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Discoloration of Charged Models Wastewater with Reactive and Dispersed Dyes by the Combined Process of Coagulation-Ultrafiltration

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Abstract

The discoloration of models wastewater loaded with dyes from textile finishing industry by physicochemical processes of coagulation-ultrafiltration (CO-UF) in combined system was performed in the present work, exploiting two families' different dyes. The first family was reactive azo type is red cibacron 3 (RC3) and the second was dispersed type antraquinonique which is dispersed blue 7 (BD7). During the coagulation, we used the emulsion of lime ($Ca(OH)_2$) as a coagulating agent at a concentration of 800 mg/l and the asymmetric membrane based on polysulphone PSU UDEL P1700 as selective barrier in the case of ultrafiltration. The effect of pH on the bleaching performance was studied by adjusting their values by acetic acid and sodium hydroxide. When discoloration made by the CO-UF, the results of discoloration obtained from effluents loaded by red cibacron 3 and dispersed blue 7 were respectively recorded in the values 97.43 % and 100 % at the optimum pH equal to 12. According to the proposed study, it shows that the pH has a significant effect on the discoloration of colored water loaded with reactive and disperses dyes, on the one hand, and on the other hand, the combined process of coagulation-ultrafiltration has also improved the performance of their discoloration.

1. Introduction

The textile industry and clothing sector is characterized by diversity, both in terms of raw materials (wool, silk, linen, cotton, synthetic fibers...) or on the stages of manufacture including spinning, warping, weaving and finishing. The textile finishing is the most important step in the textile industry and includes pre-treatment procedures, sizing, desiring, scouring, bleaching, mercerizing, dyeing, printing and finishing [1, 2]. During these proceedings, several dyes, additives, detergents and chemicals are used with a large amount of water [1-8].
The choice of the family of dyes to operate up to the nature of the textile substrate to be dyed and, depending on the brand and the modes required according to the needs of the market. In addition to their chemical structures, the synthetic dyes used in the dyeing of textile fibers, are classified according to their applications and the chemical nature of this type of fibers [7]. Therefore, we find, acid, basic, vat, direct, mordant, azo, developed, reactives and disperses dyes [2, 7, 9-19]. Reactives dyes are soluble in water [2], they enter increasingly frequently in the dyeing of cellulose fibers and possibly those of the wool and the polyamides. Conversely, disperses dyes are sparingly soluble in water [2] and applied as a fine powder dispersed in the dye bath and generally used for dyeing the synthetic fibers [20, 21].
The extensive use of the reactives and disperses dyes with water and other chemical additives in the dyeing of natural and synthetic fibers, produces vis-a-vis a large volume of liquid effluent loaded with dyes, organic matter, inorganic and organometallic [12, 22, 23]. These compounds are toxic to aquatic fauna and flora [22, 24], and therefore for environment [25]. This prompted several countries to adopt some stringent regulations in the processing of colored effluent before discharge into receiving waters [26].
To purify this type of wastewater, many processing techniques have been developed namely those biological, physical and chemical [27-30]. Various physicochemical processes have been used for bleaching effluents loaded with dyes, while signaling to coagulation [31-36], flocculation [34], flotation [31, 37], chemical

oxidation [34, 38], adsorption [34, 39,40] and membranes processes including microfiltration, ultrafiltration, nanofiltration, reverse osmosis [41-43]... To improve the performance of the discoloration of colored wastewater, several researchers based on physic-chemical and biological combined [44-46] in this case the processes of coagulation-precipitation [44], coagulation-flocculation [47-49], coagulation-adsorption [50], coagulation-ultrafiltration [51, 52], etc.
The objective of this study is to discolor the models effluent water loaded with the reactive dye which is red cibacron 3 and that which is disperse dye is blue dispersed 7, using the hybridized process of coagulation-ultrafiltration, while building of lime as a coagulant agent and the asymmetric membranes based on polysulfone PSU UDEL P1700 as selective ultrafiltration barriers and, after the study of the effect of pH and their optimization on relative performance discoloration each model water used.

