

Membrane treatment of the bleaching plant (EPO) filtrate of a kraft pulp mill

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ABSTRACT

The objective of this study was to evaluate the use of membrane technology to treat oxygen and peroxide-reinforced extraction stage (EPO) filtrate from a kraft pulp mill bleach plant. Three different types of tubular membranes were tested in a pilot plant: (i) tight ultrafiltration (UF); (ii) open UF followed by nanofiltration (UF + NF); and (iii) nanofiltration (NF). According to the separation performance, considering the chemical oxygen demand (COD) and colour removal, permeate flux, operational simplicity and cost, the results indicated that the best option for treatment of (EPO) filtrates was the tight UF membrane. This membrane obtained a COD removal of 79% with a colour reduction of 86%. The effect of (EPO) filtrate UF treatment on the mill effluent treatment plant was evaluated. Compared with the actual mill effluent, the results indicated that if the UF permeate was recycled in the bleaching area, the COD reduction efficiency increased by 7%, the final effluent colour decreased by 8%, the biological sludge production decreased by 18%, and the energy consumption decreased by 40%. In the tertiary treatment plant, the coagulant dosage decreased by 40%, and the tertiary sludge production decreased by 46%.

Key words | (EPO) effluent, membrane treatment, pulp mill effluent, ultrafiltration

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INTRODUCTION

Because of the continuous increase in environmental restrictions and societal awareness, the pulp and paper industry is looking for options to reduce water consumption and enhance effluent quality (Bajpai 2012). In a kraft pulp process, the removal of the residual lignin from the pulp suspension by means of chemical reactions is carried out in a bleaching plant. The bleaching process is composed of several stages, in which the pulp is chemically treated and then washed to remove spent bleaching chemicals and dissolved pulp components (Sixta 2006). After the first ClO_2 stage (D), an extraction stage is employed to dissolve the compounds soluble in alkali. In a modern bleaching sequence, the extraction stage is reinforced with oxygen and hydrogen peroxide (EPO), increasing the lignin removal and brightness (Brogdon 2008).

Normally each bleaching stage is composed of a reactor followed by a pulp washer. The bleaching plant filtrates from the washers constitute the major source of effluent sent to the effluent treatment plant (ETP). The alkaline (EPO) filtrate contributes significantly to the final effluent's high organic load and colour (Puro *et al.* 2010).

The treatment of specific in-plant streams seems to be an attractive technical and economical approach because it allows for the use of advanced technologies such as membrane filtration (Kasher 2009). Membrane-based processes, such as ultrafiltration (UF) and nanofiltration (NF) are very important alternatives among the possible effluent treatments.

Several studies have reported the use of a membrane to treat alkaline bleaching plant effluent (Mänttari *et al.* 2002; Nordin & Jönsson 2010; Amaral *et al.* 2013), but at this time, industrial applications for a kraft pulp mill have not been reported. The development of a new membrane material with better resistance to extreme conditions (pH and temperature) and with higher fluxes is useful for industrial applications (Sharma & Chellam 2005). The objectives of the present study are to: (i) compare three types of membranes to treat the alkaline extraction (EPO) filtrate of a kraft pulp mill using pilot plants and to select the best option according to the chemical oxygen demand (COD), colour removal and permeate flux; (ii) determine the best operation conditions of the selected membrane; and (iii) study the effect of (EPO) UF on the ETP.

MATERIAL AND METHODS

The present study was divided into four phases:

- (i) Phase 1: Membrane configuration selection.
- (ii) Phase 2: Determining the best operation conditions for the selected configuration.
- (iii) Phase 3: Long-term operation of the filtration system.
- (iv) Phase 4: Determining the effects on the ETP.

(EPO) effluent

The characteristics of the (EPO) filtrate for the kraft pulp mill's softwood and hardwood campaigns are presented in Table 1. The COD, colour, SO₄, Al, K, Ca and Na were determined by using standard methods for the examination of water and wastewater (Eaton *et al.* 2005). The filtrates were obtained directly from the pulp mill during normal operation. Na, Ca, K, Al, SO₄ values are the mean value of two samples, and COD and colour values are the mean value of ten samples. The temperature was 70 °C and the pH was 10 in both campaigns.

