulation; oxidation stage; water recirculation.

Introduction

Competition and mitigation of environmental impacts in the pulp and paper industry have led to more efficient production processes (Mahmood and Elliott 2006, Kamali et al. 2016). These processes include genetic improvement of plants for wood production, reduction of resource uses and production of materials with higher quality (Hung et al. 2015, Frigieri et al. 2015, Chen et al. 2016).

The production of wood pulp consumes a high volume of water and generates effluents with a high organic load, which can overload the effluent treatment plant, increase pulp production costs and create an environmental liability (Hermosilla et al. 2015). As a result, many mills are looking for solutions to reduce their environmental impacts, such as water conservation and effluents with lower organic loads.

Bleaching is the process with the highest water consumption and effluent generation in kraft pulp mills and, therefore, with the highest demand for water use reduction. Closing the water circuit is an option for accomplishing this goal; however, it should be carried out in a cautious way to avoid the accumulation of non-process elements (NPE) that can generate incrustation and corrosion in pipes and equipment (Amaral et al. 2012, 2014), besides reducing the quality of the pulp (Santos et al. 2015).

Water circuit closure should be evaluated to avoid equipment damage and pulp quality reduction. Technical feasibility can be assessed using computational programs that efficiently simulate the mass balance of

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industrial processes from input parameters that represent the experimental data (Renon and Prausnitz 1968).

The Aspen-Plus® software, developed by the Massachusetts Institute of Technology (MIT) and the US Department of Energy, accurately simulates and models the behavior of the chemical reactions of various industrial sectors (Magnusson 2005). The concentration of the ions in each stage can be calculated with the Aspen-Plus® software from the Non-Random Two-Liquid (NRTL) thermodynamic model, using a Redlich-Kwong state equation as a basis for calculation applicable to liquid-liquid equilibrium in non-ideal systems (Renon and Prausnitz 1968).

Closed cycle systems for manufacture of wood bleached chemical pulp are systems that use processes wherein water and other chemicals are recycled and reused and thus waste for disposal is minimized. The first attempt to obtain a zero-effluent mill was proposed by Rapson in 1967 and adopted by Great Lakes Forest Products in 1974 in a pulping process developed by ERCO Envirotech in Canada. This mill was operated from 1977 to 1985 aiming the complete elimination of bleaching effluent by recycling it to black liquor recovery cycle. Unfortunately, several problems arose during the operation mainly due to the accumulation of the non-process elements causing deposits and corrosion in several equipment. Also, an increase in the bleaching chemicals was observed (Reeve and Silva 1997).

Since the first attempt, several projects for recycling the bleach plant effluents have been proposed based especially in the previous treatment of the effluents and on new bleaching sequences but most of them faced economical or technical burdens (Gleadow et al. 1997, Ulmgren 1997). Costa et al. (2006) recirculated the filtrate from the acid stage and found an increase in the chemicals use as well as a reduction on the brightness (from 90 to 88.5 % ISO). Moreira et al. (2011) reduced 20 % the water consumption of a kraft bleaching plant while reusing the filtrates, with no endangering on kappa number, brightness and viscosity. It is clear that the feasibility of such recycling systems is to protect the environment without jeopardizing processing cost or value of saleable products.

The search for the new opportunities to minimize water consumption in kraft bleached pulp mills without risking the pulp quality or the process equipment lifespan led to the objective of this research. The objective was to evaluate the technical feasibility of using the filtrate of the first stage of bleaching (D_0) to control the consistency at the oxygen pre-delignification stage (Pre-O) replacing the hot water from the heat exchangers.

Materials and methods

The study was developed in bench-scale tests at the Pulp and Paper Laboratory of the “Universidade Federal de Viçosa” (Viçosa, Minas Gerais state, Brazil), and the computational analysis was carried out using the Aspen-Plus® software.

The experiments simulated a bleaching sequence of $D_0(EP)D_1$, replacing hot water from the heat exchangers for the control of the pulp consistency of the delignification Pre-O stage (Figure 1.A) with filtrate from the D_0 stage (Figure 1.B). A bleached pulp was produced from a mixture of 70 % eucalyptus and 30 % pine wood.