2. Experimental details

2.1. *Dyes used*
The dyes operated in the preparation of wastewater solutions are commercial models of two different families whose chemical structures and properties showed respectively in the **Table 1** and the **Table 2**. The first dye is cibacron red 3 reagent, which was supplied to the company Ciba (Swiss) and the second is dispersed type disperse blue 7, which was provided to the company Dye Star (Germany).

Table 1: Chemicals structures of dyes reactive RC3 and dispersed BD7.

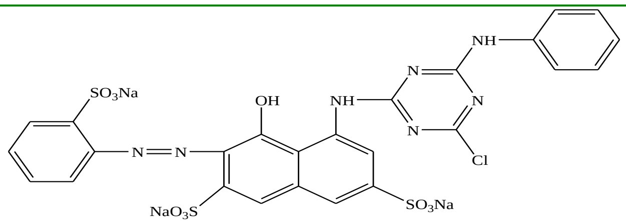
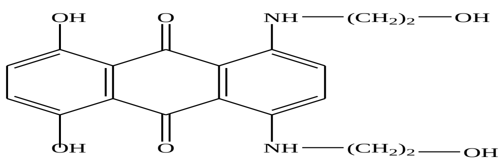
Dyes	Chemical structures
RC3	
BD7	

Table 2: Properties of dyes reactive RC3 and dispersed BD7.

Dyes	Dyeing classes	C.I.	CAS	Chemical formulas	Molar masses
RC3	reactive	18159	23211-47-4	$C_{25}H_{15}ClN_7Na_3O_{10}S_3$	774.05 g/mol
BD7	disperse	62500	3179-90-6	$C_{18}H_{18}N_2O_6$	358.35 g/mol

2.2. Coagulant agent of lime
The coagulating agent used in the coagulation of the prepared colored water is lime $Ca(OH)_2$ of 97% purity and relatively high synergistic properties [34]. The choice of lime as a coagulant returns to its high performance of removing synthetic dyes of textile finishing [1, 34, 53, 54]. The emulsion of lime obtained was prepared at a concentration of 800 mg/l.

2.3. Ultrafiltration of membranes
The membranes of ultrafiltration achieved were prepared in our laboratory (TOCP/LARPPE) by dissolving 12% of the mass of polysulfone in the dimethyleformamide (DMF) [43]. The choice of polymer used in the synthesis of the membranes up to the excellent properties physical, mechanical, thermal and chemical of the polysulfone [55-58] and the membrane resistance to chemical attack vis-a-vis bases and acids [36]. **Tables 3**, **4** and **5**respectively include the chemical structure of the polysulfone, the properties of the polysulfone and the hydrodynamic characteristics of the synthesized membranes.

Table 3: Chemical structure of the polysulfone [43].

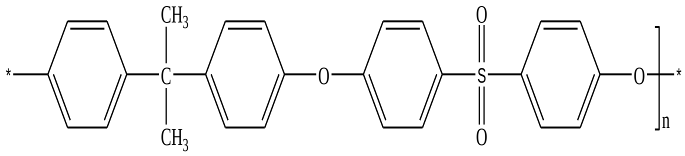
Polymer	Chemical structure
Polysulfone PSU UDEL P1700	

Table 4: Properties of polymerpolysulfone.

Polymer	Supplier	Aspect	Color	Number of monomers	Molecular weight (monomer)
Polysulfone PSU UDEL P1700	Union Carbide (Solvay)	granule	to yellow	$50 \leq n \leq 80$	444 g/mol

Table 5: Hydrodynamic characteristics of membranes obtained[43].

Membranes	Form	Permeability	Selectivity	Thickness	Profile
	flat and white	permeable	88 % to the molecular weight of dextran 298 000 g/mol	180 µm	asymmetric structure

2.4. Sample preparation models of wastewater

The wastewater samples operated to conduct the experimental part of this study were prepared as follows: In a glass bottle, we have solution 50 mg of dye red cibacron 3 in one liter of hot water (60°C). To solubility the dye, the resulting mixture was stirred several times with a magnetic stirrer, knowing that the measured pH of the colored water sample obtained was 6.4. In the case of the dispersed blue 7 dye, we set mixing 50 mg of this dye in a liter of hot water (90°C), and to solubility it, we have added a few drops of acetic acid, such the obtained sample was of pH = 5.

