



Application of activated sludge process followed by physical-chemical processes in the treatment of industrial saline effluent for reuse

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ABSTRACT. The treatment of complex effluent with high salinity and sometimes with toxicity rates from a chemical plant is investigated. Two reactors were monitored continuously: control reactor R1 and reactor R2 adapted for saline effluent with 25h-HRT. The adaptation process to saline effluent (0 - 100%) was effective in removing COD and N-NH₄⁺, respectively with 70 and 85% efficiency. After adaptation, the sequence coagulation/flocculation (40 mg L⁻¹ PAC coagulant and 0.3 mg L⁻¹ cationic polymer), rapid downflow sand filter (120 m³ m⁻² day) and reverse osmosis to obtain water for reuse was analyzed. Results obtained by coagulation/flocculation and sand filter sequence were satisfactory, mainly with the removal rates of turbidity at 50-65 and 98%, respectively. Average removals of 91, 87, 98 and 98% were obtained for COD, N-NH₄⁺, TOC and Cl⁻, respectively, in reverse osmosis, with all parameters below the limits for reuse in cooling towers. However, membrane fouling was detected.

Keywords: water reuse, saline effluent, activated sludge, coagulation/flocculation.

Aplicação do processo de lodos ativados seguido de processos físico-químicos no tratamento de efluente industrial salino visando o reuso

RESUMO. Neste trabalho foi estudado o tratamento do efluente complexo de uma indústria química que apresenta alta salinidade e por vezes, toxicidade. Foram monitorados dois reatores em regime contínuo, sendo R1 o reator controle e R2 o reator adaptado com efluente salino (Efsalino) com TRH de 25h. Verificou-se que o processo de adaptação ao Efsalino (0 - 100%) foi efetivo na remoção de DQO e N-NH₄⁺, sendo obtidas eficiências de remoção de 70 e 85%, respectivamente. Após a adaptação, foi investigada a sequência de coagulação/floculação (40 mg L⁻¹ do coagulante PAC e 0,3 mg L⁻¹ do polímero catiônico), filtro de areia rápido descendente (120 m³ m⁻² dia) e osmose inversa visando obter água para reuso. Os resultados obtidos pela sequência coagulação/floculação e FRD foram satisfatórios, com remoção principalmente de turbidez na faixa de 50 - 65 e 98%, respectivamente. Obteve-se uma remoção média de 91, 87, 98 e 98% de DQO, N-NH₄⁺, COT e Cl⁻, respectivamente, na osmose inversa, estando todos os parâmetros estudados abaixo dos limites máximos permitidos para reutilização em torres de resfriamento. No entanto, foi possível detectar incrustação na membrana.

Palavras-chave: reuso, efluente salino, lodos ativados, coagulação/floculação.

Introduction

Due to excessive population growth and intense industrial development during the last decades, environmental problems, such as the pollution of surface and ground water by fertilizers, pesticides, oils, toxic leachate from landfills and great varieties of industrial and domestic effluents, have become not only more frequent but increasingly critical factors.

Pesticides have been used for over 40 years for their effectiveness in controlling a wide variety of pests, diseases and crop-infesting weeds (EPA, 2004; VRYZAS et al., 2011). However, despite the benefits

to society, pesticides have many undesirable effects such as toxicity, carcinogenicity and mutagenicity (KOURAS et al., 1998).

Industrial effluents vary quantitatively and qualitatively, depending on the types of products manufactured, and on the raw material processes used. Industries generate complex effluents which are characterized as highly difficult to deal with (COSTA et al., 2003).

Owing to environmental requirements and increasing water costs, there is a current trend in industries to optimize its use and engender its reuse (MILLER, 2006).

Efficient advanced treatment techniques have been investigated on industrial and pilot scales so that the above-mentioned objective may be achieved. A process that has been highlighted is that which uses separation by membranes, both for water and for the treatment of effluents. Membrane systems for wastewater treatment may be used independently or coupled to aerobic or anaerobic biological processes, according to the quality of the effluent to be treated and to the desired quality for reuse. Processes with membranes use a smaller area when compared to biological or physical-chemical treatment processes (CARVALHO; MACHADO, 2010; JUANG et al., 2007; METCALF; EDDY, 2003).

