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Treatment of Effluent Textile Using Nanofiltration: Study of Fouling and Antifouling

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Abstract: The textile industry is a large water consumer. As regulations become stringent and the cost of freshwater increases, reclamation of wastewater becomes more and more attractive. Membrane methods, including UF, MF and NF, belong to high-efficiency processes which can be competitive with traditional methods of water treatment. In this paper the nanofiltration membrane, has shown their effectiveness as a solution for the cleaning and recycling of textile effluents. Experiments demonstrated that membrane treatment is a very promising advanced treatment option for pollution control for textile industry effluents. However, the phenomenon of fouling seems to be their major disadvantage, thus causing a crucial reduction in productivity and a high cost of maintenance. Trying to contribute to the existing efforts to cure to this problem, our study is related to the analysis and the development of the parameters which assign the fouling and the cleaning of the NF membranes during the filtration of the textile effluents containing reactive dyes. This study enables us to get rid of this phenomenon, which occurs during the frontal filtration of the solutions containing a mixture of "dye-salt". For this purpose, three theoretical models were compared. We concluded also that rapid cake formation occurred during initial filtration and caused an initial sharp decrease in permeate flux, and the most descriptive model is cake filtration. A regeneration efficiency index allows increasing the optimal conditions of this operation.

Keywords: Nanofiltration, Fouling, Antifouling, Effluent Textile, Membrane

1. Introduction

The textile industry is one of the most polluting industries in the world. This industry consumes a large amount of water that involves chemical substances such as dyes, salts, and surfactants. The processes of dyeing, rinsing produce a large quantity of waste water wich cannot be discharged directly because of their dramatic impact on the environment [1].

The wastewater from the dying processes is generally characterized by a high color index (caused by dyes, salts...), chemical oxygen demand (COD), derived additives such as acetic acid, detergents, complexing agents, and suspended solids, including fibers. Textile industries produce about 200-500 liters of waste water per kilogram of finished product [2] an average causing pollution of 0.1 kg COD per kilogram of tissue [3].

Reactive dyes have received a lot of attention in the field of membrane research because the efficiency of their elimination by biological processes is very low. Also, reactive dyes are the major sources of color in textile effluent and have low rates of fixation on tissue.

Many techniques are used for textile waste water treatments [4]: Biological (aerobic; anaerobic),

physicochemical (coagulation-flocculation), chemical oxidation, adsorption, and membrane technologies.

Intensive studies are devoted to their purification by membrane filtration [5, 6]. Despite their expensive cost, these processes are widely repaid by significant savings through the reuse of chemicals, dyes, and water.

In particular, pressure-driven techniques, such as microfiltration (MF), ultrafiltration (UF), nanofiltration (NF) and reverse osmosis (RO), play a very important role in water purification nowadays, because of their small environmental impacts [7].

For these reasons, nanofiltration has been selected for the treatment of dye baths containing salt and reactive dye. They have been evaluated to remove color and reuse of effluents [2]. Commercial NF are essentially thim film composites containing a polyamide layer over a more parous and hydrophobic polysolfone or polyether sulfone support layer.

The main problem with membrane technology is the loss of flux, defined as the decrease in the volume of liquid passing through the membrane by unit of time and area. It occurs because of two phenomena: concentration polarization of chemical elements and clogging.

The purpose of the work is to develop the factors influencing clogging and membrane washing.

2. Materials and Methods

2.1. Materials

The used membrane is an NF polyamide OSMONICS

Deasal 5 DK (polysulfonepolyamide) supplied by Osmonics as a flat sheet, thin film nanofiltration membrane and characterized by an approximate molecularweight cut-off of 150-300 Da. The dye is a CIBA (Cibacron BLACK WNN HC). It's a dye reactive (at 60°C) highly concentrated giving shades very deep black on the cellulosic fibers. The molecular weight of this dye is relatively large compared to that of NaCl (923g.mol[-1]) NaCl was chosen because it's the most commonly used salt in the textile industry. Sodium chloride and distilled water were also used in the preparation of different dye and NaCl concentrations in the laboratory conditions.

It was conditioned by filtration of pure water with applied progressive pressure levels for 15 minutes before testing. The clean membrane permeability was 5.06 L.h[-1].

2.2. Experimental Step-up and Methods

Firstly, the membrane was kept in pure water for about 24h to remove unwanted compounds. Then, high pressure pump was used for generating the required pressure and for circulating the solutions in the membrane cell. The membrane was installed in the cell of the operating fonctionnement 8 bar for about 2h. Simultaneously, pure water is circulated to ensure membrane compaction.

We chose dye/salt mixtures at different concentrations. All experiments were carried out at a constant temperature of 25+1°C.

The details of synthetic solutions are presented in table 1.

Table 1. Composition of synthetic treated solution.

Solutions references	dye concentration (g.L^{-1})	Nacl concentration (g.L^{-1})
S1.1	1	1
S1.10	10	1
S1.20	20	1
S5.1	1	5
S5.10	10	5
S5.20	20	5
S10.1	1	10
S10.10	10	10
S10.20	20	10

2.3. Antifouling Baths

To study the influence of operating conditions on membrane cleaning, four parameters were followed: the washing time, the temperature, the concentration of complexing, agent and the pH.