2.5. Wastewater characterization models

To characterize the wastewater prepared, we conducted a spectrophotometric analysis of each sample using a spectrometer JP. SELECTA.sa model 2100 shown in **Fig. 1** below, provided with two thick tanks 1cm, one of quartz and the other of glass, covering the wavelengths between 190 and 1100 nm. In order to determine the maximum wavelength for which colored wastewater, we determine subsequently the optical densities before and after fading, and therefore to determine the rate of discoloration (% D) of wastewater samples depending on the models following relationship [43, 52, 59]:

$$\%D = \frac{DOi - DOf}{DOi} \times 100 \qquad \textbf{(1)}$$

Such as:
DO_i is the optical density of the colored wastewater model sample before fading;
DO_f is the optical density of the water after blanching.
The measurement of the coloration corresponds to the measurement of the absorbed light at different wavelengths based on the Beer-Lambert law:

$$\mathbf{A = \varepsilon_{\lambda max}.\ l.\ C} \qquad \textbf{(2)}$$

Or:
A: absorbance,
L: distance traveled by the light beam through the solution (L = 1 cm),
C: concentration of the colored water sample (mg/l or mol/l),
$\varepsilon_{\lambda max}$: molar extinction coefficient of the colored solution at a maximum wavelength (λ_{max}).

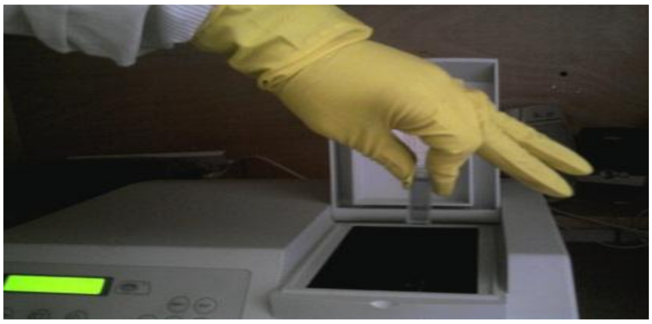


Figure 1: Absorption spectrometer JP.SELECTA.sa Model 2100.

2.6. *Effect of the pH on the fading of colored water*

To study the effect of pH on the performance of bleaching wastewater samples used, we took three volumes of colored water from the specimen and to vary the pH using acetic acid for the acidic pH and caustic soda for the basic pH. Besides the mothers samples of colored water whose pH 6.4 and 5 towards the dyes of RC3 and BD7, we adjusted the pH values to 4.32; 9; 12 in the case of solutions charged by the RC3dye and to values 3; 9; 12 in the case of samples loaded by the BD7 dye. After fixing the values of pH, we have measured the absorbency of each sample of colored water. The pH measured was performed using a pH-meter type HANNA instruments, which is previously calibrated with buffer solutions at constant pH.

2.7. Discoloration of water colored by the combined process CO-UF

During the first stage of this part, we conducted a primary treatment of coagulation by adding to each model of colored water3 ml of lime emulsion and at different values of pH, under agitation fast with a speed 200 tr/min for 2 min using a magnetic stirrer at temperature of 25 ± 5 °C and then settling was made in the same beaker for 10 min. During the second stage, we performed the tertiary treatment by membrane filtration using asymmetric membranes permeable polysulfone through an Amicon ultrafiltration cell and volume of 36 cm^3, the schema and principle of which are shown in **Fig. 2**. Note that, for the pore size of the UF membrane, it is not necessary to achieve a flocculation, because the membranes used in this study enable the selection of microflocs [52, 60].

