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Nanofiltration of secondary effluent for wastewater reuse in the textile industry

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Abstract

Textile industries represent an important environmental problem due to their high water consumption. In many Spanish regions with water scarcity, this fact can be an argument to make wastewater reuse necessary. In this work, a biologically treated wastewater from a cotton thread factory was subjected to nanofiltration (NF) in two ways, direct NF treatment and NF after a pre-treatment stage by ultrafiltration (UF). Nowadays, the factory effluent is treated by an activated sludge process. This effluent has high values of COD (200 $mgO_2\,L^{-1}$) and TDS (5000 $mg\,L^{-1}$) which makes its use in the textile processes impossible.

In such situations, reverse osmosis (RO) has been typically used with the purpose of reuse. However, NF can achieve enough permeate quality for certain processes at a lower operating pressure than RO.

To verify the possibility of reusing textile wastewater with NF, three NF membranes of different pore size (NF90, NF200 and NF270 from Dow-Filmtec) were studied. In order to select the most effective one, experiments were carried out at four different pressures in a pilot plant equipped with a flat-sheet membrane module. The studied responses were permeate flux, salt retention and COD removal.

The NF90 yielded a COD reduction of 99% and the highest salt retention (75–95%). As the permeate quality obtained with this membrane was satisfactory, long duration experiments were performed with a spiral-wound module in order to scale-up the system and to study the effect of fouling. These experiments showed that the levels of COD removal and salt retention were not significantly affected by fouling and that a high flux percentage could be retrieved after cleaning.

Finally, UF and NF experiences were coupled in order to study the effect of UF as pre-treatment in a NF system. In this case, permeate flux of NF increased a lot (about 50%) and COD concentration in NF feed was reduced (about 40%).

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Therefore, it could be concluded that the use of NF with a suitable pre-treatment foresees an important percentage of reuse resulting in less environmental impact with lower energy costs compared to a RO based system.

Keywords: Nanofiltration; Textile wastewater; Reuse; COD removal

1. Introduction

Textile industries represent an important environmental problem due to their high water consumption. In the production of a tone of textile product 200–350 m^3 of water are consumed [1,2]. This water is used both in specific operations like dyeing, bleaching, finishing, etc. and in cleaning operations for which a good quality of water is necessary.

Parameters of major interest in the textile wastewaters concern chemical oxygen demand (COD), total organic carbon (TOC), conductivity and colour. The most common treatments applied to treat and recycle textile wastewater comprise biological treatment, precipitation, coagulation/flocculation, flotation, oxidation and adsorption [3–7].

However, the efficacy of these treatments is not sufficiently high as most compounds cannot be easily degraded. This originates a residual colour and COD. Besides, these methods are not able to decrease salinity.

Therefore, membrane filtration can be an optimal solution to remove colour, COD and salinity [8,9]. One of the most promising technologies is nanofiltration (NF). This membrane technology can achieve higher COD rejection than ultrafiltration (more than 90%) with greater flux than reverse osmosis (RO) and sometimes less fouling problems [10]. On the other hand, NF membrane separation has been used successfully in the textile wastewater treatment to obtain industrial water from textile effluents [11–13]. Despite this, in order to obtain good efficiency and to prevent fouling in the NF membranes, a correct pre-treatment has to be considered [14,15].

NF falls between ultrafiltration and reverse osmosis; its separation characteristics are based on sieve effect, but most commercial NF membranes are also charged. So, ion rejection by NF membranes results from the combination of electrostatic and steric interactions associated with charge shielding, Donnan exclusion and the degree of ion hydration [16,17].

The aim of this paper is to verify the possibility of reusing the secondary effluent from a textile industry by a nanofiltration (NF) process.

In this work, the experimentation has been structured in the following stages:

In the first experimental stage, experiments with flat sheet membranes of different pore size were performed to select the most effective one. Then, the second stage consists of long duration experiments with a spiral-wound module of the NF membrane selected. In these experiments, the effect of fouling is studied through a continuous measure of permeate flux, salts and COD retention. The last experimental stage consisted of experiments in which the performance of NF was tested after an UF stage.