The complexing agent chosen is the chelating EDTA (ethylene diamine acid tetra-acetic acid): its detergent power is close to that of surfactants. The synthetic solution selected

for this study is S1.1 (1 g/LNaCl+ 1g/L Dye) and the volume of the bath was fixed at 300 mL.

As the permanent hydraulic resistance (R_p) increases after each treatment, we opted to make an evaluation of the effectiveness of each washing procedure by computing the recovery flow obtained over the rate of flow lost during the filtration.

The sequence of tests is summarized in the following table.

Table 2. The composition of antiflouling baths.

Tests	Time (min)	pH	Temperature (°C)	[EDTA]g.L^{-1}
E1	5-10-15-30	7	25~30	0.5
E2	10	9.5-10-10.5-11	25~30	0.5
E3	10	7	25-30-40-50	0.5
E4	10	7	25~30	0.5-1-1.5-2

For each test we have determined:
1) The flow of pure water (J_0) for the clean membrane at a constant pressure ($\Delta P_m = 10\ Bar$).
2) The flow of pure water after filtration of S1.1 (J_t) in constant pressure ($\Delta P_m = 10\ Bar$).
3) The flow of pure water after the washing treatment (J_L) to constant pressure ($\Delta P_m = 10\ Bar$).

The recovery of flux (%) is determined by:

$$RF\% = \frac{J_L - J_t}{J_0 - J_t} \times 100$$

3. Results and Discussions

3.1. Permeability

The investigation of the flux variation with pressure for each synthetic solution enabled the calculation of their relative permeability through the NF membrane. The obtained curves do not pass through the origin, which highlights the effect of significant osmotic pressures.

For all obtained curves, the increase of the dye concentration for a constant concentration of NaCl decreases the permeability.

Table 3. *Effect of the variation of the salt concentration on the permeability.*

Solutions	$L_p(L.h^{-1}.m^{-2}.bar^{-1})$	$\Delta\pi(bar)$
S11	4.27	0.75
S51	3.99	4.75
S101	2.37	8.9

The increase in dye concentration increases the amount of organic material which may block the pores of the membrane by adsorption to the surface and create a (thing that blocks or stops) the passage of the solvent through the membrane.

On the other hand, the important molar mass of dye compared to NaCl hints that the osmotic pressure received is basically caused by salt. Therefore, since the concentration of NaCl is constant, osmotic pressure remains unchanged [8].

The most important is the reduction in flux caused by increasing the osmotic pressure with increasing NaCl concentration. The value of the last thing just mentioned

went from 2 bar to the less stronger/made stronger solution (S1.1) to the highest possible value of about 9 bar of solution (S10.1).

The intensive use of salt in the fabric industry, seems to be a serious problem because it involves the operation of more processes under higher pressures and higher costs [9].

Furthermore, the effect of NaCl concentration on the permeability is very little compared to that of the dye, given the lowest steric characteristics of salt. Its reduction remains important since the value obtained is reduced by almost half in the concentration from the lowest to the highest.

Table 4. *Effect of variation of the dye concentrations on the permeabilities.*

Solution	$L_p(L.h^{-1}.m^{-2}.bar^{-1})$
S11	4.27
S110	2.91
S120	2.57

3.2. Flux

In Figures 1, 2, 3 we represented the evolution of flux over time for different synthetic solutions at a constant pressure equal to 10 Bar. We observe an overall rapid flux decline over time for all tests of membrane filtration, highlighting the clogging phenomenon.

The increase in the dye concentration reduces the flux over time (a decrease of 50% after 2 hours), especially for a low salt concentration (solution S1.1). During the change from 1 to 10 g.L^{-1}, the fall of the stream is more than that reached during the passage of 10 to 20 g.L^{-1}. In fact, with low concentrations and with the gradual decrease in the volume of water in the cell filtration, the dye is deposited slowly on the surface of the membrane. At this level, the membrane resistance is more important than that caused by the cake, then we have achieved a flow stabilizing bearing. The decrease in flux at this stage is low because it is mainly caused by both: the adsorption of organic material (dye) on the surface of the membrane and by the compressibility of the cake, which results in a change of physical properties (porosity, pore diameter, thickness...) and increase in the inherent strength of the cake [10, 11].
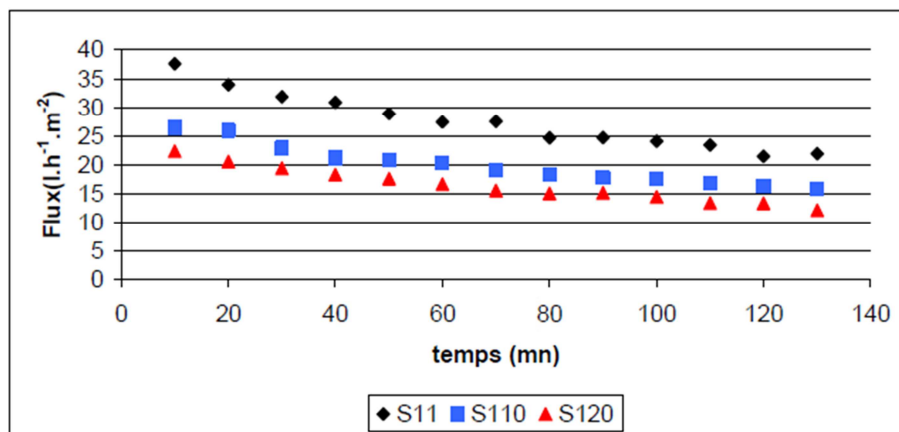