The delignification system consists of two distinct liquid streams (Line 1 and Line 2). Line 1 has a washer filter as the last component of the washing and Line 2 has a washer press. Both use hot water (from heat exchangers) to control consistency in the re-pulper and send the pulp to ECF bleaching process. In Line 1 (relative to the washer filter) the pulp consistency is raised from 1–2.5 % to 18 %. In the repulper, the pulp is diluted with hot water for about 10 %. In Line 2 (relative to the washer press), the consistency is increased from 4.5 % to 27 %. In the repulper, the pulp is diluted with hot water for about 10 %. After the convergence of the lines the pulp is forwarded to the first stage (D_0) of ECF bleaching. In the detailed process, the control of the pulp consistency consumes $110 \text{ m}^3 \text{ h}^{-1}$ of hot water in the current situation, which is equivalent to 13 % of the water consumption of the entire mill.

The main characteristics of the effluent streams are showed in Table 1.

Laboratory simulation

A variety of proportions of replacement (0 %, 25 %, 50 % and 100 %) of the volume of hot water with filtrate from the D_0 stage was simulated in the laboratory.

Four samples of pulp were bleached to a brightness of 84 % ISO using the $D_0(EP)D_1$ bleaching sequence. The conditions for the bleaching sequence were standardized (Table 2).

The load of chlorine dioxide and hydrogen peroxide remained constant while the dosage of sulfuric acid or sodium hydroxide (pH control) varied due to the different volumes of filtrate added per sample.

Bleaching

The bleaching stages were carried out in duplicate and, after each stage; the pulp was washed with the equivalent of 9 m^3 of distilled water per ton of dry pulp. After each stage,

Figure 1: Laboratory simulation of bleaching pulp (A) using hot water as control of the consistency of oxygen-delignified pulp; (B) using filtrate of the D₀ stage. as control of the consistency of oxygen delignified pulp. (C: control of consistency system; W: washing system; WTP: wastewater treatment plant).

Table 1: Water and effluents characterization.

	D ₀ filtrate	EP filtrate	D ₁ filtrate	Condensate	Fresh water	White water	Hot water
pH	3.59	9.52	4.06	9.44	7.0	7.3	7.3
Conductivity (μS cm ⁻¹)	1281	2330	1324	231	18	292	84
Color (CU)	864	950	580	395	< 1	280	17.6
Turbidity (NTU)	16.7	32	13.8	10.0	< 1	2.26	0.30
COD (mg L ⁻¹)	1062	1641	708	1062	ND	132	8.5
Total Solids (%)	0.181	0.282	0.132	0.023	ND	0.160	0.003
Fe (mg L ⁻¹)	0.135	0.169	0.032	0.012	ND	ND	ND
Cu (mg L ⁻¹)	0.006	0.007	0.003	ND	ND	ND	ND
Cl (mg L ⁻¹)	ND	3.30	2.79	5.84	2.67	30	2.04
K (mg L ⁻¹)	10.3	15.4	8.79	3.59	1.70	2.59	1.81
Si (mg L ⁻¹)	5.0	22.5	19.5	6.0	6.0	8.5	5.0
Al (mg L ⁻¹)	ND	0.001	0.008	0.002	0.061	0.025	0.034
Mg (mg L ⁻¹)	13.5	20.6	9.73	0.25	1.86	2.51	2.20
Ca (mg L ⁻¹)	37	16	20	1.36	6.63	6.38	7.18
Mn (mg L ⁻¹)	0.70	0.51	0.24	ND	ND	ND	ND

ND: not detected

5 g of pulp and residual filtrate samples were collected for analysis. Sulfuric acid and sodium hydroxide were used for pH control in each stage.

Oven dry brown stock (230 g) was bleached with chlorine dioxide in the D₀ stage; in the EP stage, 225 g of pulp

from the previous stage were bleached using hydrogen peroxide and sodium hydroxide; and in the D₁ stage, 220 g of pulp from the (EP) stage were bleached with chlorine dioxide. In all bleaching stages, the pulp was washed at room temperature.

Figure 2: Flowchart of the D_0 stage of the bleaching plant used in Aspen-Plus® software studied with two tanks (B2 and B7), two pumps (B5 and B9), two dynamic mixers (B3 and B4), two flow separators (B1 and B8), an atmospheric reactor (B6) and 14 flow streams with characteristics of the flowchart components defined.