2. Experimental

2.1. Wastewater characteristics

The feed stream was, in all experiences, the effluent from an industrial wastewater plant (IWP) of a textile industry located in Valencia (Spain). In this industry the main operations are dyeing and scouring. After all textile processes, wastewater presents a dark blue colour, a very high pH and high conductivity. This effluent is treated in the IWP, which includes a physicochemical process and a biological reactor. The main residual

Table 1
Textile wastewater characteristics before and after biological treatment

Parameter	Feed water IWP textile	Secondary effluent
pH	12–14	7.5–8.5
Colour	Dark blue	Light yellow
COD ($mgO_2\ L^{-1}$)	1500–2000	100–150
Conductivity ($\mu S\ cm^{-1}$)	6000–7500	6000–7000
Temperature (°C)	35–40	30–35

contaminants are salts, colour and COD and their concentrations are highly variable (Table 1).

A typical value for the feed to NF system, obtained in a complete analysis, is showed in Table 2.

2.2. Materials and methods

2.2.1. Experiments for membrane selection

The experiments with the aim of determining the optimal NF membrane were carried out on a laboratory scale pilot plant. A scheme is shown in Fig. 1.

The NF module was equipped with four flat sheet membranes and a total filtration area of 120 cm^2. Three commercial thin-film composite polyamide membranes manufactured by Dow-FilmTec were tested: NF90, NF200 and NF270.

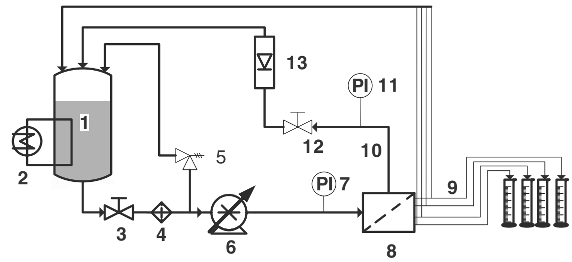

Fig. 1. Scheme of the NF laboratory plant. 1: feed tank; 2: heat exchanger; 3: valve; 4: security filter; 5: safety valve; 6: feed pump; 7: manometer; 8: NF module; 9: permeate stream; 10: concentrate stream; 11: manometer; 12: regulation valve; 13: rotameter.

Membranes were previously characterised with deionized water, and afterwards, they were exposed to the secondary effluent. The temperature was established at 25°C. The effect of the pressure was studied with experiences over the range of 6 to 22 bar. The recirculation flow and temperature were fixed at 340 L h^{-1}. The studied responses were permeate flux, salt rejection (based on conductivity) and COD removal.

2.2.2. Pilot plant studies

After the selection study with flat-sheet membranes, the NF90-2540 spiral-wound

Table 2
Secondary effluent

Parameter	Value	Parameter	Value
pH	7.8	Ammonium (mg L^{-1})	2
Conductivity (µS/cm)	7500	Chlorine (mg L^{-1})	1200
SS (mg L^{-1})	48	Sulphates (mg L^{-1})	1780
BOD_5 ($mgO_2\ L^{-1}$)	45	Bicarbonates (mg L^{-1})	560
COD (mg L^{-1})	150	Nitrates (mg L^{-1})	1.2
Colour	1/20	Silica (mg L^{-1})	5
Sodium (mg L^{-1})	1700	Iron (mg L^{-1})	29.6
Potassium (mg L^{-1})	17	Phosphor (mg L^{-1})	3.1
Calcium (mg L^{-1})	80	Oil total (mg L^{-1})	5
Magnesium (mg L^{-1})	20	Detergents (mg L^{-1})	0.2

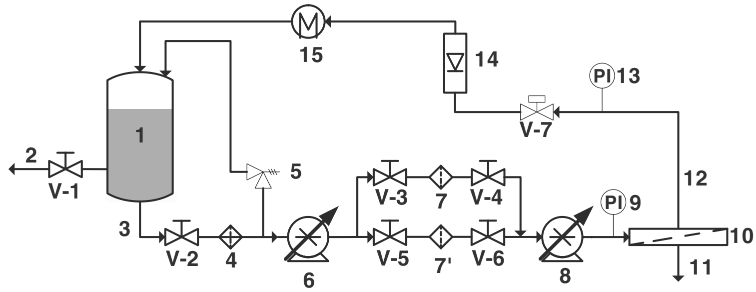

Fig. 2. Scheme of the NF industrial pilot plant. 1: feed tank; 2: tank emptying; 3: feed stream; 4: security filter; 5: security valve; 6: low-pressure pump; 7: 7' cartridge filter system; 8: high pressure pump; 9: manometer; 10: NF spiral wound module; 11: permeate stream; 12: concentrate stream; 13: manometer; 14: rotameter; 15: heat exchanger.

module was selected for the experiments in an industrial pilot plant (Fig. 2) situated in the factory.