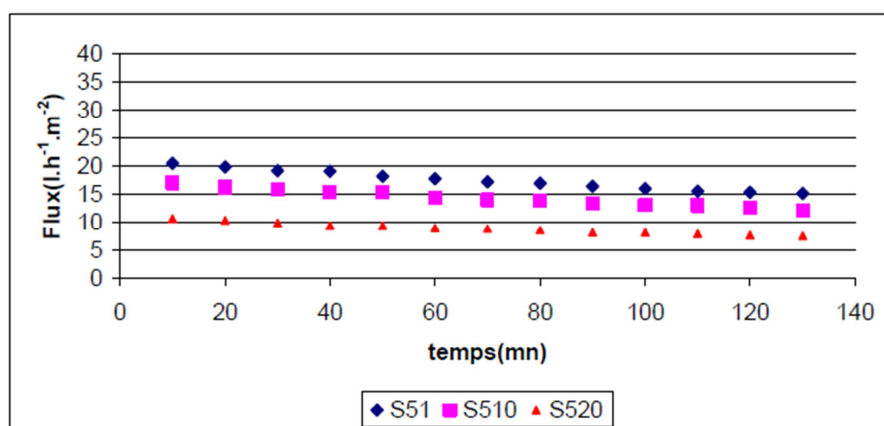**Figure 1.** *Variation of the flux as a function of the concentration of the dye for 1 g.L^{-1} NaCl.*

Figure 2. Variation of the flux as a function of the concentration of the dye for 5 g.L^{-1} NaCl.
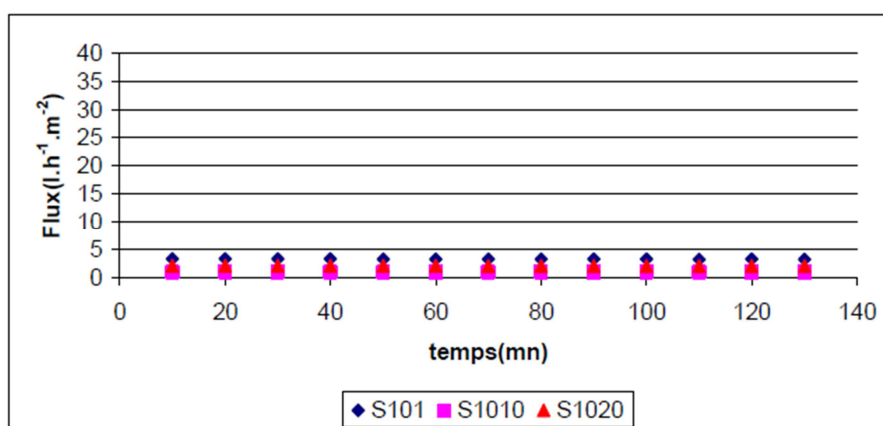

Figure 3. Variation of the flux as a function of the concentration of the dye for 10 g.L^{-1} NaCl.

The effect of the increase in NaCl concentration on the flux decline is more marked than that of the dye. Also, the passage from a concentration of 1 to 10g.L^{-1} of NaCl (dye concentration 1 g.L^{-1}), creates a decrease of 24,22 L.h^{-1}.m^{-2}, while an increase in dye concentration of 1g/L to 20g.L^{-1} (NaCl concentration 1g.L^{-1}) generates a decrease of 11,12 L.h^{-1}.m^{-2}. This is caused mainly by the increase in osmotic pressure in the solution so that electrostatic repulsions occur [12].

We also note that for highly concentrated solutions of electrolytes (S10.x; where x is the concentration of dye (1g.L^{-1}; 10g.L^{-1} or 20g.L^{-1}), the effect of the concentration of the dye on the variation of flux becomes too weak.

This leads us to the conclusion that the salts increase the tendency to clog membranes, not only by providing an additional osmotic pressure but also by changing the physical and chemical structure of the material layer formed on its surface [11, 12].

Large concentrations of electrolytes used in the dye bath for reactive dyes decrease the solubility of the latter and increase its tendency to aggregate, so the effect of the mixture "dye-salt" on clogging membranes depends on the mixing ratio and the nature of the elements that compose it. At low electrolyte concentrations, clogging is caused essentially by the blocking effect and clogging of the pores by the dye. But a high concentration of NaCl, and with the

formation of the gel layer on the surface, clogging is caused by the additional resistance introduced by the new physical structure formed as well as the organic matter adsorbed on the membrane surface and inside the pores [13, 14].

3.3. Comparison of Filtration Models

To better understand the phenomenon of clogging caused by the mixtures of "Dye-salt" the following clogging models were compared.