In Brazil, the most significant reuses occur in urban, industrial, agricultural areas and in others associated with the artificial recharge of aquifers, particularly for non-potable purposes. An alternative industrial reuse is the use of treated secondary effluent as makeup water in cooling towers, which in Brazil represents only 17% of industrial non-potable water demand (EPA, 2004; HESPANHOL, 2007; METCALF; EDDY, 2003).

Cooling towers are responsible for the highest water consumption rate in most industries and generally do not require high quality water. Consequently, reuse becomes viable (CARVALHO; MACHADO, 2010).

The use of treated secondary effluent in cooling systems, already a successful experience in Brazil and abroad, is a possibility that may be achieved in the short term and with low investment rates (DIAS, 2005).

Table 1 shows the recommended standards for makeup water in cooling towers.

Table 1. Physical and chemical requirements necessary for reuse in cooling towers.

Parameters	Cooling Towers
COD (mg L ⁻¹)	≤ 75 ¹
N-NH ₄ ⁺ (mg L ⁻¹)	≤ 1.0 ²
TOC (mg L ⁻¹)	≤ 1.0 ⁶
Chloride (mg L ⁻¹)	500 ¹
Conductivity (μS cm ⁻¹)	800 - 1200 ⁴
Total Hardness (mg L ⁻¹)	50 - 180 ⁴
Turbidity (NTU)	≤ 3.0 ⁵
pH	6 - 9 ³
TSS (mg L ⁻¹)	≤ 30 ³

¹Metcalf and Eddy (2003); ²Mancuso and Santos (2003); ³US EPA (2004); ⁴EPA (2004), values for San Francisco city; ⁵EPA (2004), values for Texas; ⁶EPA (2004), values for the state of Washington.

The above rates show that water reuse in industries is viable and it is already a fact in various industries. So that the feasibility of water reuse programs in industry may be demonstrated, current analysis presents the results of a study involving the treatment of effluent generated by a pesticide-

producing chemical plant through a combination of physical and chemical processes, emphasizing its suitability for reuse in cooling towers.

Material and methods

Wastewater used as feed for the biological reactors came specifically from a tank which received several types of effluents (with a 25h-retention time) prior to arrival at the equalization tanks. On the other hand, the control effluent came from the primary settling (after the introduction of effluent from the plant's incinerator), before entering into the biological activated sludge process, respectively called EfSaline and EfControl. EfSaline characterization was carried out after the primary treatment in the laboratory with pH adjustment and settling. Samples were provided monthly and characterized by several parameters.

The first stage comprised the assessment of activated sludge process under continuous conditions with 25h-HRT in the adaptation of saline effluent (EfSalino) (0 to 100%). A control reactor (R1) fed with effluent from the primary settling of the chemical industry (EfControle) was employed to compare the results with the reactor fed with the saline effluent (R2) from the same industry. The analyzed parameters were COD (Chemical Oxygen Demand) and N-NH₄⁺ according to procedures in Standard Methods (APHA, 2005).

After the adaptation period, coagulation/flocculation tests with the biotreated effluent were done in Digimed jar test (flocculator module Mf-01) with several types of coagulants and coagulation aids to establish the best conditions.

Tables 2 and 3 show three coagulants and two cationic polyelectrolytes.

Optimal concentrations of poly aluminum chloride coagulant (PAC) and cationic polymer flocculant of low MW (650) were determined. The effluent passed through a descending rapid sand filter with a filtration rate of 120 m³ m⁻² day to remove suspended solids. The experimental system consisted of a descending rapid filtration column (FRD) in a glass measuring 3.92 cm diameter and 45 cm height. The filter bed contained a 20 cm-layer of sand with a particle size between 0.841 and 1.84 mm, as recommended by Di Bernardo (2003).

Table 2. Coagulants used in coagulation/flocculation tests.

