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Performances of Continuous Adsorption-Ultrafiltration Hybrid Process for AO7 Dye Removal from Aqueous Solution and Real Textile Wastewater Treatment

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Abstract

A hybrid treatment combining the adsorption by PAC with Ultrafiltration (PAC-UF) was applied to the color removal from Acid Orange 7 (AO7) aqueous solutions. The effect of different parameters on color removal and permeate flux evolution such as dose of PAC, pH, TMP and adding of surfactants, was studied. The best operating conditions for a total removal of color were 3 bar TMP, 5 pH, and dose of PAC beyond 150 mg/L in presence of cationic surfactant. A constant permeate flux was observed for all the tests. The evolution of the permeate flux with time can revealed the formation of a PAC-dyes layer due to the retention of these particles by UF membrane. The AO7 not adsorbed by PAC cannot be removed by UF alone. The application of hybrid treatment reduced the UF membrane fouling and the dose of PAC. Permeate flux was then increased from 340 L/h.m² (only UF) to 480 L/h.m² (PAC-UF) and PAC dose was reduced from 300 mg/L to 150 mg/L when adsorption followed by UF treatment applied in two stages was changed to a single stage PAC-UF hybrid process. Applied to real waste water treatment coming from textile company using reactive dyes, the results with the continuous hybrid adsorption/UF over a period of three hours were in agreement with those obtained with the aqueous dyeing solution in terms of permeate flux and color removal behavior. In this case, a quasi-stabilize permeate flux of an average value of 450 L/h.m² and a color removal of 97% were recorded.

Abbreviations PAC: Powder Activated Carbon; PAC-UF: Powder Activated Carbon - Ultrafiltration; AO7: Acid Orange 7; C_0: Initial Dye Concentration; C_p: Concentration in the Permeate; Da: Dalton; UF: Ultrafiltration; Jw: Water Permeate Flux; Lp: Water Permeability; MB: Methylene Blue; TMP: Transmembrane Pressure

Introduction

Currently, the chemical pollution of water is one the big challenges. The presence of micro pollutants such as dyes in the aquatic environment can reduce the penetration of light into water and subsequent photosynthesis and dissolved oxygen, thereby endangering the aquatic organism and microorganism's life. Moreover, dyes are toxic, carcinogenic and persistent with classical physicochemical and biological treatment.

AO7 is a synthetic organic dye that is considered to be a persistent substance because of its complex structure [1]. It is common in many dyes applications and is widely used in the textile industry.

In the sector of textile industry, huge amount of water are required during the different stages of textile processing with dyeing, washing, bleaching, etc. [2].

Physical, chemical, and biological processes are usually used for the treatment of dyeing effluent. Biological treatment such as activated sludge resulted in the removal of COD but has little effect on the color because of the biological persistence of the dye. Moreover, large areas and long retention time are generally required [3]. Coagulation-Flocculation can be an efficient process for the treatment of textile wastewater effluent, but it generates large amounts of sludge due to the addition of chemicals which increases the treatment cost [4].

In the recent years, the potential of the membrane technology to produce high quality effluent has led to its growing applications. Combination of membrane filtration process with other separation operations such as adsorption, coagulation- flocculation, ions exchange, in hybrid systems has many benefits and improvement in terms of enhancing produced water quality, reduction of membrane fouling, environmental friendless and reduction in the capital and operating costs [5].

The hybrid adsorption-membrane filtration process can constitute an effective solution providing totally discolored water [6-8]. The color removal efficiency can be controlled by varying adsorbent dosage, the type of the adsorbent and the addition of the membrane separation process [9,10]. Many adsorbent materials are available for color removal from dyeing waste water treatment. However PAC is among the most popular. Many techniques using PAC as adsorbent have been developed. In particular, hybrid adsorption/membrane separation was found as an effective method to enhance adsorption capacity and treated water quality. For example, the hybrid adsorption-UF process applied to remove small organic pollutants showed good results in terms of fouling reduction and enhancement of permeation flux at the optimal PAC dose [11,12].

In addition, Juang et al. [13] reported that increasing PAC concentration in hybrid adsorption-UF process leads to the reduction of the permeate flux which can be enhanced by an increase of the cross flow velocity and transmembrane pressure.

On the other hand, Banat et al. [14] evaluated the effect of AC adsorbent particle size on the effectiveness of hybrid Adsorption-UF process applied to MB color removal. It was found that the same optimal doses were obtained for both adsorbents. However, the best results in terms of permeate flux and color retention was observed with the lowest adsorbent particle size.

Tansakul et al. [15] studied the effect of adsorbent surface area (PPAC1 and PPAC2) in hybrid adsorption-UF mode applied as pretreatment to reverse osmosis applied to seawater desalination. They found that the increase of carbon dose increases the retention of organic matter in presence of PPAC1 and PPAC2. Therefore, the use of hybrid treatment leads to the limitation of fouling due to PPAC adsorption and then to the increase of the permeate flux in comparison with the use of simple UF.

Yanan et al. [16] investigated the removal of orange G dye for dyeing solution by activated carbon (PAC) adsorption-UF hybrid process. The results show that under optimum hydrodynamic conditions, total dye retention reaching was observed and that UF permeation flux was slightly reduced by the increase of PAC dose, but remains higher than that of conventional nanofiltration membranes under similar conditions.