The NF plant was operated in the batch recirculation mode. Permeate and retentate were recycled back to the feed tank of 1 m^3. Every week the feed tank was filled with secondary effluent from the IWP and prefiltered over a 50 micron cartridge filter. The following responses were studied: temperature, permeate flux, COD and conductivity rejection.

Two types of experiments were performed:
– Direct NF experiments
– Combined UF/NF experiments

In the direct NF experiments, NF was operated at a pressure of 10 bar and at a feed flow rate of 750 L/h. Fig. 3 shows the COD and conductivity values of the treated wastewater. During six months the plant was operated five days a week. Each day an experiment, which lasted 9 h, was performed. This meant a total experimental time of 527 h. Every week, the feed tank was filled with fresh wastewater. The membranes were cleaned by flushing tap water during the hours while the filtration was not active.

In the combined NF experiments, permeate from a 10 kDa Persep^{TM} (4-inch) spiral-wound membrane supplied by Orelis-Novasep was used as feed solution. The feed flow and operating pressure were also 750 L/h and 10 bar.

3. Results and discussion

3.1. Results of experiments for membrane selection

The permeability results for the three membranes studied when treating the secondary effluent are shown in Fig. 4.

Theoretically, attending to the MWCO of the membranes used, the flux sequence should be J(NF270) > J(NF200) > J(NF90). However, some of the sheets of the NF200 membrane yielded higher flux than NF270. Besides, NF200 and NF270 showed high dispersion in the permeability. Results show that NF200 and NF270 yielded similar salt rejection, and that NF90

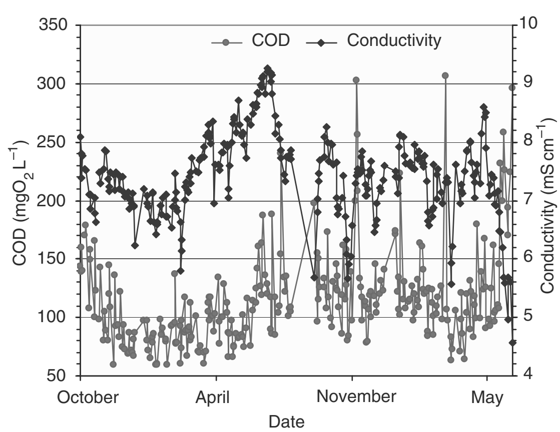

Fig. 3. Conductivity and COD values of treated textile wastewater during six months.

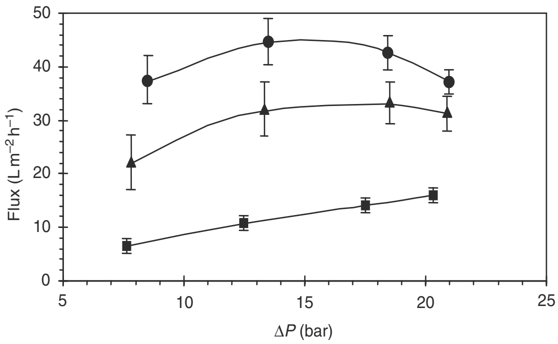

Fig. 4. Flux obtained for secondary effluent in different flat-sheet membranes. (●) NF 270, (▲) NF 200, (■) NF 90.

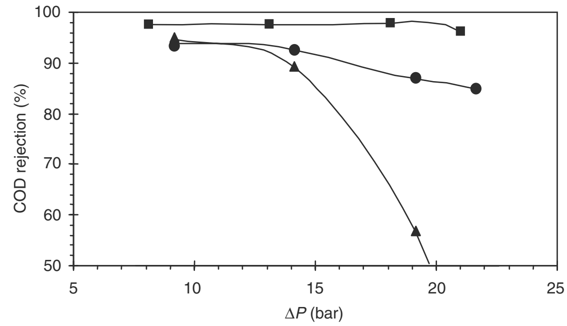

Fig. 6. Minimum values of COD rejection obtained for secondary effluent in different flat-sheet membranes. (●) NF 270, (▲) NF 200, (■) NF 90.

was able to achieve a salt rejection index over 85% (Fig. 5). It was also observed that the higher the pressure, the higher the salt rejection for all membranes studied. Nevertheless, the COD rejection showed a different behaviour (Fig. 6). For the membrane NF90, high COD rejection values were always obtained and their values were very independent of pressure. The NF200 and NF270 had always smaller values of COD rejection and for both it was observed that the effect of pressure was to slightly decrease the COD rejection.