Membranes

The membrane material and average pore sizes are presented in Table 2. All membranes used in this study were tubular and manufactured by PCI.

Pilot plants

The membrane configuration selection and the determination of the optimal conditions were carried out using a batch feed one-stage pilot plant. The pilot plant was equipped with a PCI B1 series flow module, with a single flow through the membrane. The membrane was 1.2 m long and 1.3 cm in diameter. The feed pump operated at a pressure of 4–26 bar. In all tests, the flow rate was maintained at approximately 19 L/min, which gives a tangential velocity of 2.6 m/s.

The operation transmembrane pressure (TMP) was adjusted manually with the retentate outlet valve. The permeate volume and flows were measured with volumetric containers and timers.

The long-term operation was carried out using a continuous feeding two-stage pilot plant. Each stage had three parallel membrane modules. The six type B1 PCI membranes were 3.6 m long and 1.3 cm in diameter.

In both cases, the pilot plant was fed with (EPO) filtrate from the bleaching area of the pulp mill.

Membrane configuration

The objective of this phase was to evaluate the different configurations of the filtration systems, including: (i) ultrafiltration (UF1 and UF2 membranes); (ii) UF followed by nanofiltration (UF3 + NF1 membranes); and (iii) nanofiltration (NF1 membrane). The TMP tested for each UF membrane was 6 and 21 bars: for the NF 21 bars and for the combination of UF followed by NF 2 and 21 bars,

Table 1 | Characterization of the (EPO) filtrate for the softwood and hardwood campaigns

(EPO) filtrate	Flow (L/s)	Na (mg/L)	Ca (mg/L)	K (mg/L)	Al (mg/L)	SO ₄ (mg/L)	COD (mg/L)	Colour (UPT-Co)
Softwood	200	603	8.5	11.8	0.20	250	1890 ± 150	850 ± 120
Hardwood	200	625	2.6	7.3	0.26	n.d*	1600 ± 200	640 ± 110

*n.d: not determined.

Table 2 | Tested membranes

Membrane code	Commercial name	Type	Material	MWCO*
UF1	ESP04	Tight UF	Polyether sulfone	4,000 g/mol
UF2	XP197	Tight UF	Polyether sulfone	1,500 g/mol
UF3	FP200	Open UF	Polyvinylidene difluoride (PVDF)	200,000 g/mol
NF1	AFC30	NF	Polyamide film	350 g/mol

*Molecular weight cut-off.

respectively. The pH tested on the NF was 9 and 7 and in the UF the pH was kept equal to 10. The evaluation of the selectivity of each configuration was made according to three parameters for the effluent quality, including COD and colour. In total, 17 tests were performed by treating the (EPO) filtrate produced in the kraft pulp mill in the softwood campaign.

Optimal operation condition

In this phase, the optimal operation condition of the system, TMP and cross flow velocity (CFV) was established. The tested TMP varied from 1.7 to 6.5 bar and the CFV from 2.0 to 3.0 m/s. The treated (EPO) filtrate was obtained from a softwood campaign pulp mill.

Long-term operation

In this phase, the separation was carried out in a continuous feed two-stage pilot plant, and the objective was to confirm the best operating conditions for a long operation time. The pilot plant operated continuously for 3 months and performed 48 tests, each 20 hours long. Of these, 37 tests were made during the softwood campaign and 11 during the hardwood campaign.

Each test was carried out in two stages. A lower volumetric concentration factor (VCF) was used in the first, and the VCF was then increased in the second stage. The VCF of the first stage was kept at around 2–3, whilst the VCF of the second stage varied from 2 to 100. The TMP varied from 1.8 to 7.0 bar.

Effects on the ETP

The mill's effluent biological treatment plant is an activated sludge (extended aeration) plant. The hydraulic retention time of the reactor is approximately 19 hours.

Three scenarios were evaluated. Scenario 1: simulate the biological treatment of the current industrial effluent as a reference; Scenario 2: simulate the biological effluent treatment without the (EPO) filtrate but with the added UF permeate (Figure 1); and Scenario 3: simulate the biological effluent treatment without the (EPO) filtrate and without the UF permeate (the permeate from the UF was recycled).