The first model is the "model of clogging by complete blocking of the pores" (it will be designated by H1). This model interprets the variation of the porosity in the over time.

$$J_P = J_0 \exp\left(-\frac{\alpha_{block}.S.J_0.C_m}{N_0}t\right) \quad (1)$$

The second model is "the standard model of clogging by blocking progressive pore "(it will be designated later by H2). It interprets the change in volume of the pores of the membrane, assumed to be all cylindrical, over time.

$$J_P = J_0\left(1 + \frac{\alpha_{pore}.S.J_0.C_m}{\pi.r_p^2.\delta_m}t\right)^{-2} \quad (2)$$

The third model is "the model of filtration cake" (it will be designated by H3). This model focuses on the properties

colmatantes of the solution to befiltered, and especially the hydraulic resistance caused by the layer of porous gel deposited on the surface of the membrane.

$$J_P = J_0 \left(1 + \frac{2.\,\alpha_{gateau}.J_0.C_m}{R_m} t\right)^{-\frac{1}{2}} \qquad (3)$$
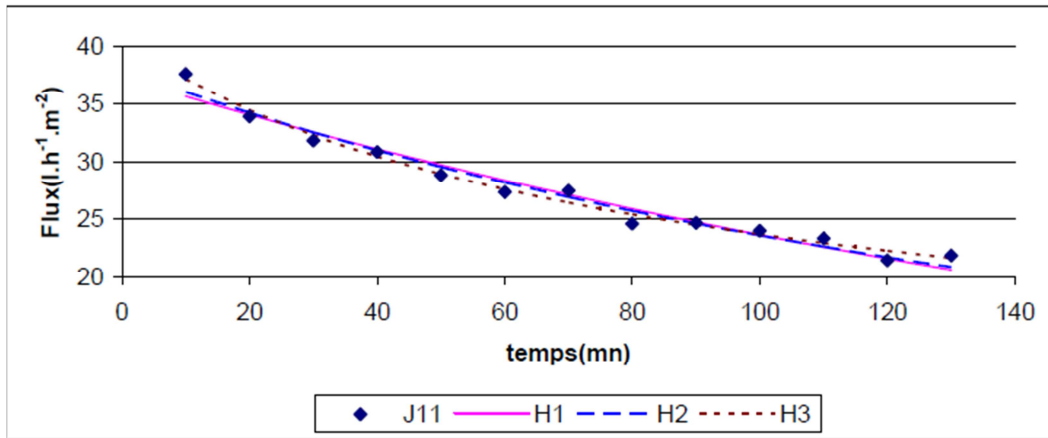

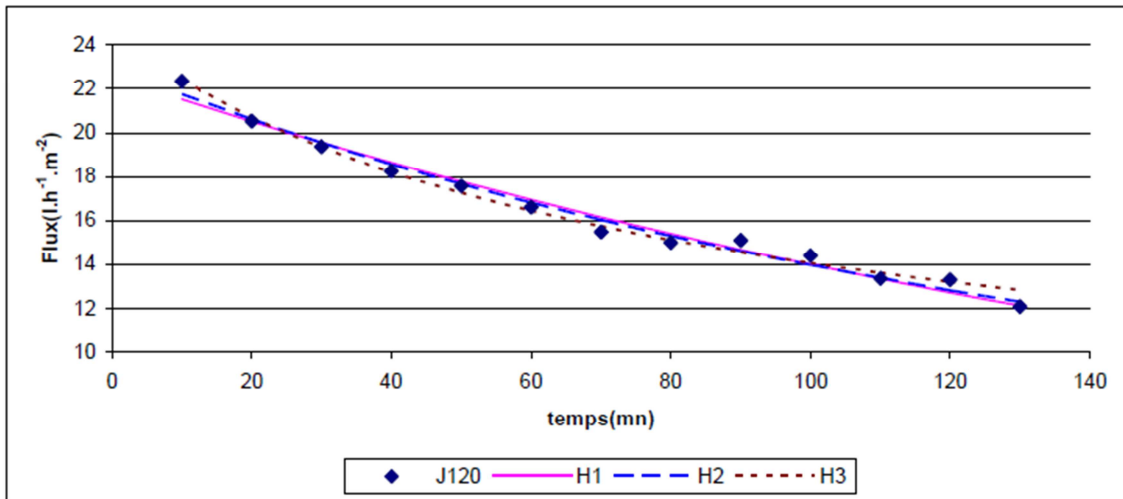***Figure 4.*** *Comparison of clogging models for solution S11.*

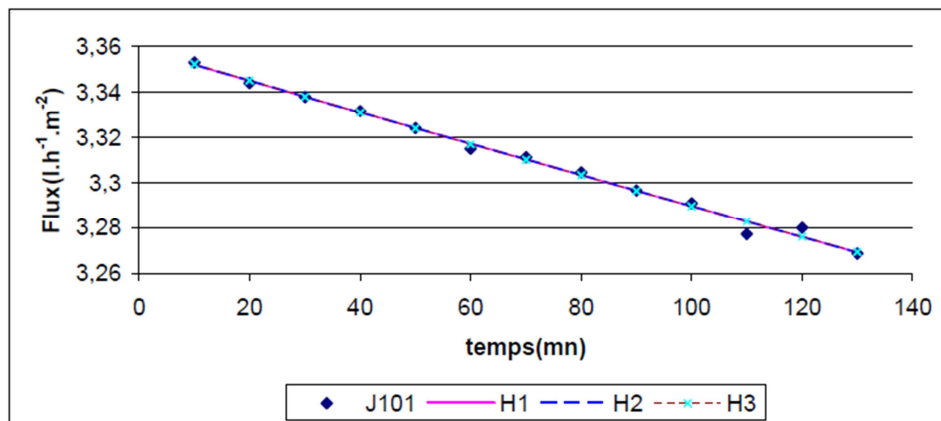***Figure 5.*** *Comparison of clogging models for solution S120.*

Figure 6. *Comparison of clogging models for solution S101.*

We have chosen to compare the flows at J11, J120 and J101 for these three filtration models (Pressure was equal to 10 bar) Figures 4, 5, 6.