Considering these facts, the membrane NF90 was selected for further experiments as its permeate was able to meet the minimum specifications required for the factory. A pressure of 10 bar was also selected as a balance between the flux and the rejection values.

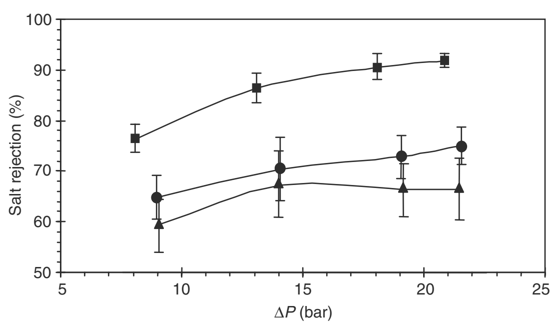

Fig. 5. Salt rejection obtained for secondary effluent in different flat-sheet membranes (●) NF 270, (▲) NF 200, (■) NF 90.

3.2. Pilot plant studies with direct NF

Fig. 7 shows the results of normalized flux at 30°C obtained treating the secondary effluent of the factory. It can be seen that the water cleaning made it possible to retrieve almost all the flux during the non-experimentation period. The flux decrease between experiments was attributed to the increase in COD and salt concentration of the feed. Salt rejection (Fig. 8) was always in the range of 95–97% being not very influenced by feed concentration. Permeate conductivity (Fig. 9) always meets the specification required by the factory (<400 μS/cm).

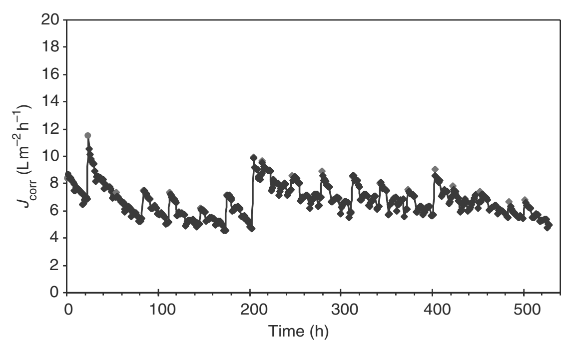

Fig. 7. Normalized flux at 30°C of NF90-2540.

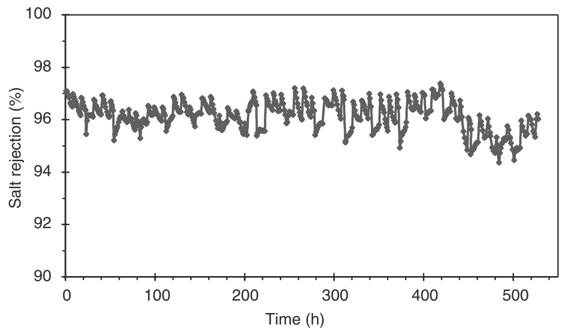

Fig. 8. Salt rejection of NF90-2540.

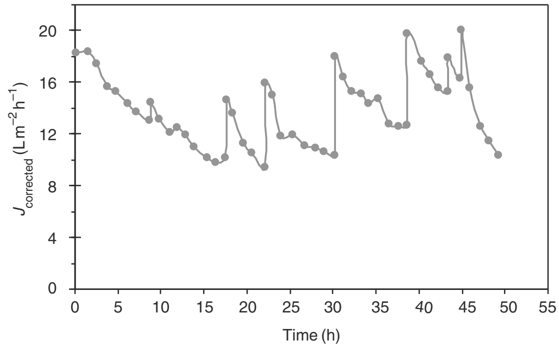

Fig. 10. Flux at 30°C of NF90-2540 after a UF 10 kDa Peresep™.