Product Name	Chemical Description	Manufacturer's Name
Aluminum Sulfate	Inorganic Coagulant	AQUAFLOC
Ferric Chloride	Inorganic Coagulant	VETEC
Poly Aluminium Chloride (PAC)	Inorganic Coagulant	AQUAFLOC

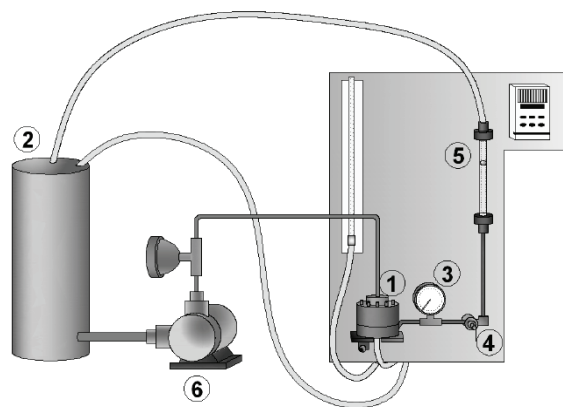
Table 3. Flocculants used in coagulation/flocculation tests.

Product Name	Chemical Description	Manufacturer's Name
650	Cationic polymer (low molecular weight)	AQUAFLOC
800	Cationic polymer (high molecular weight)	AQUAFLOC

Minimum application rate for rapid filtration was estimated by a true hydraulic load of 12 cm water column. Since the filter's purpose was only to analyze the quality of the filtrate, it was performed at declining rates, i.e., the rates were reduced during the period to maintain the same hydraulic load and the best quality effluent. Analyses of color, turbidity, chemical oxygen demand (COD), ammonia nitrogen, total suspended solids (TSS), volatile suspended solids (VSS), Total Organic Carbon (TOC), conductivity and micro-organism counts were carried out held at the input and output of the filter, following procedures by Standard Methods (APHA, 2005).

After this stage, bench scale reverse osmosis system was fed with output effluent from the descending rapid sand filter at a constant 30 bar pressure. Before the experiment, the membrane was characterized by determining the hydraulic permeability and NaCl rejection ($2,000 \text{ mg L}^{-1}$). The pressure ranged between 10 and 30 bar during the characterization procedure.

Reverse osmosis tests were performed on an assembled system at the Laboratory of Membrane Processes (PAM/COPPE/UFRJ), at bench scale, as Figure 1 shows.

**Figure 1.** Reverse osmosis system.

The permeation system comprises a stainless steel cell (1), in which the membrane is conditioned, a feed tank (2) with approximately 4 L, a manometer (3) at the output of the permeation cell, regulating valve pressure (4), rotameter (5) and pump (6) for recirculation of the feed solution. The system works with concentrate and permeate recycles.

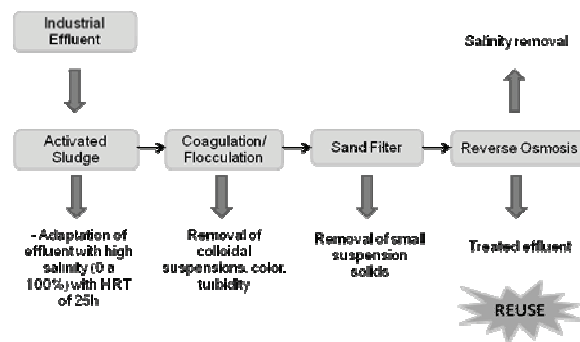
The reverse osmosis membrane for tests was FILMTEC/DOW (BW30-2540) with the following ratings: hydraulic permeability $1.2 \text{ L h}^{-1} \text{ m}^2 \text{ bar}$, salt rejection 99.4%, free chlorine lower than 1 mg L^{-1} , pH between 2 and 11, maximum temperature 45°C , maximum operating pressure 69 bar; maximum degree of recovery 15%. The membrane was chosen due to its efficiency in NaCl rejection when compared to other membranes and to its frequent use in water recycling (ALI ALTURKI et al., 2010; SAGNE et al., 2008).

The permeate was characterized by analysis of conductivity, TOC, ammonia nitrogen, chloride and hardness with 10 and 20% recovery rates. The parameters analyzed at this stage followed procedures recommended by Standard Methods (APHA, 2005). A total carbon analyzer Shimadzu 5000-A was used to determine TOC.