The biological treatment was simulated using 2-litre sequencing batch reactors, one reactor for each scenario. The temperature and dissolved oxygen were maintained at 37 °C and 2 mg/L, respectively. The principle of the sequencing batch simulation was to incorporate all units of the

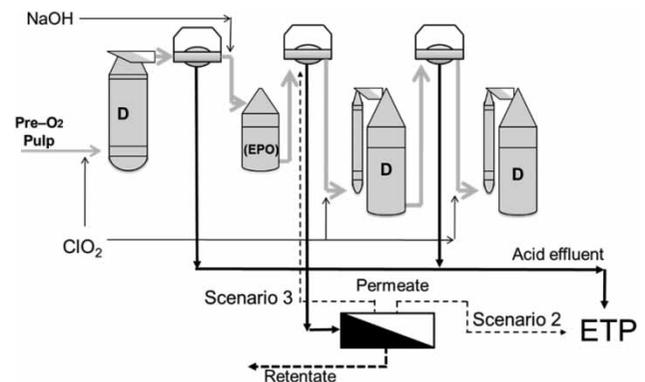


Figure 1 | Flowchart of the bleaching area in Scenario 2 and Scenario 3.

activated sludge process in a single reactor. Each cycle had a reaction time of 20 hours, a settling time of 2 hours and a decanting time of 2 hours.

One thousand milliliters of sludge (VSS = 3000 mg/L) and one thousand milliliters of effluent were added to each reactor. The COD reduction and the final colour were used as performance indicators.

The mill tertiary treatment was composed of a physical chemical unit, i.e., coagulation, flocculation and flotation. This process was simulated with jar test equipment to determine the optimal coagulant dosage ($Al_2(SO_4)_3$) for all scenarios.

The effluent used for these simulations was obtained from the biological treatments of the laboratory (sequencing batch reactors). In total, 25 tests were carried out for each scenario to determine the optimum dosage and the COD and colour removal efficiency. The jar test conditions were a 150 rpm mixture for 60 seconds followed by a 50 rpm mixture for 180 seconds.

Cleaning procedure

A chemical cleaning method to restore the performance of the membrane was carried out after each run. The pilot plant was stopped at the end of the run, and the content of the feed tank was drained and refilled with hot water to flush the pipelines. The cleaning solution (caustic detergent, chlorine and a dispersant) was added to the feed tank and circulated at 23 L/min for 40 minutes, at 70 °C and an inlet pressure of 5 bar.

RESULTS AND DISCUSSION

The characteristics of the effluent to be treated directly affect the selection of the membrane. The factors that most affect

membrane structure are the temperature and pH (Nilsson *et al.* 2008). Studies report that in the case of nanofiltration, most commercial membranes can operate at a maximum temperature of 40 °C. Anything above this temperature can present negative effects on the membrane structure. According to studies, the pore size may increase during operation with a high temperature (50 °C), directly affecting the selectivity of the membrane (Sharma & Chellam 2005). The (EPO) filtrate has a temperature of 70 °C, so for a nanofiltration membrane, it would be necessary to cool the temperature to 40 °C.

A high pH can produce a dissociation of the functional groups of the membrane and may cause the pores to swell, modifying the molecular weight cut-off of the nanofiltration membrane (Luo & Wan 2013).

When selecting the membrane, both the temperature and pH must be considered.

Membrane configuration

As expected, the best results in terms of permeate flux, COD and colour removal were obtained with ultrafiltration (UF3) followed by nanofiltration (NF1), i.e., 92% COD and 95% colour removal (Table 3). The results obtained in a single separation stage of nanofiltration showed a high removal of colour and COD, at 70 and 93%, respectively. Separation with an ultrafiltration membrane (UF1) achieved a 64% removal of COD and 87% removal of colour.

Although the UF2 membrane had an average pore diameter smaller than that of the UF1 membrane, it did not promote a higher removal of these parameters. This was probably because the pore structure of the UF2 membrane was affected by the characteristics of the effluent (pH and temperature). Studies show that the temperature and pH affect the geometry and size of a pore, thus modifying its selectivity (Sharma & Chellam 2005; Luo & Wan 2013). It is therefore necessary to neutralize the pH and decrease

the temperature, resulting in high costs and increased complexity of the operation (Al-Rawajfeh 2011).