Mozia et al. [17] applied the adsorption–UF (PAC-UF) system to remove humic substances using three polymeric membranes with different surface properties. The results shows that the use of hydrophobic membrane leads to a stabilize flux resulting from adsorption phenomena whereas, with both other membranes, the interaction of the humic substances with the membrane surface causes a decrease of permeate flux due to fouling.

Therefore, based on previous studies showing very diverse results on different effluents, there is a need to conduct further studies on the effect of adsorbent addition on the membrane separation performances during continuous adsorption-membrane separation hybrid process.

The aim of this novel study is the application of continuous adsorption - UF (PAC-UF) hybrid process for AO7 color removal. Activated carbon (PAC) as adsorbent was selected due to its wide utilization in the industrial scale. The effect of the PAC dose, pH, TMP, on the permeate flux and color removal was determined in presence of AO7 dye aqueous solution which was used as a representative reactive dye.

The continuous adsorption-UF was also considered under the optimum conditions for real dyeing waste water generated from a textile company which uses reactive dye. The effect of different conditions on the system performances in terms of permeate flux and dye removal was examined.

Experimental

Materials

Powder activated carbon (PAC): The activated carbon was purchased from Norit (USA) and its characteristics are listed in Table 1 (data provided by the manufacturer).

Density (g/ml)	0.37
Molar mass (g/mol)	180
Particle size (µm)	37
Surface area (m²/g)	1400

Table 1: Characteristics of PAC.

Acid Orange 7 (AO7): The characteristics of the anionic acid orange 7 (AO7) provided by SIGMA ADRICH (France) were given in Table 2. The molecular structure was presented in Figure 1.

Molecular formula	$C_{16}H_{11}N_2NaO_4S$
Molar mass (g/mol)	350
Zeta potential (mv)	-15.3
λ_{max} (nm)	483

Table 2: Characteristics of AO7.

Figure 1: Molecular structure of AO7.
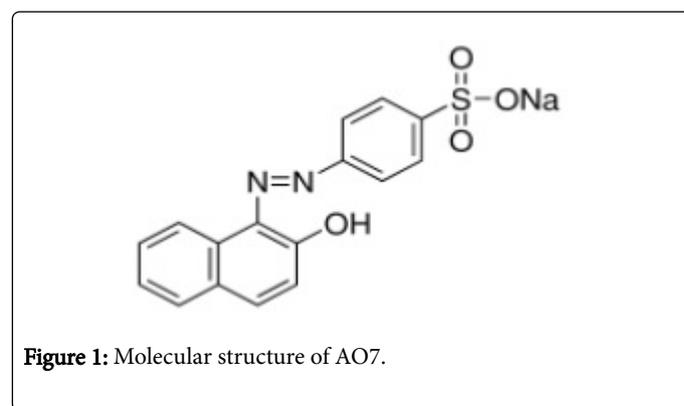
Real textile effluent: The effluent considered in this study contained a mixture of 3 reactive dyes with different percentages, coming from the dyeing operation. Their chemical formulas and concentrations were summarized in Table 3. The characteristics of the studied effluent were summarized in Table 4.

Experimental set-up and procedure

The schematic of laboratory scale adsorption-UF hybrid system is illustrated in Figure 2. The unit was equipped with a feed tank, control valves, pressure gauges and a feed pump.

The membrane module contains single channel ceramic tubular membrane (Novasep, France) of 150 KDa Cut-off, having the following characteristics: 40 cm length, 6 mm diameter, $7.635 \ 10^{-2} \ m^2$ area and 200 L/m²h.bar as water permeability.

Dye	Formula	Concentration (g/l)
Reactive yellow 145	$C_{28}4H_{20}ClN_9O_{16}S_5Na_4$	1.33
Reactive red 120	$C_{44}4H_{24}Cl_2N_{14}O_{20}SNa_6$	3.68
Reactive blue 222	$C_{37}H_{23}ClN_{10}O_{22}S_7Na_6$	1.24

Table 3: Composition of the dyeing effluent.

Hybrid PAC/UF process: In the hybrid process (Figure 2), the feed volume was maintained constant by operating as follows: the feed reactor was continuously feed from a reservoir filled with a mixture of