The presence of membrane fouling was verified by membrane analysis with scanning electron microscopy (SEM) before and after reverse osmosis test using the Scanning Electron Microscope FEI Company Quanta 200 with Oxford Micro Analysis Instruments - Penta FETx3 and by the determination of Silt Density Index (SDI) (ASTM, 2002).

SDI is important for the design of a reverse osmosis system since it correlates water quality and future potential blockages and decreases flow through the membrane (SILVA et al., 2009).

Figure 2 shows the treatment of effluent by several processes and their sequence in current analysis.

**Figure 2.** Flowchart of process in the treatment of industrial effluent used in current assay.

Results and discussion

The effluent used in current assay came from a pesticide-producing chemical plant producing pesticides and its composition featured high complexity and variability, with high salt contents and sometimes significant toxicity levels (WORLD BANK GROUP, 1998). Effluent's complexity was

related to the large number of chemicals produced (it also varied according to the different cultures at different times of the year), to unit operations and to chemical transformations involved in the products' various production stages.

Effluents contained significant amounts of soluble and recalcitrant organic materials, such as phenolic compounds and polyaromatic hydrocarbons, and several aromatic compounds used in organic synthesis reactions.

Table 4 presents the characterization of the effluents with the parameters' variation range.

Table 4. Characterization of industrial effluents.

Parameters	EfControl	EfSalino
COD (mg L ⁻¹)	390 – 1170	400 – 1640
TOC (mg L ⁻¹)	50 – 120	90 – 200
N-NH ₄ ⁺ (mg L ⁻¹)	34 – 56	36 – 50
pH	7.8 – 8.4	7.6 – 8.3
Chloride (mg L ⁻¹)	2400 – 7000	2900 – 8800
Total Phenols (mg L ⁻¹)	0.11 – 0.45	0.14 – 0.47
Conductivity (μS cm ⁻¹)	7000 – 16600	17000 – 20400
TSS (mg L ⁻¹)	55 – 770	60 – 75
VSS (mg L ⁻¹)	30 – 300	25 – 54
Turbidity (NTU)	16 – 62	19 – 65
Appearance	Dark yellow, opaque	

It may be observed that the effluent showed a high variability with regard to most parameters analyzed which may interfere in the performance of the biological process. Such variability originated from the variety of products manufactured and from a greater production of certain products to the detriment of others according to the season.

The parameters analyzed at this stage were compared with the effluent disposal limits according to state (INEA) and federal (CONAMA) legislation given in Table 5.

Table 5. State and federal legislation with regard to effluent discharge.

Parameters	CONAMA 357/05	NT202/86 and DZ205/91 (INEA, 1986, 1991)
COD (mg L ⁻¹)	-	250
N-NH ₄ ⁺ (mg L ⁻¹)	20.0*	5.0**

*Rate for direct or indirect releases of liquid effluents from polluting activities into open water-bodies, such as the Sarapuí river; **Rate for direct or indirect releases of liquid effluents, from polluting activities into water bodies from lakes in the state of Rio de Janeiro.

The main standards and guidelines on the release of wastewater into receiving water-bodies in the state of Rio de Janeiro are produced by the INEA (Environment State Institute), the state's environmental agency. The standards and guidelines determined by the INEA are in accordance with Brazilian environmental legislation, as determined by CONAMA (National Environmental Council).

Figures 3 and 4 present the monitoring of COD concentration in the feed and at the outlet of

reactors R1 and R2 and their removal efficiency during the period of sludge acclimatization to EfSalino in R2.

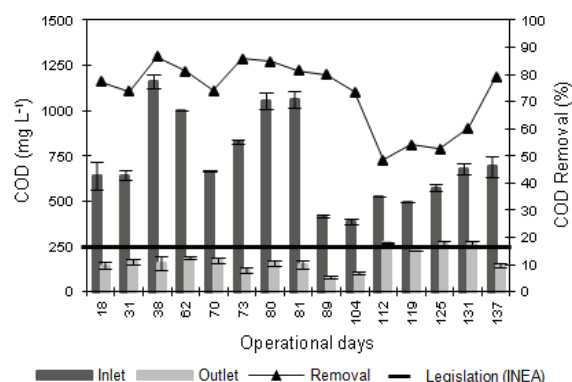


Figure 3. Monitoring of COD with EfControl (R1).