We have also noted that for the highly concentrated electrolyte solutions, the curves merge, and this is caused by the small variation of flows in time. So at this level, there is the coexistence of the three theories of cell filtration: complete blockage of the pores; an overlay of the organic

matter within the pores and a gel layer forming on the membrane surface [15, 16].

3.4. Hydraulic Resistances

Clogging is an inevitable drawback of membrane filtration of textile waste. The organic material and the electrolytes form a resistance to additional hydraulic, causing a significant drop in filtration flow, which requires, in part periodic washing treatments.

To be able to choose the adequate treatments of unclogging membranes, we must understand the nature of the hydraulic resistance resulting from the filtration of solutions containing a mixture of "Dye-salt".

Indeed, the hydraulic resistance Total R_T is a result of the own resistance of the membrane R_m and the resistance of clogging $R_c.$

$$R_T = R_m + R_c = R_m + R_t + R_p \qquad (4)$$

R_t: Temporal resistance, which expresses the cake resistance to membrane surface and of electrostatic repulsion force.

R_p: Permanent resistance, which results from adsorption of the organic material to the interior of the pores and the surface of the membrane.

We are therefore interested in this part, which leads to variation in different hydraulic resistances compared to mix proportions "dye-salt". The results obtained are presented in Figure 7.
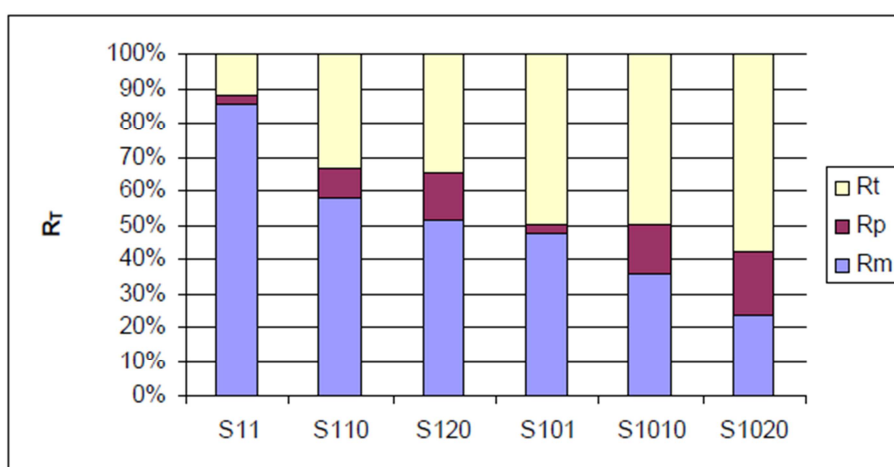
Figure 7. *Total hydraulic resistance (R_T) based on the own membrane resistance (R_m), the temporal resistance (R_m) and constant resistance (R_P).*

For a fixed concentration of NaCl, the participation of permanent resistance R_P increases with the increase in the concentration of dye. This implies that the quantity of material absorbed at the surface of the membrane or embedded in the interior of the pores increases depending on the concentration of used dye [17]. Similarly, at low NaCl concentrations (1 g.L^{-1}) during the progression of a dye concentration from 10 to 20 g.L^{-1}, the supply of temporal resistance remains nearly constant while the supply of permanent resistance increases. This implies that the electrolytes push the system toward the adsorption of organic material. This result is more obvious in the case of S120 and S1020 solutions, where the increase in salt concentration to 20g.L^{-1} of dye, promotes adsorption [18, 19].
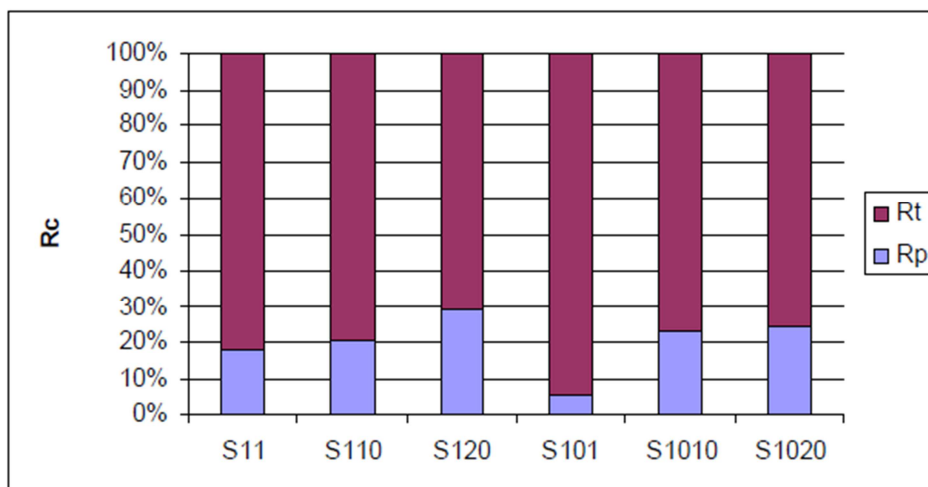
Figure 8. *The resistance of clogging (R_c) as a function of the temporal resistance (R_T) and the permanent resistance (R_P).*

Figure 8 shows that the resistance due to clogging is formed essentially by the temporal resistance provided by the cake layer and the electrostatic repulsion forces.