AO7 solution and PAC adsorbent. The volume added in the feed tank (V_f) is equal to the volume of the permeate extracted (V_p). The adsorption process was performed in the fed reactor.
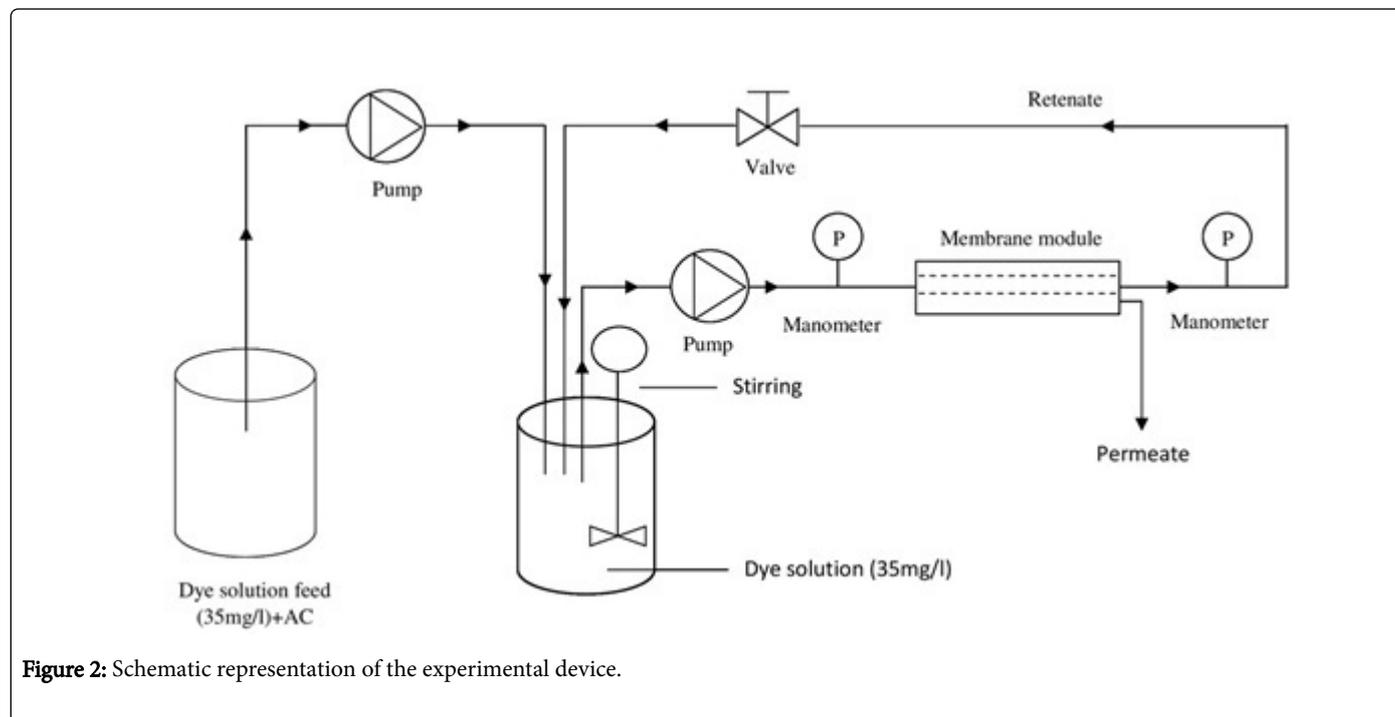
Figure 2: Schematic representation of the experimental device.

The temperature of the feed solution was maintained constant using a heat exchanger. The pH of the solution was adjusted using acidic (HCl 0.1 M) or basic (NaOH 0.1 M) solutions.

To respect the same conditions than those in the presence of real dyeing waste water effluent, three different surfactants having different functional charges, dodecyl trimethylammonium bromide C12 TAB (cationic surfactant), sodium dodecyl sulfate SDS (anionic surfactant) and brij 35 (nonionic surfactant) were added in the feed tank already containing the suspension forming by PAC- dyeing solution (Tables 4 and 5). These surfactants were used at their critical micelle concentration [18-20].

pH	10.41
ABS	8.24
COD	2.36 g/L
Conductivity	46.28 mS/cm

Table 4: Characteristics of the real effluent.

Surfactant	CMC (mM)
C12TAB	15.2
SDS	8
Brij 35	0.009

Table 5: Characteristics of surfactants.

Cleaning and regeneration of the membrane: The membrane water permeability (Figure 3) is a very important parameter to determine the performance of the membrane. The water permeability was achieved using deonized water. According to Darcy's law, a linear increase of the permeate flux with water should be obtained:

Lp=Jw/TMP

Where:

Jw=water permeate flux (L/h.m^2)

TMP=transmembrane pressure (bar)

Lp=water permeability (L/h.m^2.bar $^{-1}$)
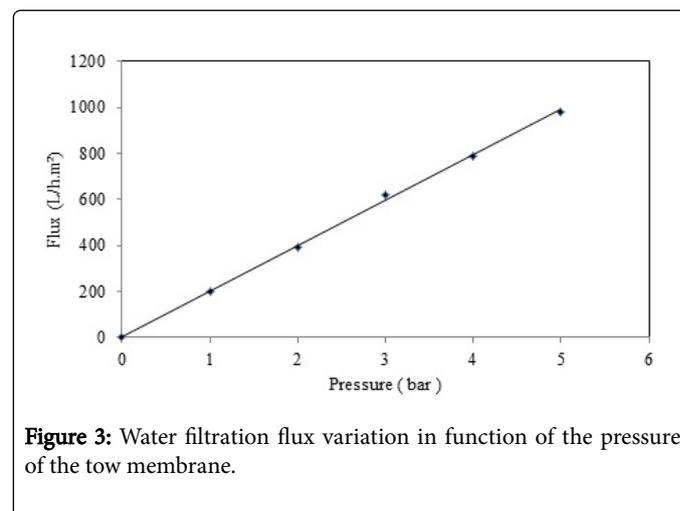
Figure 3: Water filtration flux variation in function of the pressure of the tow membrane.

Fouling is a result of the accumulation of particles on the membrane surface leading to a decrease in the permeate flux. To overcome this phenomenon, a chemical cleaning of the membrane is generally

applied. In our case the membrane regeneration was achieved by a simple rinsing with deionised water at a pressure of 2-3 bar for a period between 20 and 30 min, until the permeate flux at 1 bar matches almost the permeability of the clean membrane (200 L/h.m^2.bar^{-1}).