Table 2: Conditions and parameters for the laboratory simulation of the bleaching sequence $D_0(EP)D_1$.

Parameters	Bleaching stage		
	D_0	EP	D_1
Consistency (%)	10	10.8	10
Time (min)	90	45	90
Temperature (°C)	80	90	75
Final pH	3	11	5
$MgSO_4$ ($kg\ t^{-1}$)	–	1.4	–
Pressure (atm)	1	2.71–3.1	1
H_2O_2 ($kg\ t^{-1}$)	–	4.5	–
ClO_2 ($kg\ t^{-1}$)	9–10	–	2–3
O_2 ($kg\ t^{-1}$)	–	3.5	–

Analytical methodology

Water, filtrates and effluent analyses followed the American Public Health Association procedures prescribed in the 20th edition of the Standard Methods for the Examination of Water and Wastewater (APHA 1998).

The physical, chemical and optical properties of the pulp were determined after bleaching according to the T248 sp-08 (refining in PFI mill), T205 sp-06 (sheet formation for physical tests), T452 om-08 (Schopper Riegler and brightness), T1214 sp-07 (opacity), T536 om-07 (air passage resistance), T414 om-04 (tear resistance), and T494 om-06 (elongation and tensile index) procedures of the Technical Association of the Pulp and Paper Industry (TAPPI 1998), and statistical analyses were carried out using the Tukey test at 5 % of significance to evaluate the differences between the results.

Computational modeling

The Aspen-Plus® software was used to evaluate the feasibility of replacing hot water with filtrate from the D_0 bleaching stage for the pulp consistency control system of the last pre-delignification reactor with O_2 (Pre-O). The flowchart of the stages and equipment used during the simulation process consisted of two tanks (B2 and B7), two pumps (B5 and B9), two dynamic mixers (B3 and B4), two flow separators (B1 and B8), one atmospheric reactor (B6) and 14 flow streams with the characteristics of the flowchart components defined (Figure 2). According to the flowchart, the flow separator responsible for the system recirculation (B8) had its characteristics omitted in order to be calculated by the software. This equipment is crucial for the simulation, since it is responsible to determine the percentage of filtrate that could be recirculated.

The accumulation of non-process elements (NPE) – Fe, Cu, Cl, K, Si, Al, Mg, Ca and Mn – throughout the water-circuit closure was simulated from input variables inserted into the Aspen-Plus® software (Table 3). The components ClO_2 , steam and metals content, which are part of the process, were inputted from the Aspen-Plus® database, and the more complex components, such as residual lignin, were inserted into the modeling according to Sixta (2006).

The saturation index (SI) of the D_0 stage filtrate (control variable) was calculated from the Kps values (Allaga et al. 1992). The behavior of the concentrations of each ion was obtained from the Aspen-Plus® simulation.

Table 3: Convergence parameters for the characteristics of the components used in the simulation flowchart: equipment, pressure, temperature, equilibrium phases, maximum number of interactions, tolerable error and thermodynamic model.

Equipment	Pressure	Temperature	Equilibrium phases	Maximum number of interactions	Tolerable error	Thermodynamic model
Tanks	1 atm	–	Liquid	30	0.0001	NRTL
Pumps	10 bar	–	Liquid	30	0.0001	NRTL
Mixers	6 bar	–	Liquid-steam	30	0.0001	Wilson
Separator B1	10 bar	70 °C	Liquid	30	0.0001	NRTL
Atmospheric reactor	1 atm	80 °C	Liquid	30	0.0001	NRTL

Table 4: Results of the laboratory experiment simulating the D₀(EP)D₁ sequence at different recirculation levels of the filtrate D₀ as control of the pulp consistency in pre-O: final pH, brightness, lightness *L, redness/greenness a* and yellowness/blueness b*.