The increase in NaCl concentration increases R_c (clogging resistance) and it consequently increases the total hydraulic resistance R_T. The NaCl concentration essentially affects temporal resistance. Therefore, the electrolytes participate in R_c clogging resistance by electrostatic repulsion phenomena as well as the modification of the structure of the cake layer formed on the surface of the membrane. This implies that reducing the electrolyte concentration in textile waste, can remove most of the hydraulic resistance, so we should directly address the problem of clogging [20].

3.5. The Index of Clogging I_c

According to the results above, the temporal flow resistance R_t forms the major part of the clogging resistance. This resistance is caused mainly by the electrostatic repulsion and the cake layer formed on the surface of the membrane, so to insulate these two phenomena and study each separately, the clogging index I_c must be calculated [21, 22].

The index of clogging I_c is defined as:

$$I_c = \alpha_{gateau} . C_m \qquad (5)$$

Where α_{gateau} is the specific resistance of the cake and C_m is the material.

We had to determine the I_c (clogging index) for the different synthetic solutions. The following figure 9 gathers the results.
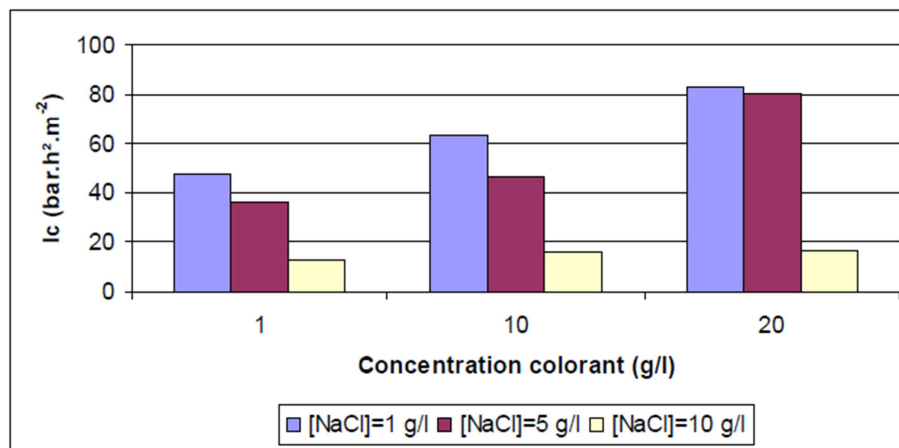

Figure 9. *The index of clogging of the different synthetic solutions.*

Reactive dyes, in their hydrolysed form and in the presence of electrolytes, form organic material aggregates. These aggregates in turn form the cake layer on the surface of the membrane. If we increase the concentration of dye, we increase the amount of organic material and, hence, we obtain a more voluminous structure (Vg increases) and a more compact one (the porosity decreases). This explains the increase of I_C as a function of dye concentration [22, 23].

However, for highly concentrated electrolyte synthetic solutions (10 g.L^{-1} NaCl), I_c remains almost unchanged depending on the concentration of the dye. This implies that the structure formed on the membrane surface is not influenced by the addition of organic material. Therefore, the temporal clogging effect R_t is increased in these conditions by electrostatic repulsion forces [16, 11]. The increase in salt concentration decreases the solubility of the dye; it then seeks another equilibrium state: by aggregation, so aggregate particles increase in size, by adsorption on the surface of the membrane or inside the pores, which consequently increases the permanent resistance R_P. The larged-size aggregate particles form between them a larger interstitial space, which reduces the specific resistance of the cake. As a result, I_c decreases as the salt concentration of the same dye concentration increase [23, 24].

For low salt concentrations, a cake layer is formed by low-sized aggregate particles, so the resulting structure is compact and has a greater specific strength. This resistance is increased by the addition of organic material to the mixture. In contrast, if the salt concentration is increased, the system pushes the formation of larger aggregates of particles, the structure formed becomes spongy and the specific cake resistance decreases [25].