Process performances

The experiments were carried out at various TMP, pH values and PAC dose. The TMPs was controlled in the range 1-3 bar and the feed pH was adjusted to be in the range 5-9 using acidic (HCl 0.1 M) or basic (NaOH 0.1 M) solutions. The adsorbent (PAC) dose was adjusted to be between 50 and 300 mgL^{-1} in the feed tank.

All tests were conducted in duplicate. The permeate flux was measured every 10 min.

Decolorisation study

The dosage of the color was achieved with UV-visible spectrophotometer at 483 nm wavelength. The concentration is determined by the law of Beer Lambert. A=ε.l.C where A and C are the absorbance and concentration respectively. The color removal was determined by the following equation:

R (%)=(1–C_p/C_0) × 100

Where C_0 and C_p are respectively the initial concentration of dye before the addition of the adsorbent in the feed tank and the concentration in the permeate.

Results and Discussion

Effect of PAC dose

From Figure 4, it is worth noting that the permeate flux decreases drastically from 530 L/h.m^2 to 330 L/h.m^2 after 60 min of filtration when simple UF was applied. However with hybrid treatment, flux stabilization can be observed whatever the PAC dose used (50 mg/L–300 mg/L). The stabilized flux was of 450 L/h.m^2 for PAC dose between 50 mg/L and 100 mg/L and increased to 480 L/h.m^2 for PAC doses between 150 mg/L and 300 mg/L.
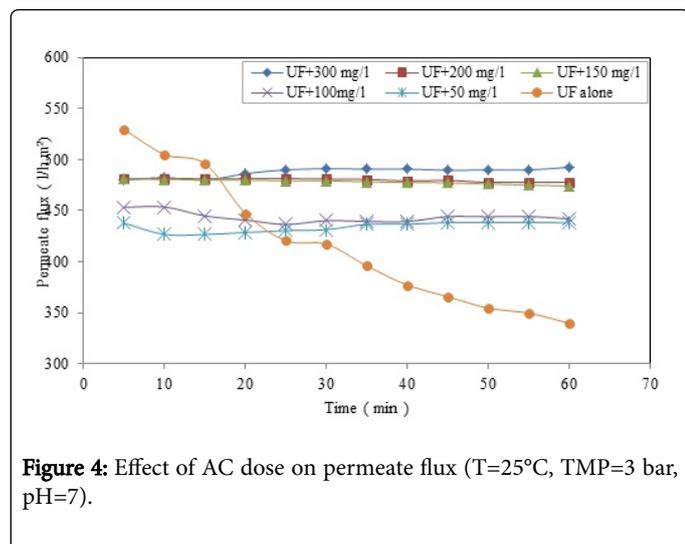
Figure 4: Effect of AC dose on permeate flux (T=25°C, TMP=3 bar, pH=7).

In simple UF process, the permeate flux decreased drastically since the first minutes of filtration because the size of dye molecules of 300 Da is very smaller than UF membrane weight cut-off of 150 KDa, which induce membrane fouling due to the pore clogging by AO7 molecules in addition to the possibility of the interaction with the membrane surface or into the pores [21].

The application of hybrid AC-UF process shows two behaviors:

- A slight decrease of the permeate flux during the first minutes of filtration and then stabilization at 440 L/h.m² for PAC doses between 50 mg/L and 100 mg/L.
- A stabilized flux obtained since the first minutes of filtration at 480 L/h.m² for PAC doses between 150 mg/L and 300 mg/L.

The role of PAC suspension during the hybrid treatment is to retain the dye molecules that cannot be removed by a simple UF membrane (Figure 5).
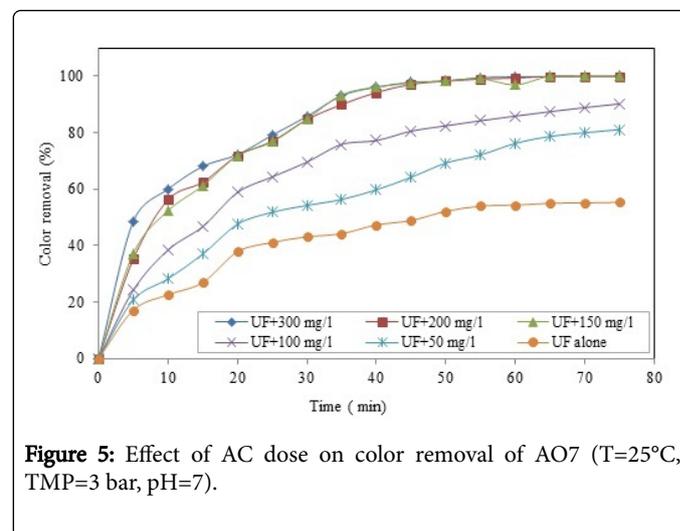
Figure 5: Effect of AC dose on color removal of AO7 (T=25°C, TMP=3 bar, pH=7).

When the feed solution is stirred in presence of PAC, the adsorption of dye molecules by the PAC in the stirred reservoir reduces membrane fouling by preventing interactions between membrane surface and dye molecules which lead to the increase of the stabilized permeate flux [22].

For a relatively high dose of adsorbent, 150-300 mgL^{-1}, similar quantitative performances were noticed indicating that for a dose of 150 mg/L, the deposition of PAC particles might reach equilibrium.