Stage	Replacement (%)	Final pH	Brightness (%ISO)	*L	a*	b*
D ₀	0	3.6	62.0 a	90.39 a	1.84 a	13.83 a
	25	3.4	61.5 b	90.09 b	1.98 b	13.76 a
	50	3.6	58.1 c	88.41 c	2.34 c	14.04 b
	100	3.5	57.4 d	88.14 d	2.39 c	14.19 c
EP	0	11.2	77.7 a	95.31 a	0.03 a	8.79 a
	25	11.3	77.7 a	95.30 a	0.01 a	8.76 a
	50	11.2	76.4 b	94.90 b	0.19 b	9.09 b
	100	11.3	76.0 c	94.80 b	0.18 b	9.25 c
D ₁	0	4.6	85.7 a	97.44 a	−0.34 a	5.45 a
	25	4.6	85.7 a	97.39 a	−0.32 a	5.55 a
	50	4.6	85.5 a	97.22 a	−0.28 b	5.66 b
	100	4.6	85.2 a	97.19 a	−0.28 b	5.69 b

Means followed by the same letter do not differ by the Tukey Test at 5 % of significance

The concentrations of the ions were calculated with the non-random two-liquid thermodynamic model (NRTL) using the Redlich-Kwong state equation.

The NRTL model was compared to the existing industrial bleaching plant by the assembly of the process in the computational simulation until its validation, i. e., when the industrial process and the computer simulation program were similar.

Results

Laboratory simulation

The D₀(EP)D₁ sequence at four filtrate recirculation levels (0 %, 25 %, 50 % and 100 %) reduced the brightness and *L (lightness), and increased *a (redness/greenness) and *b (yellowness/blueness) of the pulp (Table 4).

The difference in brightness of the various recirculation ratios was more remarkable in the initial stages of D₀ and EP according to the Tukey Test at 5 % (Figure 3). The

D₀ stage showed brightness and *L reduced significantly in all proportions of recirculation. The EP stage had the brightness and *L changed significantly for the recirculation of 50 % and 100 % of the filtrate. The final D₁ stage did not undergo statistically significant changes in any of the proportions of recirculated filtrate.

The elongation values and pulp viscosity were not altered in any proportion of the filtrate used (Table 5).

The addition of the filtrate decreased the tensile index, the tensile energy absorption (TEA) and the tear index when adding 25 % of the filtrate; however, upon increasing this value, the results remained stable.

Computer simulation

The results of the Aspen-Plus® model for the NPE accumulation in the D₀ stage of the full-scale bleaching plant were similar to those of the filtrate in the industrial plant studied. The concentration of NPE for the actual filtrate was 0.135, 10.3, 13.5, 37 and 0.70 mg L^{−1}, respectively, for Fe²⁺, K⁺, Mg²⁺, Ca²⁺ and Mn²⁺, while the Aspen-Plus® software

Figure 3: Brightness of each stage (D₀, EP and D₁) from the bleaching experiment using the recirculation of D₀ filtrate.

Table 5: Results of the physical tests performed on the pulp after bleaching experiment with recirculation of D₀ filtrate: tensile index, elongation, tensile energy absorption (TEA), tear index and viscosity.

Replacement (%)	Tensile index (N m g ⁻¹)	Elongation (%)	TEA (J m ⁻²)	Tear index (mN m ² g ⁻¹)	Viscosity (cm ³ g ⁻¹)
0	19.51 a	1.68 a	24.33 a	4.72 a	713 a
25	18.72 b	1.69 a	23.21 ab	4.49 b	714 a
50	18.32 b	1.61 a	21.80 c	4.40 b	693 a
100	18.14 b	1.63 a	22.17 bc	4.45 b	703 a

Means followed by the same letter do not differ by the Tukey Test at 5 % of significance

obtained results of 0.145, 11.1, 14.5, 40 and 0.76 mg L⁻¹, respectively, for the same components.

The concentrations of Cu²⁺ and Al³⁺ remained at 0.006 and 0.0 mg L⁻¹, respectively, for both the real filtrate and the software simulation. The variation between the actual and proposed values of the Aspen-Plus® software was lower for Fe²⁺ and Mg²⁺ ions, 7.4 % for both, and higher for the Mn²⁺ ion, 8.5 %. The Al³⁺ ion was not detected in the filtrate.

The modeling used served, at the end of the validation, as the basis for the simulation of the water closure circuit process studied – since the model adhere 93 % in relation to the real filtrate. The concentration of NPE simulated by the Aspen-Plus® software increased with recirculation of the filtrate in the D₀ stage of the ECF bleaching in proportions of 0, 25, 50, 75 and 100 % instead of the hot water from the heat exchanges (Figure 4). The increase of Cu²⁺ was the most significant (66.7 %), and the other ions increased between 46 and 48 % with the replacement of 100 % of the hot water with filtrate.