To select the best option, the selectivity, cost and operation simplicity were considered. The nanofiltration membranes require a pre-treatment to reduce the temperature and neutralize the pH. The tight UF membrane (UF1) showed a lower selectivity, but it was considered sufficient for the mill requirements. Moreover, this configuration presented advantages over the nanofiltration options, such as a simpler operation, higher resistance to pH and temperature (would not require pre-treatment), and has a higher permeate flux, resulting in a lower investment cost.

According to the results of Phase 1, the selected configuration was the tight UF with the UF1 membrane. The other phases of this research were conducted using this configuration.

Optimal operation condition

In this phase, the effect of TMP on the permeate flux was evaluated. TMP had a positive effect on the permeate flux. However, above 10 bars, a decrease in the flux was observed (data not shown). Therefore, the maximum TMP considered was 8 bars. Nordin & Jönsson (2006), also found similar results for the UF treatment of bleaching effluent from a sulfite mill.

Table 4 shows that the best colour and COD removals were obtained under conditions of a high CFV (3.00 m/s) and a low TMP (1.7 bar). In these conditions the highest removals were achieved (65% of the COD and 93% of the color). However, the permeate flux was relatively low (70 L/m².h), probably due to the low TMP applied.

On the other hand, the highest flux (197 L/m².h) was obtained under conditions of high TMP and high CFV. In these conditions, the COD and colour reduction were 58 and 91%, respectively.

High CFV allows a smaller boundary layer thickness between the liquid and the membrane, because of the

Table 3 | Efficiency treatment of the (EPO) filtrate with different membrane configurations

Parameter	UF1		UF2		UF3 + NF1	NF1	
pH	11	11	11	11	9	9	7
TMP (bar)	6	21	6	21	2 and 21	21	21
COD reduction (%)	53.4	64.3	59.1	59.3	91.5	74.3	70.6
Colour reduction (%)	86.9	93.8	87.2	87.1	94.8	93.0	92.9
Average permeate flux (L/m ² .h)	220	220	113	170	340 and 200	130	150

TMP: Transmembrane pressure.

Table 4 | Colour and COD reduction with UF1 membrane separation, with different transmembrane pressures and cross flow velocities

CFV (m/s)	TMP (bar)	Colour	COD	Permeate flux (L/m ² ·h)
		Reduction (%)	Reduction (%)	
2.0	1.9	93	63	133
	4.7	88	54	149
	6.6	90	57	172
2.5	1.7	65	41	106
	4.4	89	58	189
	6.5	92	55	205
3.0	1.7	93	65	67
	4.4	88	54	148
	6.4	91	58	202

CFV: Cross flow velocity; TMP: Transmembrane pressure.

higher self-cleaning condition produced by the CFV. The thinner boundary layer avoids fouling and decreases the forces of resistance on the passage of particles. Studies have confirmed this phenomenon. Therefore, CFV is an important parameter in permeate flow optimization and in reducing fouling in microfiltration and UF (Choi *et al.* 2005).

The chosen option was based on the highest permeate flux, considering that in these conditions the COD and colour reduction were considered acceptable.

Long-term operation

In this phase, the operation of a two-stage pilot plant with longer periods of operation was evaluated for the treatment of (EPO) filtrates from the softwood and hardwood campaigns to confirm the separation performance of the UF1 membrane and the flux rates.

The results presented in Table 5 show the effect of the TMP on the different VCFs for each step. In general, an increase in TMP led to an increase in the permeate flux. These results were obtained during the softwood campaign.

It was observed that at a TMP of 8 bar, the permeate fluxes at a low final VCF (2) were higher than those at 7

Table 5 | Effect of TMP on the UF permeate flux in a two-stage pilot plant simulation

VCF 1	Permeate flux step 1 (L/m ² h)		VCF 2	Permeate flux step 2 (L/m ² h)	
	Pressure			Pressure	
	7 bar	8 bar		7 bar	8 bar
1.45	146	198	2	84	131
2.14	160	200	5	73	94
2.40	163	174	10	76	69

bar. However, at a VCF of 10, a lower TMP produced higher fluxes.