This result shows that the increase of PAC dose beyond 150 mg/L did not lead to the increase of the adsorptive PAC-layer thickness (filter cake) deposited on the membrane surface but crush surface and detach the cake layer, therefore the membrane fouling reduced and permeate flux is enhanced.

The PAC doses below 150 mg/L, i.e., between 50 and 100 mg/L are not sufficient to adsorb enough dye molecules, consequently, interactions between membrane surface and dye molecules can happen.

The separation performances in term of molecules dyes retention are illustrated by the Figure 6. The color removal increases with time for hybrid and UF treatment to stabilize after almost 60 min for all cases at 55% when applying a simple UF and 80%, 90% and 100% for hybrid treatment with PAC dose of 50 mg/L, 100 mg/L and beyond 150 mg/L respectively.
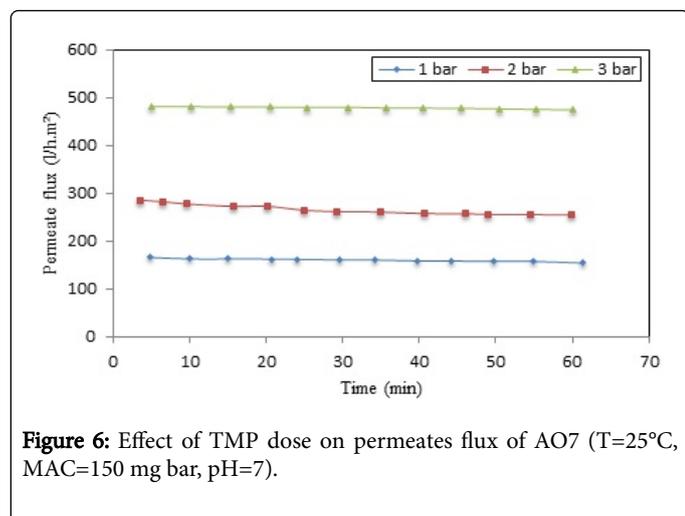
Figure 6: Effect of TMP dose on permeates flux of AO7 (T=25°C, MAC=150 mg bar, pH=7).

The significant increase of color retention in presence of hybrid treatment from 55% (simple UF) to beyond 80% may be attributed to the availability of the adsorptive sites located on the PAC surface which increase in the retentate due to the increase of PAC dose. In addition, when the dye molecules go through the PAC cake layer deposited on the membrane surface, the rapid dye molecules binding due to adsorption ensures the high percentage of color removal.

As indicated previously, the total color removal with PAC doses between 150 mg/L and 300 mg/L indicates the establishment of equilibrium layer formed by PAC particles deposition on the membrane for PAC doses starting from 150 mg/L.

Effect of transmembrane pressure

Figure 7 shows the variation of color removal with time at different transmembrane pressure. It can be observed an increase of color retention with time to obtain a complete dye removal after 160 min, 80 min and 55 min at a pressure of 1 bar, 2 bar and 3 bar respectively. TMP is the driving force in the process, so the increase of TMP also drives more PAC particles to deposit on the membrane surface. Consequently, the time needed to form PAC cake layer will be shorter under higher TMP.
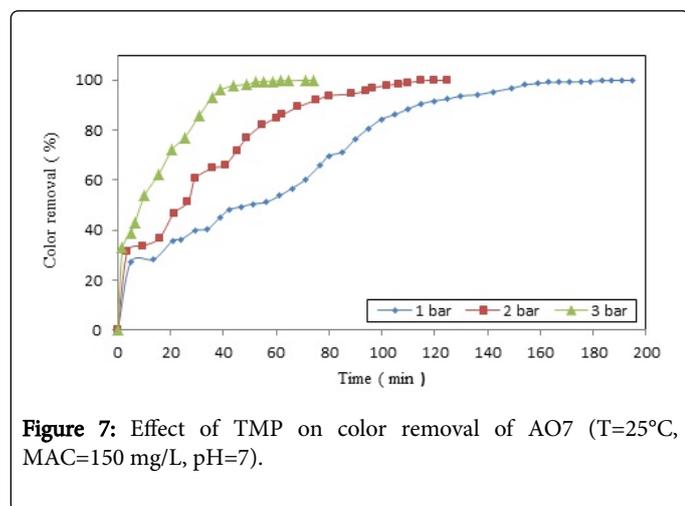
Figure 7: Effect of TMP on color removal of AO7 (T=25°C, MAC=150 mg/L, pH=7).

From Figure 8, it can be observed a quasi-stabilization of permeate flux at the different studied TMP. The increase of the TMP from 1 bar to 3 bar leads to the increase of the permeate flux from 160 L/h.m² to 480 L/h.m². This can be attributed to the convective transfer leading to the increase of the permeate flux with the increase of the TMP.
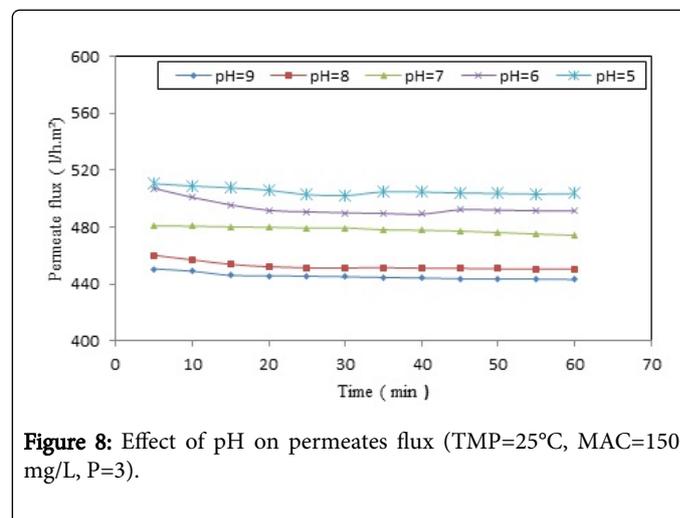
Figure 8: Effect of pH on permeates flux (TMP=25°C, MAC=150 mg/L, P=3).