The saturation index (SI) increased linearly from 0.136 mg L⁻¹ without recycling the filtrate to 0.162 mg L⁻¹ with 100 % of recycling, an increment equivalent to 19.32 %. However, the SI remained below 1, confirming no incrustation even with total recirculation of filtrate.

Discussion

Laboratory simulation

The increase of the values of the matrices *a and *b with the use of the filtrate is explained by the return of the chromophore groups (Andrady et al. 1999), giving the red and yellow color to the pulp.

The reduction of differences in pulp quality between each recirculation level is due to the ability of the bleaching process to absorb chemicals and organic loads throughout each stage (Sixta 2006).

The stability of the elongation is justified by its relation with the specific surface of the fibers and tracheids and, since the viscosity was not altered, the degree of polymerization of the cellulosic chains, fibers and tracheids of the pulp was not altered either (Carneiro et al. 1995), maintaining the fiber's ability to undergo deformations. The viscosity remained stable, indicating that, in isolation, this parameter is not indicative of pulp strength, since the other physical properties varied, similar to that reported for eucalyptus pulps studied by Carneiro et al. (1995).

The reduction of the tensile, tear and TEA indexes occurred due to the decrease in the number of interfiber bonds, which were obstructed by the presence of the

Figure 4: Increase of non-process elements concentration with the replacement of hot water by D_0 filtrate in different proportions (0, 25, 50, 75 and 100 %).

non-process elements that reduce the quality of the pulp, which could make it difficult to insert in the market, besides degrading the machinery. The reduction of these values can be corrected in subsequent processes, such as refining (Buzala et al. 2016), which increase the number of interfiber bonds and, consequently, the resistance of the pulp (Biermann 1997, Bhardwaj et al. 2007).

Computational modeling

The values presented in Aspen-Plus® show that the models generated from hypothetical scenarios can represent the proposed scenario, which qualifies the program as adequate for predicting the concentration of NPE in the D_0 stage filtrate. The software modulates and accurately describes the fouling process by identifying the primary conditions that need to be met for this process, but is not able to predict where and when this will occur (Rudie and Hart 2005). The NRTL thermodynamic model is applicable to partial miscibility systems, i. e., liquid-liquid equilibrium, and provides a good representation of those systems which are considered to be non-ideal (Renon and Prausnitz 1968).

The computational simulation based on this model shows that it is possible to replace 100 % of the volume of hot water with D_0 -stage filtrate. However, the constant reuse of this filtrate can increase the saturation index, causing problems such as fouling and clogging of the pipes. The saturation index of less than 1 indicates that, when hot water is replaced by filtrate from the D_0 stage, it is possible that the salts dissolve in the liquid, therefore not allowing incrustations to occur (Allaga et al. 1992). If corrosion occurs it may impact the properties of the pulp.

Conclusions

- The model developed on Aspen-Plus® was similar to the industrial process;
- In the current situation (with no recirculation of the filtrates), there are no probability of incrustation on the system;
- The recirculation of the filtrate gradually affects the saturation indexes;
- The increase of the recirculation of the filtrates in different proportions increased the incrustation and deposition potential;
- Total replacement of hot water by filtrate (100 % of filtrate recirculation) was not able to generate incrustation in the system (incrustation index kept smaller than 1);
- More than 50 % of water replacement affects the brightness and the *L, *a and *b matrices;
- Up to 50 % of water replacement did not affect the optical and physical characteristics of the bleached pulp;
- There was no change of pulp viscosity after 100 % filtrate recirculation;
- The addition of D_0 filtrate affected the pulp resistance, but no difference among the different proportions (25 %, 50 % and 100 %) was observed.

It was possible to replace hot water by D_0 filtrate in the consistency control of the pulp without jeopardizing the process – no raise in the scaling potential and no loss in the optical, physical properties of the bleached pulp. However, the replacement of the water by filtrate must be conducted gradually until 50 %, with small losses of pulp re-

sistance. The results shows an economy of $55 \text{ m}^3 \text{ h}^{-1}$ of water consumption, equivalent to a reduction of 7 % of water consumption of the entire mill. The same amount of previous effluent generation is reduced, unburden the Effluent Treatment Plan (ETP) as well. It is also important to mention an increase in the maintenance costs and design of the heat exchanger may occur.

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