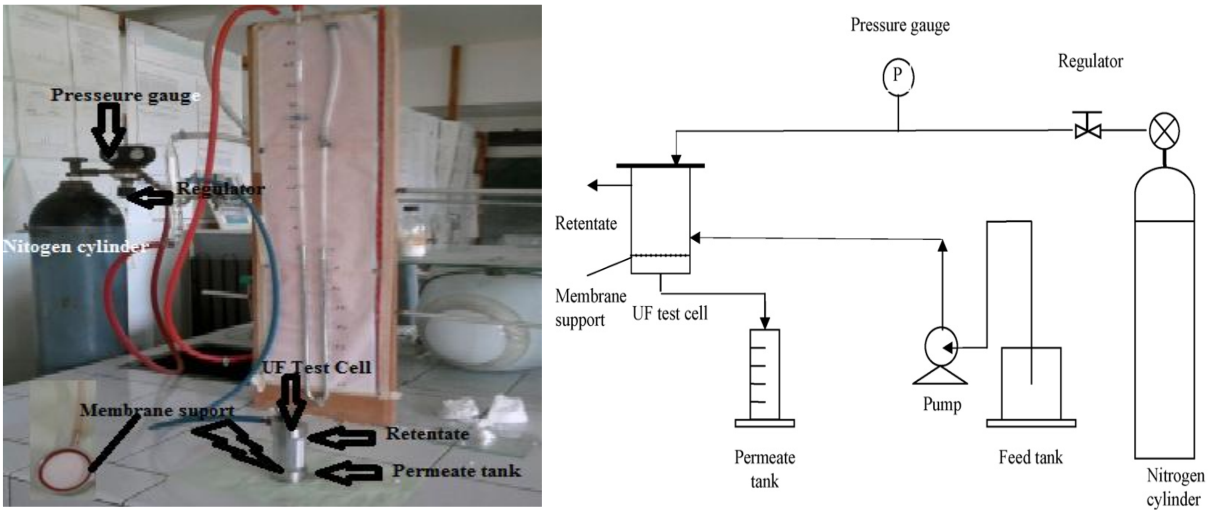


Figure 2: Mounting and principle of ultrafiltration Amicon cell (UF) [43, 61].

To avoid too much room for error in this stage, the parameters of the membrane pressure and stirring speed of magnetic bar of the UF cell must be constant.

3. Results and discussion

3.*1. Sample spectrometry analysis models wastewater*

3.1.1. Absorption wastewater models

The absorption spectra initialof wastewater model compounds the RC3 (pH = 6.4) and BD7 (pH = 5) dyes are shown in the **Fig. 3**. All measurements were performed at different wavelengths in the visible spectrum (400-800 nm).

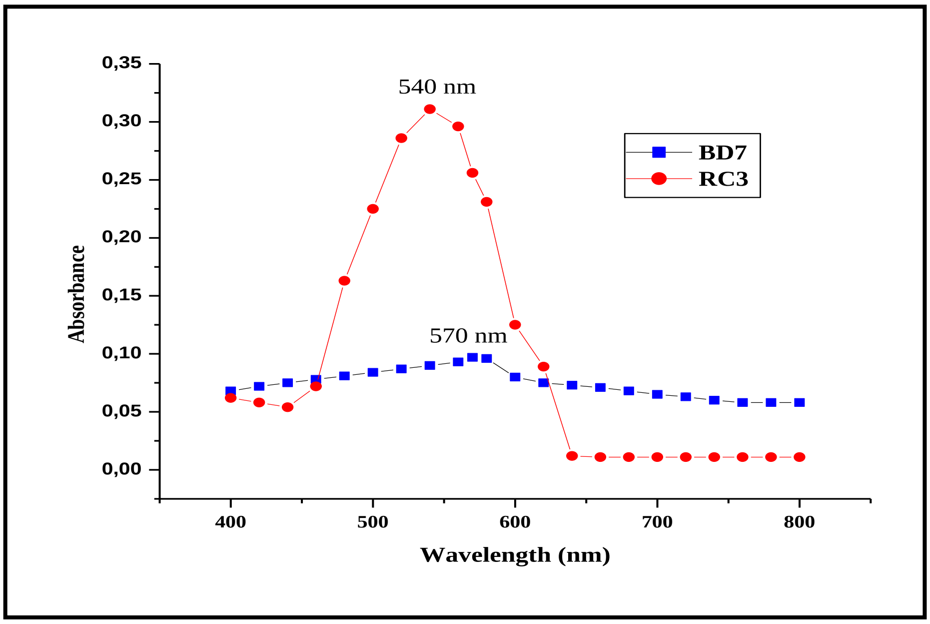


Figure 3: Absorption spectra of red cibacron 3 (**RC3**) and disperse blue 7 (**BD7**) dyes.