3.6. Study of the Operational Conditions Affecting the Membrane Sealing

3.6.1. Washing Time Effect

We chose to vary the washing treatment time of the membrane after filtration of the S11 solution for 1 hour. The results are illustrated in the following figure 10.
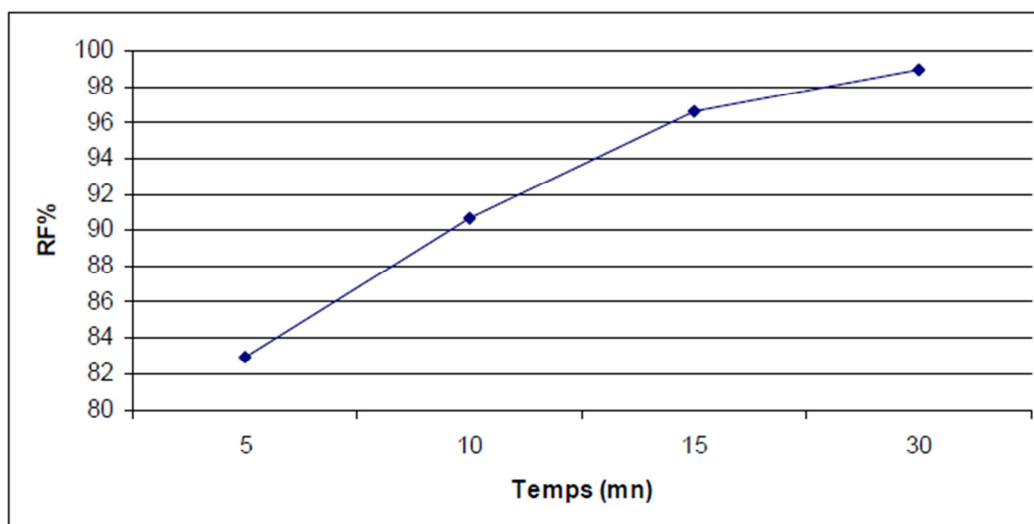
Figure 10. Effect of the washing time over RF%.

Figure 10 clearly shows that by extending the time of washing treatment, loss of stream is recovered, so it reduces the effect of continuing resistance R_p. However, the effect of time is less accentuated after 15 minutes despite its approach to the initial conditions of the membrane after 30 minutes of treatment. Indeed, the more we increase the time of treatment, the more we increase the power of the wash bath detergent and allow better elimination of adsorbed organic material.
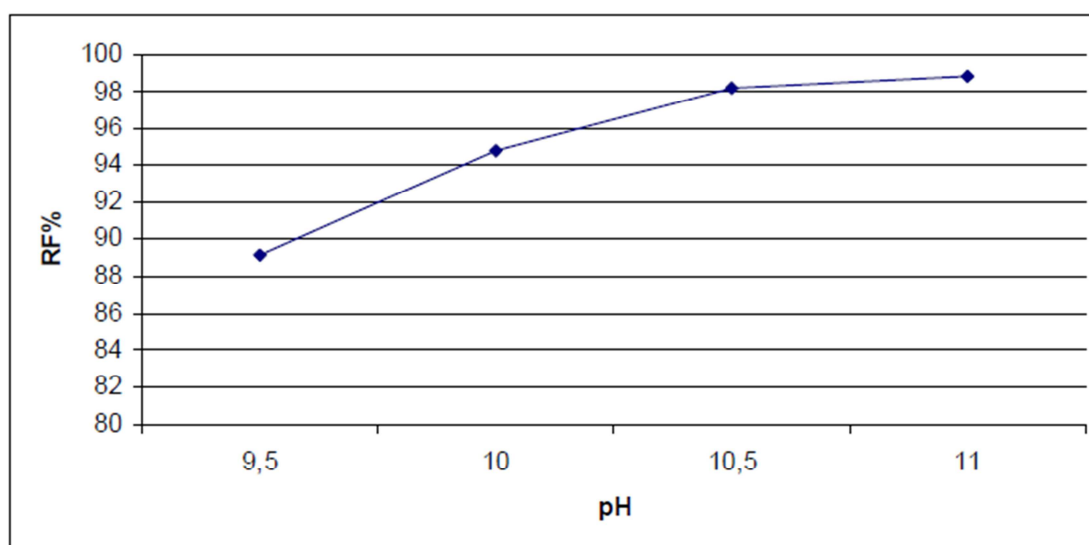
3.6.2. pH Washing Effect

Figure 11. Effect of pH on RF%.

The recovery of flows increases for basic pH to 10.5 but the trend is less pronounced for a pH of 11. More than a pH of 10.5 effects on RF% becomes less obvious. In fact, the basic medium increases the activity of surfactants and detergent generates a power capable of removing organic material [26, 27].