Similar phenomenon was also reported by other researchers using powder activated carbon. So, the increase of TMP has a positive effect on membrane permeation flux and on color retention.

Effect of pH

From Figures 9 and 10, it can be shown that the UF behaviour from pH 5 to 9 is similar. The stabilized permeate flux increased when pH decreased. The highest flux of 510 L/h.m² was obtained at pH 5 and the lowest corresponding to a value of 440 L/h .m2 was obtained at pH 9.
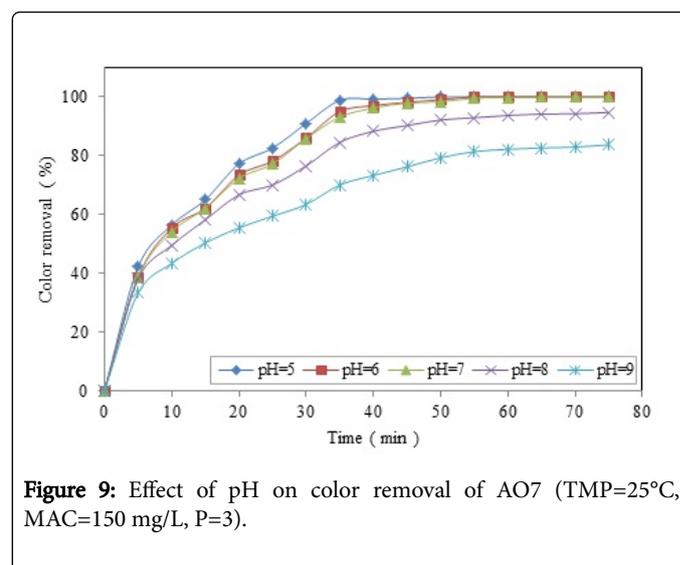
Figure 9: Effect of pH on color removal of AO7 (TMP=25°C, MAC=150 mg/L, P=3).
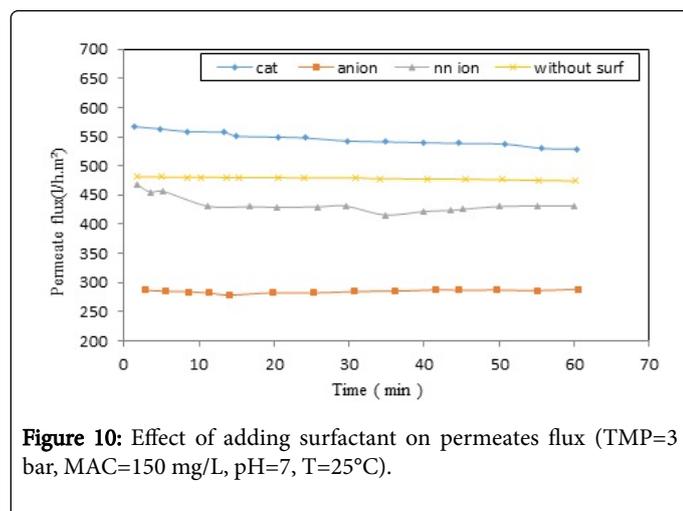
Figure 10: Effect of adding surfactant on permeates flux (TMP=3 bar, MAC=150 mg/L, pH=7, T=25°C).

As given by Figure 10, the AO7 retention increases with time to reach a stabilized value of 80% and 90% after 60 min of filtration for pH 9 and pH 8 respectively. The total color retention was obtained after 40 min of filtration within pH range 5-7.

Above all, for the purpose of the neutrality of the medium, a pH range 6-7 should be taken into consideration.

According to Masmoudi et al. [23], the ceramic membrane can be characterized by the isoelectric point (IEP) which has a value of almost 7. Considering the pH value of the dye solution during ultrafiltration, ceramic membrane is charged depending on this pH value.

Within the solution range pH=7-9, the surface of membrane is negatively charged whereas within the pH range 5-6, the membrane is positively charged.

On another hand, the dye retention by activated carbon at basic or acidic conditions can be attributed to electrostatic mechanism or hydrophobic–hydrophobic mechanism [24]. According to Al-Degs et al. [25], it was found that the adsorption capacity of activated carbon for reactive dye was higher in acidic rather than in basic solution. Therefore, the decrease in AO7 removal by UF membrane at higher pH (7 to 9) can be explained by the magnitude of the electric repulsion or attraction between the membrane surface and the PAC particles retained by the membrane, or the aggregate formed by reactive dye in solution taking into account that the membrane is negatively charged beyond its isoelectric point (pH=7), neutral at pH=7 and positively charged for pH lower that 7 [26,27]. The groups AO7/PAC whose formation are promoted in acidic solution, are more retaining by the membrane and allow a reduce of the problem of membrane fouling which explains the higher permeate flux obtained in comparison with that in basic solution.