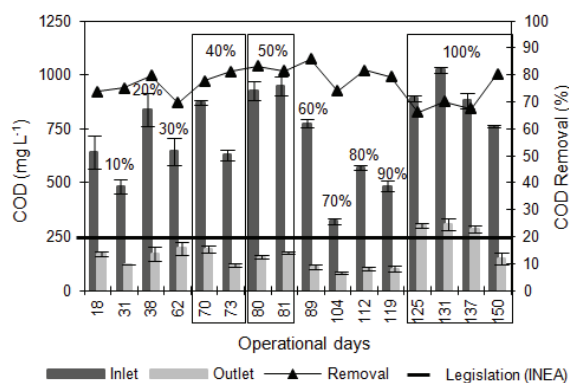


Figure 4. Monitoring of COD in the period of adaptation to EfSaline (R2).

When percentage of EfSaline in R2 increased every 10%, rates of output and COD removal were similar to those of R1 values throughout the adaptation period. COD removal rate was always above 70% and frequently below the limit prescribed by state legislation for effluent disposal (250 mg L⁻¹) (INEA, 1991).

The depuration efficiency of the biological process was also assessed by the determination of ammonia nitrogen rates. Figures 5 and 6 show the concentrations of ammonia nitrogen at the reactor's feed and outlet and its efficiency of removal.

Average efficiency of 90 and 85% N-NH₄⁺ removal was achieved in reactors R1 and R2, respectively, during the adaptation period to EfSalino in R2. It should be emphasized that N-NH₄⁺ removal was high during the operation of reactors. Recalcitrance and possible toxicity of the effluent failed to change their removal which was always below the limits allowed by state (5.0 mg L⁻¹) (INEA, 1986) and federal legislation (20.0 mg L⁻¹) (CONAMA, 2005) for effluent disposal.

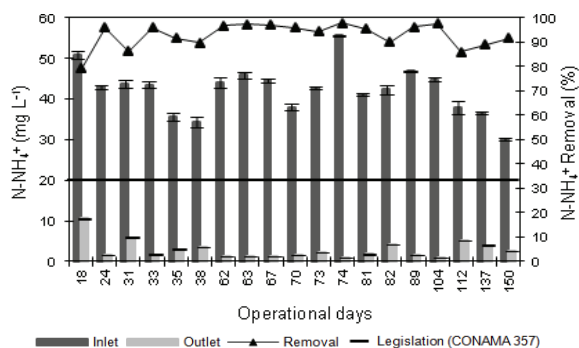


Figure 5. Monitoring of N-NH_4^+ with EfControl (R1).

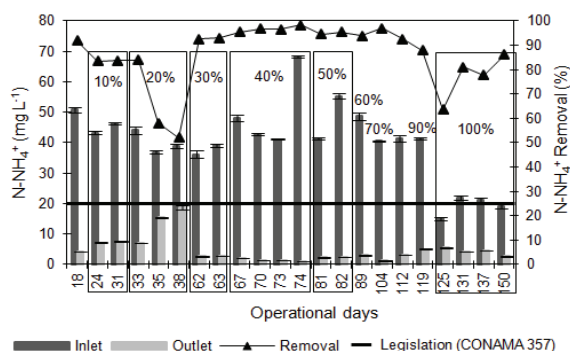


Figure 6. Monitoring of N-NH_4^+ during the adaptation period to EfSalino (R2).

The range of color and turbidity removal in jar-test assays for the different sets of coagulant and polyelectrolyte investigated is shown in Table 6.

Table 6. Results of coagulation/flocculation tests for color and turbidity removal.