According to the spectra of **Fig. 3**, the minimum removals obtained of wastewater loaded with RC3 and BD7 dyes exploited in the present work, corresponding to maximum wavelengths, are stored respectively the values 540 and 570 nm. The peak wavelength obtained from the RC3 is relatively with the literature [62]. This difference can return to the purity of the colors and the uncertainty of the measurement computers working.

3.1.2. Calibration curves of exploited wastewater

The calibration curves shown in **Fig. 4** (**a**) and (**b**) were determined in order to validate the adsorption of our samples based on Beer-Lambert law. During this study, the calibration curves corresponding to each λ_{max} model samples of colored water in a weight concentration range between 0 and 50 mg/l were made at λ_{max} = 540 nm in the case of water charged by the RC3 dye and at λ_{max} = 570 nm in the case of water loaded by the BD7 dye.

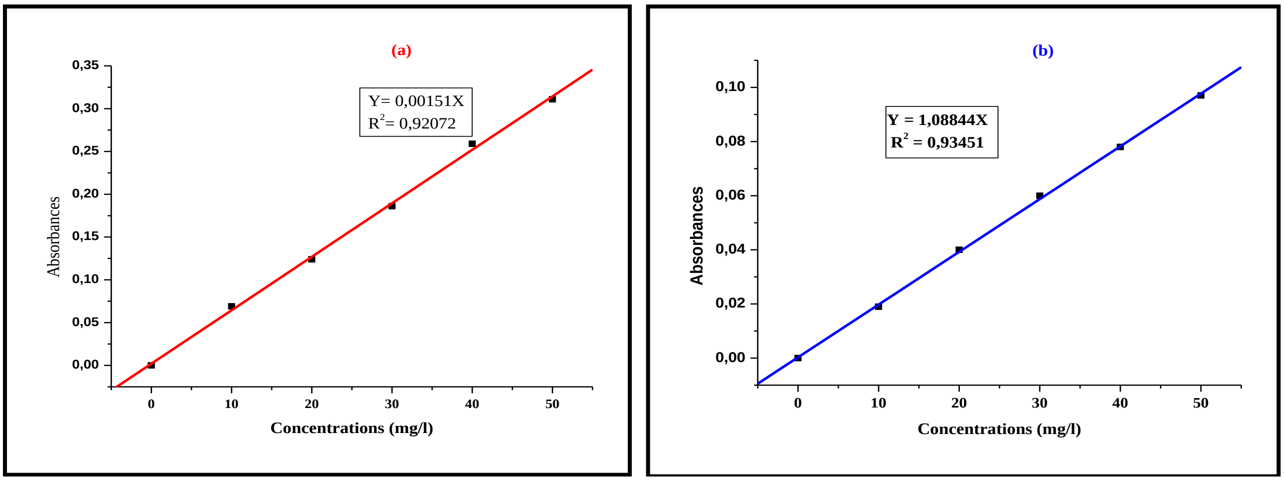


Figure 4: Calibration curves of model wastewater loaded by dyes RC3 **(a)** and BD7 **(b)**.

From these figures, we find that the curve of calibration for the colored waters is a right, indicating the Beer-Lambert law was approved and according to the latter, the coefficients of molar extinction ε_{max} each length maximum wavelength RC3 and BD7 dyes are calculated in values 4814.24 and 692.86 mol^{-1}.l.cm^{-1}.

3.2. Effect of the pH on the bleaching of colored water

The study of degradation RC3 and BD7 dyes exploited in this work was carried out at acidic pH (3; 4.32; 5; 6) and base (9; 12). The results of the effect of different pH values on the discoloration of colored water are well represented in **Fig. 5**.