The results indicate that the permeate flow did not change significantly by changing the raw materials for the production of bleached pulp (Table 6). Thus, the design of the membrane separation plant must be based on the type of effluent with a higher organic load. According to the data obtained in this study and confirmed by the data in the literature, the effluent obtained in the production of pulp using softwood showed the highest organic load (Sixta 2006).

Effects on the effluent treatment plant

Figures 2 and 3 show the results of the COD removal and final colour of the treated effluent (reactor supernatant). To achieve the steady-state condition, 20 cycles were performed for each scenario. Data obtained before the steady-state condition were not considered in this study because they do not depend exclusively on the type of effluent to be treated and affect other variables such as the adaptation of microorganisms and environmental conditions (Metcalf & Eddy 2003). The last 10 cycles (steady-state operation) of each scenario were considered for discussion.

The results showed that there is an increase in the efficiency of COD removal in the hardwood and softwood campaigns in Scenarios 2 and 3 compared to Scenario 1 (reference). The largest increase in the efficiency reduction was achieved in Scenario 2, in which the resulting UF permeate was treated by biological treatment. This can be explained by the fact that the large organic molecules are retained by the membrane and the permeate had small size molecules that were biodegradable, i.e., there was an increase in the biodegradable organic matter ratio in the effluent. Mounteer *et al.* (2007), studied alkaline bleaching filtrate from a kraft pulp mill and found that the BOD₅/COD ratio of the high (>500 Da) molecular mass fraction was 0.32 while for the low (<500 Da) molecular mass fraction it was 0.52. This means that effluents with a larger

Table 6 | Behaviour of the UF permeate fluxes in the softwood and hardwood campaigns in a two-stage pilot plant simulation

Final VCF	Flux in step 1 (L/m ² ·h)		Flux in step 2 (L/m ² ·h)	
	Softwood	Hardwood	Softwood	Hardwood
2	189	202	105	119
5	187	151	80	86
10	166	165	68	72
100	164	164	64	60

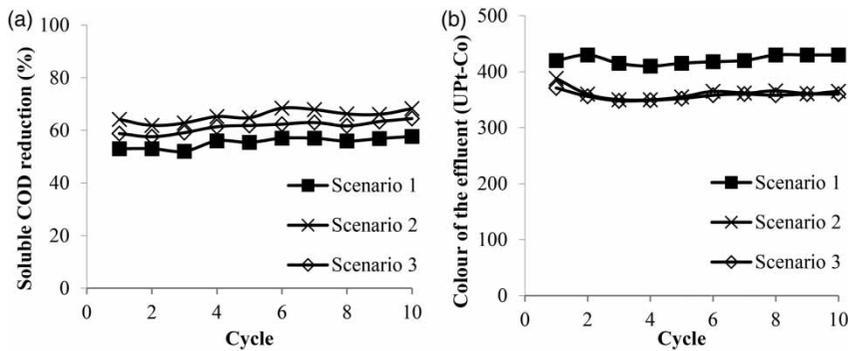


Figure 2 | (a) COD removal and (b) treated effluent final colour in the three scenarios in the softwood campaign.

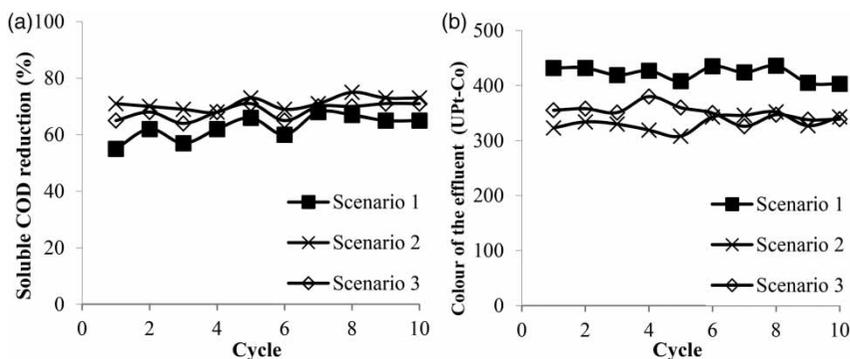


Figure 3 | (a) COD removal and (b) treated effluent final colour in the three scenarios in hardwood campaign.

quantity of low molecular mass compounds are more easily removed during effluent biological treatment.