Coagulant + Polyelectrolyte	Turbidity removal (%)	Color removal (%)
Aluminum Sulfate + 650	0 - 9.1	1.6 - 9.7
Aluminum Sulfate + 800	7.7 - 23.1	5.7 - 22.9
Ferric Chloride + 650	0	0
PAC + 650	46.1 - 54.0	44.3 - 52.9
PAC + 800	0 - 33.3	0 - 26.1

Results in Table 6 show that when PAC coagulant set and low MW flocculant (650) were used, respectively at 40 and 0.3 mg L^{-1} concentrations, turbidity and color removal rates were higher than those of other coagulants. Di Bernardo (2003) also obtained the highest turbidity removal rates with PAC coagulant when compared with those from other sulfate salts assays based on aluminum silicate and aluminum sulfate. After determining the optimal concentrations of coagulant/flocculant set, assays with 15 L were performed. Color and turbidity removal rates were obtained respectively ranging between 23 and 33% and between 50 and 65%.

Through the characteristics of the coagulation/flocculation process-treated effluent, filtration was employed as a further process.

It evaluated the behavior of the filter with regard to turbidity and small solids (colloidal particles) removal in the effluent after the coagulation/flocculation process.

Tables 7 and 8 show that parameters TSS, VSS, turbidity and color had the best results obtained during the 6-hour descending rapid sand filter operation.

Table 7. Results of descending rapid sand filter tests for color and turbidity.

Day	Time (h)	Color (uH)	η (%)	Turbidity (NTU)	η (%)
1°	0	35	35	0.69	85
	1	30	44	0.34	92
	2	33	39	0.09	98
	3	34	37	0.15	97
2°	4	31	43	0.15	97
	5	35	35	0.15	97
	6	42	22	0.25	94
Feed	-	54	-	4.50	-

Table 8. Results of descending rapid sand filter tests for TSS and VSS.

Day	TSS (mg L^{-1})	η (%)	VSS (mg L^{-1})	η (%)
Feed	29.3	-	9.3	-
1°	22.2	24	7.0	25
2°	11.2	62	5.0	46

With regard to TSS and VSS removal rates evaluated on the following two consecutive days, removal rates reached respectively 24 and 25% on the first day and 62 and 46% on the second day. In fact, TSS and VSS removal rates were more significant on the second than on the first. This was due to a slight blockage on the filter bed during the period, or rather, solids during operation were removed from the effluent and accumulated within the voids and at the top of the filter bed surface. Actually a reduction of the effluent flow was required during the period. The filter practically removed all possible sedimentary solids.

The results of color and turbidity removal rates are shown in Figures 7 and 8, respectively.

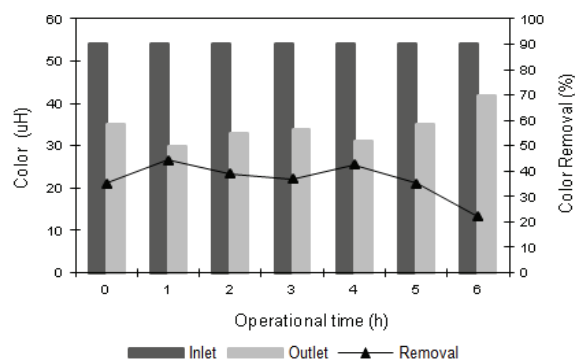


Figure 7. Monitoring of color at the reactor outlet in descending rapid sand filter.

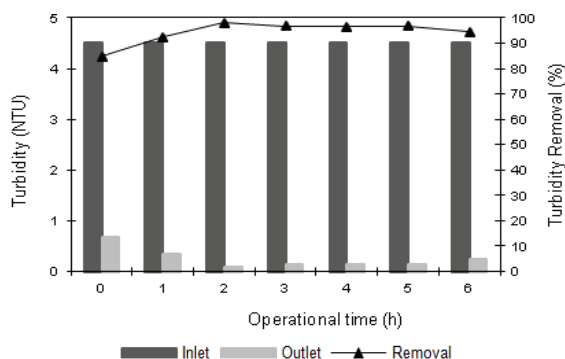


Figure 8. Monitoring of turbidity at the reactor outlet in descending rapid sand filter.

Turbidity and color removal efficiency reached 98 and 44%, respectively. According to Richter (1977), filters' performance may be over 90% in turbidity removal, when design and construction failures are excluded in a carefully planned operation.

Further, 4 L of the effluent from the outlet of the rapid descending filter were placed in the feed tank of the reverse osmosis system at a pressure of 30 bar. The system was monitored according to the permeate's recovery degree. Analyses for conductivity, COD, TOC and ammonia nitrogen were carried out at each 10% permeate volume. Tables 9 and 10 show the results.