3.3. Pilot plant studies with combined UF/NF

Comparing Fig. 7 and Fig. 10, the previous use of UF largely improved the flux of the NF membrane. The UF used removed between 30 and 40% of the COD. However, the COD fraction retained was responsible for most of the fouling. The UF module presented a severe fouling and had to be subjected to continuous cleaning operations. After the UF treatment, the NF flux nearly doubled and the initial flux after flushing with water was almost completely retrieved. As it can be seen in Fig. 11, the salt rejection was slightly smaller than in the case of direct UF.

4. Conclusions

After studying the tree NF membranes, it was concluded that the permeate obtained with the NF90 could meet the specifications for water reuse in the textile industry (conductivity < 500 µS/cm and COD < 20 mgO_2/L). The other NF membranes showed higher flux but smaller rejection with a greater variability of the permselective results. Conversely, the NF90 yielded flux and rejection values very independent of feed composition.

An important fouling was observed after direct treatment of secondary effluent with a

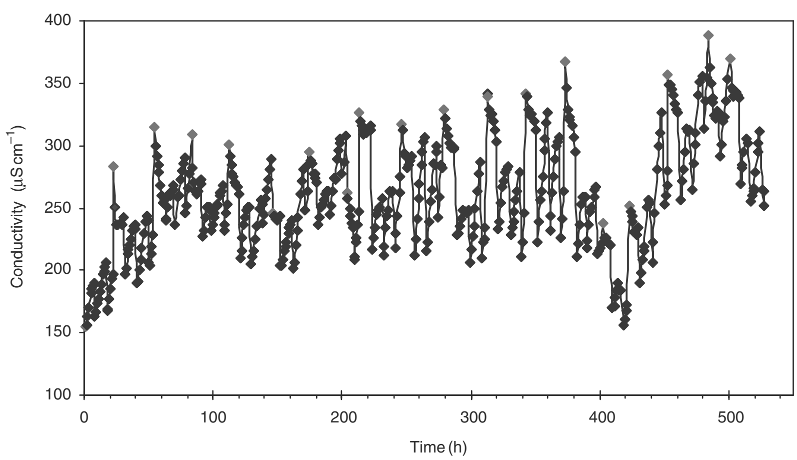

Fig. 9. Permeate conductivity of NF90-2540.

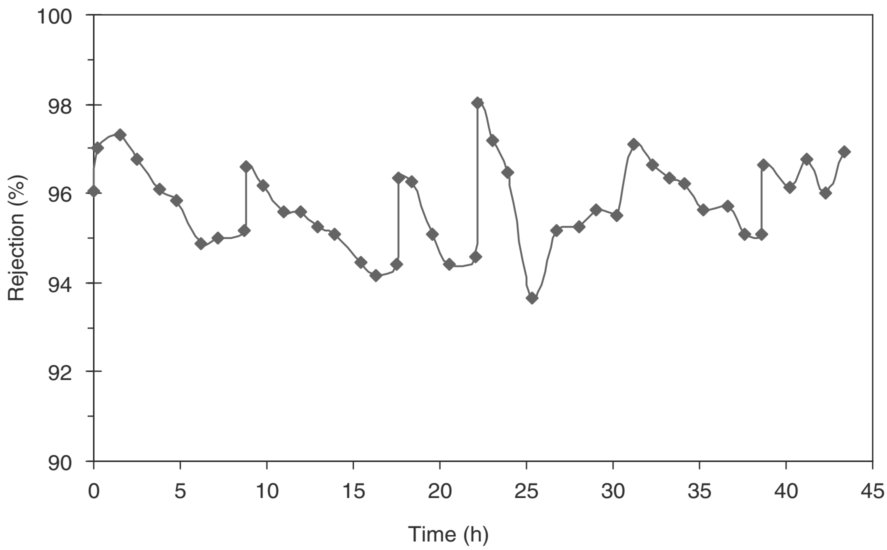

Fig. 11. Salt rejection at 30°C of NF90-2540 after a UF 10 kDa Peresep™.

NF90 spiral-wound module. However, most of membrane permeability could be retrieved by cleaning by water flushing.

The application of the UF eliminates colloids and macromolecules of MWCO greater than 10,000 that are responsible for most of the fouling. It is recommendable the use of an UF stage previous to the NF as the nanofiltration flux almost doubles and the effect of cleaning was also improved.

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