Effect of surfactant addition

The diversity of additives contained in the dyeing effluent can have an effect on different interactions that can enhance or reduce the continuous hybrid process PAC//UF. The presences of anionic surfactant in the colored solution leads to a decrease of the stabilized permeate flux from 480 to 275 L/h.m^2. However, an increase of the permeate flux to 550 L/h.m^2 was shown in the presence of cationic surfactant and a slight decrease was obtained with nonionic surfactant from 480 to 440 L/h.m^2 (Figure 10). It is worth noting that under the

study conditions (without surfactant addition) no decrease in flux was observed. This statement can indicate that the size of particles formed by the species surfactant-PAC-dyes, retained by UF membrane, remains greater than the mean pore radius of the membrane. The presence of cationic surfactant in PAC/ UF process improves the attractive force between the PAC layer positively charged in presence of cationic surfactant and the anionic dye. The increase of electrostatic interactions under the effect of cationic surfactant enhanced the fixation of AO7 molecules, which leads to the increase of color removal and the improve of permeate flux [28].

When adding an anionic surfactant, the retention of AO7 dye decreases by about 50% (Figure 11). This behavior can be attributed to the electrostatic repulsion between the negatively charged PAC layer formed on the membrane under the effect of the charge of the anionic surfactant and the anionic dye. AO7 molecules that are not fixed pass then through the membrane resulting in a decrease of color removal. A compaction of the PAC layer formed on the membrane surface in presence of anionic surfactant, can explain the decrease of the permeate flux [29].
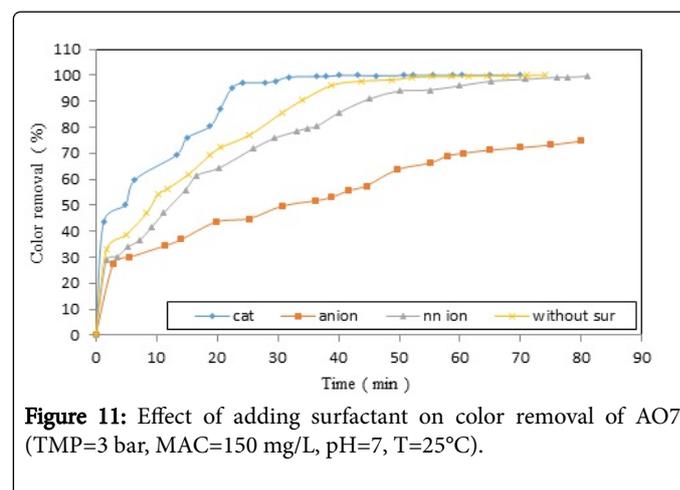
Figure 11: Effect of adding surfactant on color removal of AO7 (TMP=3 bar, MAC=150 mg/L, pH=7, T=25°C).

The non-ionic surfactant causes a slight decrease in the permeate flux and color removal. In this case, the electrostatic interaction is not involved. So, this is due to satirically hindered or to competition between AO7 molecules and the nonionic surfactant during fixation on the PAC layer formed on the membrane surface.

Application on textile effluent

Study of permeate flux: Figure 12 shows the variation of permeate flux of real waste water under both configurations, continuous PAC-UF and simple UF. The AC dose used is 150 mgL^{-1} which represents the optimal value for AO7 aqueous solution. The permeate flux behavior observed is similar to that obtained with the colored aqueous solution. The permeate flux is almost stable using the hybrid process (460 L/h.m^2-400 L/h.m^2) whereas a dramatic decrease of flux from 520 L/h.m^2 to 90 L/h.m^2 was shown when simple UF was applied. This decrease of flux is due to the membrane fouling caused in major part by the pore clogging by the particles having similar or lower size than the mean membrane pore diameter [21]. In hybrid process, the stabilization of permeate flux is due to the limitation of membrane fouling during the period of run. The retention of AC adsorbent by the membrane during filtration leads to the deposition of a porous layer on the membrane surface acting as a dynamic secondary membrane

which retains any substances contained in the real effluent that not could be adsorbed in the feed tank [22].
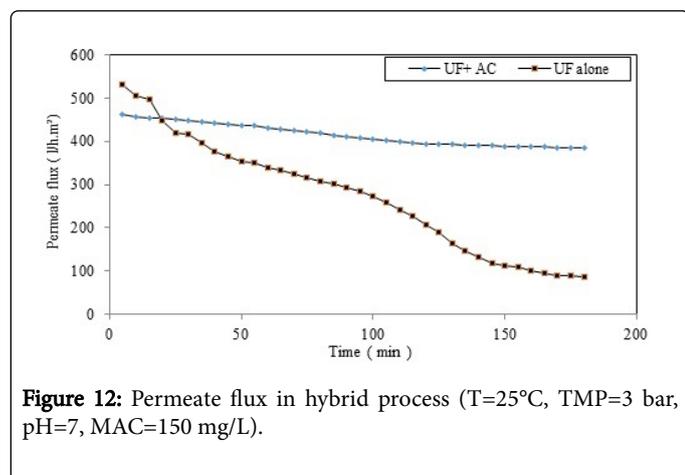
Figure 12: Permeate flux in hybrid process (T=25°C, TMP=3 bar, pH=7, MAC=150 mg/L).