Table 9. Results of reverse osmosis tests for COD and N-NH_4^+ .

Recovery Degree	COD (mg L^{-1})	η (%)	N-NH_4^+ (mg L^{-1})	η (%)
10	15	91	0.76	87
20	19	89	0.90	84
30	14	92	0.99	83
40	-	-	1.06	82
Feed	170	-	5.75	-

Table 10. Results of reverse osmosis tests for TOC, Cl^- and conductivity.

Recovery Degree	TOC (mg L^{-1})	η (%)	Cl^- (mg L^{-1})	η (%)	Cond. ($\mu\text{S cm}^{-1}$)
10	0.607	98.5	86	98	444
20	0.524	98.7	86	98	303
30	0.466	98.8	-	-	360
40	2.126	94.6	-	-	594
Feed	40	-	6600	-	23240

Tables 9 and 10 showed that the best results were obtained with recovery degree 10%. When results were compared with maximum values of water quality recommended by the literature for replacement in open recirculating cooling systems (Table 1), all parameters analyzed fitted within the re-usage scheme.

Many feeds contain contaminants (fouling) that reduce at an early period the membrane's productivity (flux). Experiments show that the flow

of the permeate through the membrane decreased with time. However, it should be noted that current assay is a bench test using a plane membrane.

According to Gabelich et al. (2003), the lowest salt rejection in conventional treatment may be attributed to membrane fouling. This fact may increase the polarization layer concentration by 40% of the recovery degree, as shown in Table 10, where the conductivity of the permeate increased and salt rejection increased.

Small or large fouling formation depends on the module's configuration and on the contaminants' type and concentration in the feed. Tests carried out to determine the membrane's fouling potential showed a rate of 17 for SDI at $t = 5$ min. This rate is higher than that recommended for the reference process ($\text{SDI} \leq 5$). Membrane fouling with a reduction in the process's efficiency was detected with this parameter.

Photographs of scanning electron microscopy (SEM) analysis of the membrane before and after the reverse osmosis test are shown in Figure 9 so that membrane's fouling may be proved.

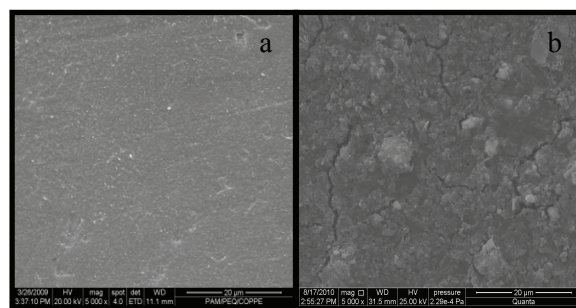


Figure 9. Photos of the BW-30 membrane surface magnified 5000X before (a) and after (b) the reverse osmosis test.

The significant difference between the membrane's surfaces before and after the reverse osmosis test may be noted. SEM analysis showed a thick layer on the membrane's surface, probably of organic origin. According to Gabelich et al. (2003), this is due to the fact that part of TOC from the feed stream was deposited on the membrane and may be used as a substrate for bacteria growth, establishing an adequate environment for biofouling.

Conclusion

The adaptation process to EfSalino (0 - 100%) was effective in removing COD and N-NH_4^+ , with removal efficiencies of 70 and 85%, respectively. These parameters were often within the permissible disposal limits (CONAMA, 2005; INEA, 1986 and INEA 1991).

Among the results obtained by combined process (coagulation/flocculation, descending rapid sand filtration and reverse osmosis), best results lay in the recovery degree of 10%, with an average removal of 91, 87, 98 and 98% respectively for COD, N-NH₄⁺, TOC and chloride, final effluent with re-usage characteristics in the feed of the cooling towers. All parameters studied were below the maximum rates for re-use. However, fouling in the membrane was detected by the SDI test and by SEM. SDI rate of 17, was much higher than that recommended for the reference process (SDI ≤ 5). Further, SEM analysis showed a thick layer on the membrane surface, probably of organic origin.

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