The colour reduction in Scenarios 2 and 3 was higher than in the reference. It is known that biological treatment does not remove colour from effluent, which can only be controlled by physical-chemical treatments (Metcalf & Eddy 2003). The colour reduction in the final treated effluent was due to the UF of the (EPO) filtrate, which has an average colour of 850 and 650 UPt-Co (platinum-cobalt colour scale) in the softwood and hardwood campaigns, respectively.

The production of biological sludge decreased by approximately 20% in Scenario 3 for the softwood and hardwood campaigns. This was because in Scenario 3, the resulting UF permeate was recycled to the bleaching plant, decreasing the flow of the effluent and the COD load to be treated by biological treatment. Furthermore, the energy consumption related to aeration was 20% less in this scenario because of the decrease in microorganism production.

The simulation of the physical-chemical tertiary treatment was performed using a jar test for the prior biological treated effluent. The results for Scenarios 2 and 3 are shown in Table 7. They indicate that the UF of the (EPO) filtrate decreased the coagulant dosage (in both

campaigns) by 30 to 40% according to the lower COD content of the effluents with UF treatment.

Significant decreases of approximately 45 and 30% in Scenario 3 and Scenario 2, respectively, were observed in the generation of tertiary sludge. Again, this can be explained by the fact that, in these scenarios, the COD load was lower than in the reference scenario.

Table 7 presents a summary of all of the effects of UF membrane treatment on the ETP. Of these, the most attractive scenario is the recycling of the UF permeate to the bleaching sector (Scenario 3). Further studies must be carried out to determine the usage of the retentate. One attractive option in a kraft pulp mill is to send the retentate to the black liquor recovery cycle.

CONCLUSIONS

The main conclusions of this work are as follows.

- (i) The chosen membrane configuration for the treatment of the (EPO) filtrate was the tight UF membrane. Although this configuration showed a lower selectivity than the nanofiltration configurations, the COD and

Table 7 | Effects of UF on the treatment plant

Parameter	Scenario 2		Scenario 3	
	Softwood (%)	Hardwood effluent (%)	Softwood (%)	Hardwood (%)
COD reduction efficiency	+10.1	+9.8	+6.0	+8.1
Final colour	-9.8	-8.0	-8.3	-8.0
Biological sludge production	0	0	-20.3	-17.1
Energy consumption	0	0	-20.3	-17.1
Coagulant dosage	-30	-37	-40	-40
Sludge production	-28	-35	-44	-46

(+) Increase; (-) Decrease.

colour removal was considered sufficient for the mill requirements, and the operation was simpler and the associated costs lower.

- (ii) The best conditions for a higher permeate flux (over 200 L/m².h) were a CFV of 3 m/s and a TMP of 7 bar. In this case, it was possible to achieve COD and colour removal of 79 and 86%, respectively.
- (iii) The treatment of the (EPO) filtrate using tight UF positively affected the ETP:
 - (a) If the permeate was sent directly to the ETP, the COD removal efficiency increased by 10% in the biological process and the colour of the final treated effluent decreased by approximately 9%.
 - (b) If the UF permeate was recycled to the bleaching plant, the effluent flow rate reduced by approximately 30%, the efficiency of the COD removal in the biological treatment increased by 7% and the generation of biological sludge decreased by 20%. A decrease in the coagulant dosage by 40% in the tertiary treatment and consequently a decrease of tertiary sludge by 45% was also observed.
- (iv) Further studies must be carried to determinate the best usage of the retentate.

ACKNOWLEDGEMENTS

We would like to thank the University Federal de Viçosa and the Fundação de Amparo á Pesquisa do Estado de Minas Gerais (FAPEMIG) for their financial support.

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First received 8 April 2014; accepted in revised form 23 June 2014. Available online 2 July 2014