3.6.3. Effect of the Concentration of Complexing EDTA

Figure 12 notes that by increasing the concentration of EDTA the flow recovery increases accordingly, but the shape of the curve indicates that starting from $2g.L^{-1}$ the effect becomes less pronounced. This indicates that the unclogging effect depends essentially on the nature of the complexing agent rather than its concentration since even large amount RF% evolves weakly. EDTA improves the retention of the particles and organic materials of aggregates in the washing liquor, which decreases their solubility and increases their steric effect, thus facilitating their elimination by a simple rinsing [26].
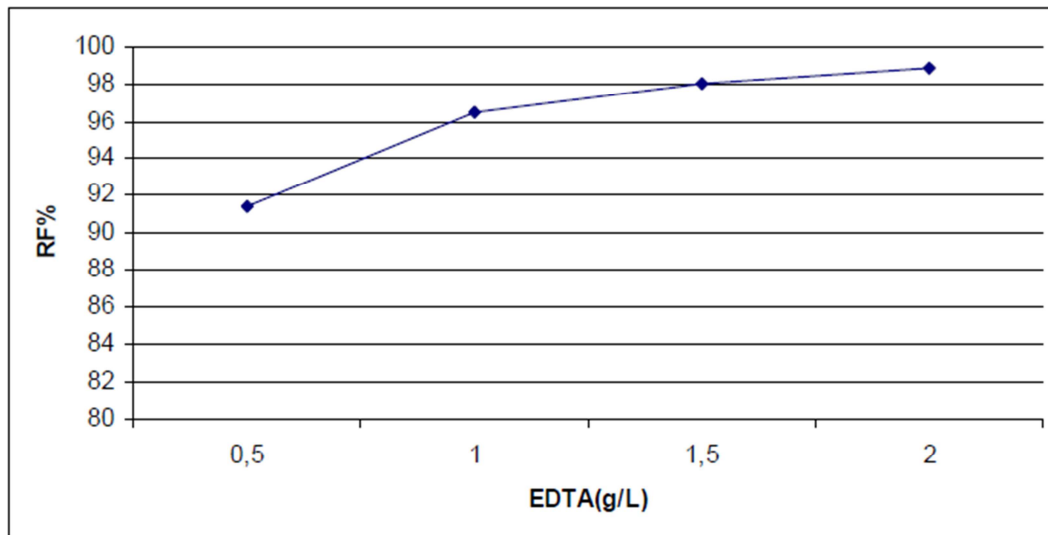
Figure 12. *Effect of the concentration of EDTA at RF%.*
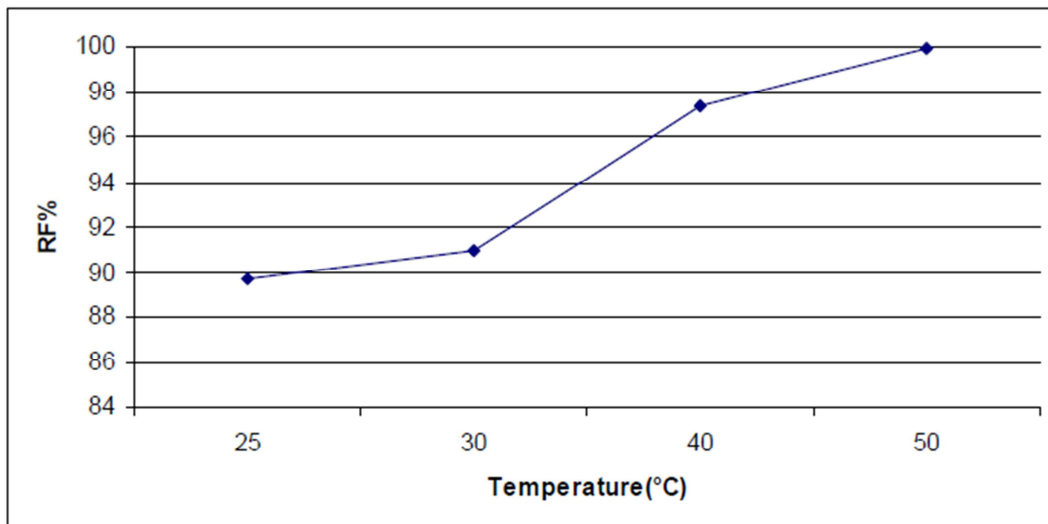
3.6.4. Effect of Temperature

Figure 13. *Effect of temperature at RF.*

The increase in temperature; stimulates the solubility of chemical entities and forces them to leave the surface of the membrane to dissolve in the unclogging bath. So temperature is a key factor that must be controlled in order to eliminate the maximum of permanent hydraulic resistance [28].

4. Conclusion

Due to the serious problems of pollution caused by discharges of textiles, the treatment processes of releases have experienced a significant progress thanks to the membrane techniques, including the NF membrane, which allow recycling water and enhancing the sub products. But the major drawback of the membrane is the clogging by discharges containing a mixture of "dye-salt" which causes a loss of flow.

We conclude from this study that the rejection of the composition plays a key role in the results achieved by the end filtration of textile waste. In fact, at low salt concentrations, organic substances (dye) pass through three different physical states during filtration. First, we observe material deposition on the membrane's surface; this step evolves over time and does not produce significant hydraulic resistance. Then there is a peace meal formation a sponge cake layer whose physical characteristics are determined by the composition of rejection, especially by electrolytes, which decrease the solubility of dyes and push them to the aggregation. The amount of electrolyte present in the discharge determines the dye aggregate size and hence the specific flow resistance of the cake layer.

The organic substances present in textile discharge promote the formation of a gel layer on the surface of the membrane; the characteristics of this layer are conditioned by the composition of waste, including electrolytes.

In addition, we have noticed that the clogging phenomenon, in the case of textile waste is mainly described by the filter cake on the model we chose to analyze the clogging index I_c.

According to the unclogging tests, we have determined that operating conditions influence directly the effectiveness of treatment, including washing time and pH. But the most influential factor is the temperature (50°C) afforded an almost complete recovery of the flow. In similar way, the presence of a surfactant complexing agent in the unclogging bath gives very satisfactory results, allowing the membrane to return substantially to its initial state.

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