Study of pollutants removal: Figure 13 shows the kinetic of the pollutants retention in terms of colour, COD and conductivity. It can be observed that the removal of the pollutants increased with time to reach quasi-stabilization after 180 min at 97% for colour, 70% for COD and 30% for conductivity. This result confirms our previous explanation about the built of AC layer on the membrane surface. The quasi-stabilization of the pollutants retention is due to the saturation of AC active sites [15].
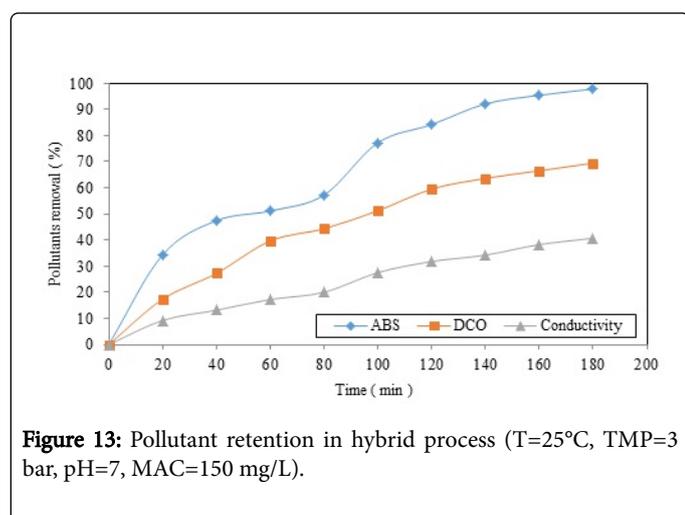
Figure 13: Pollutant retention in hybrid process (T=25°C, TMP=3 bar, pH=7, MAC=150 mg/L).

Membrane morphology: To determine the origin of fouling, Scanning Electron Microscopy (SEM, Zeiss MERLIN) photographs of membrane were taken before and after filtration treatment of real textile effluent using simple UF and continuous hybrid PAC-UF. For the study of the fouling effect on membrane morphology, the examined membrane was taken after complete filtration operation.

Figure 14 which represents the overall morphology and the cross-sectional of the membrane revealed three distinct corresponding morphologies.

Figure 14a shows a typical surface and cross section view of asymmetric structure of virgin ceramic membrane with homogenous and well defined thin top layer structure. It is worth to notice that the support had more open structure than the UF active layer.
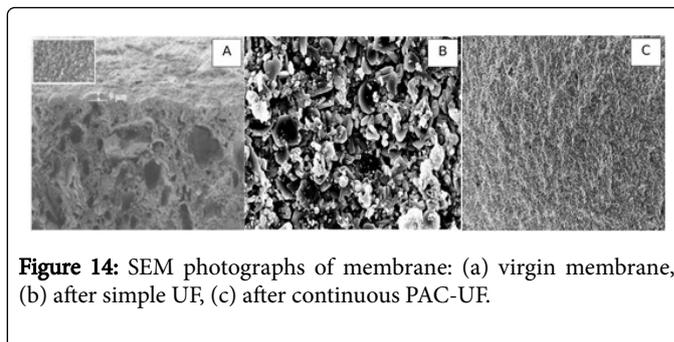
Figure 14: SEM photographs of membrane: (a) virgin membrane, (b) after simple UF, (c) after continuous PAC-UF.

The SEM results obtained after the experiments related to continuous hybrid adsorption –UF at the optimum conditions (Figure 14b) revealed the formation of aggregates by the different adsorbent–dye species that would grow into each other to form less or more open structure principally located on the membrane surface with little fouling material present in the pore networks. It appears then that the formation of the fouling layer is due principally to adsorption and growth mechanism. This result is consistent with the previously described filtration experiment.

By operating with simple UF (Figure 14c), SEM image shows that the pores seem almost totally covered and no distinct fouling layer morphology transition was observed on the surface. This indicates that the fouling material was strongly attached to the membrane which would explain the lower permeate flux obtained in comparison to the case when a continuous hybrid adsorption-UF was conducted.

Conclusion

Hybrid treatment combining adsorption on powder activated carbon-Ultrafiltration (PAC-UF) can be applied in the removal of anionic AO7 dye from aqueous solution.

Results show that the permeate flux remains almost constant during hybrid treatment while a strong decrease of flux was observed when simple UF was applied. The highest performance corresponding to a total retention of AO7 dye was observed for PAC dose at 150 mg/L. While, using only PAC adsorption in batch mode, the optimal conditions are provided by PAC dose up to 300 mg/L.

The transport properties through UF membrane are significantly influenced by several conditions such as ionic charge and the composition of the solution. The best performances were observed at pH range 5-7.

In addition, the presence of cationic surfactant in AO7 dye solution when applying hybrid treatment allows a significantly enhancement of the permeate flux and kinetic of decolorization. The stabilized permeate flux has a value quite near that of water permeability at a pressure of 3 bars. The application for real textile waste water treatment using reactive dye shows similar results to AO7 dye aqueous solution in terms permeate flux behavior and color retention, especially when the cationic surfactant was added. The study of the membrane morphology revealed that the built-up of the fouling layer depends strongly on the filtration process and that continuous hybrid adsorption-UF textile waste water treatment leads to a fouling layer demonstrating the higher permeate fluxes. Furthermore, the open structure identified on the membrane surface makes the membrane easier to clean which is very interesting for industrial scale.

Acknowledgements

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