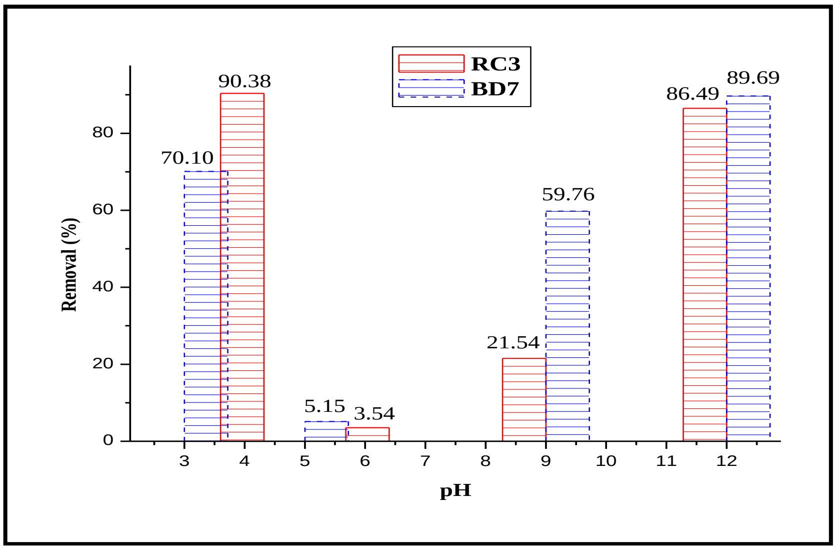


Figure 5: Variation of bleaching colored wastewater function of pH.

From this figure, we see a small discoloration of around 3.54 % at pH = 6.4 for the dye of RC3 and the order of 5.15 % at pH = 5 in the case of BD7 dye. The low discoloration indicates that we are in the initial state of the prepared water solutions and models that the results can be explained by the degradation of dyes in the light effect [63]. Furthermore, the addition of caustic soda (NaOH) to pH = 9, increased fading rate in the percentages of 51.54 % and 59.76 % vis-a-vis the RC3 and BD7 dyes. Similarly, at pH = 12, the discoloration respectively reached in the values of 86.49 % and 89.69 % for the RC3 and BD7 dyes. However, the rate of discoloration has reached the maximum 90.38 % by the addition of acetic acid to pH = 4.32 in the case of the maximum percentage RC3 and at 70.10 % in the case of pH = 3 with respect to the BD7. This is explained by the deterioration of RC3 and BD7 dyes under the effect of pH [64].

3.3. Resultsof discoloration from fading by the combined process CO-UF

The bleaching effluents operated by the hybridized CO-UF process was performed by adding 3 ml of coagulant the lime emulsion at various colored samples with various pH values [54]. The **Fig. 6** shows the results of the rate of bleaching effluent operated by the combined CO-UF process with the variouspH obtained in the previous section (**3.2**).

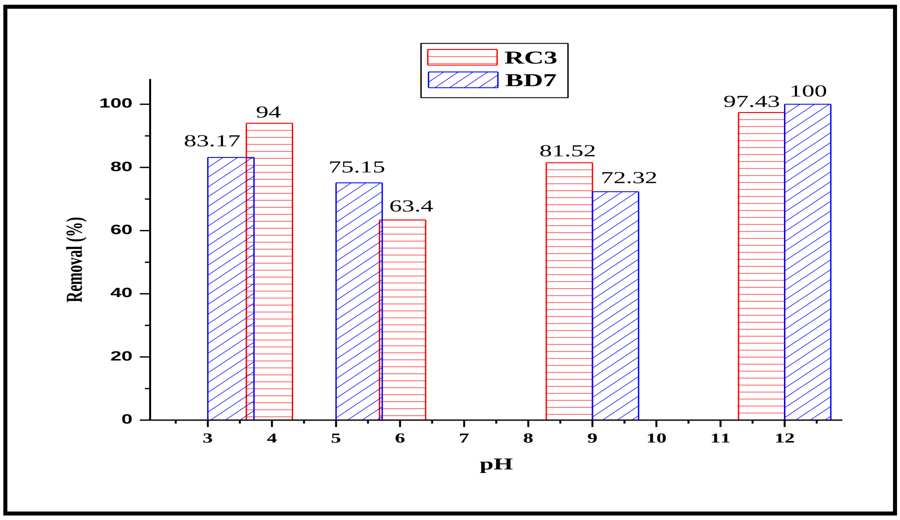


Figure 6: Bleaching of colored effluents by CO-UF process depending on the pH.

From the results shown in the **Fig. 6**, it shows that the fading rate of the wastewater by the combined process of coagulation-ultrafiltration is more significant at different pH values. To pH = 4.32 and 3, the rate of discoloration has been obtained respectively in the values 94 % and 83.17 % for the water charged by RC3 and BD7 dyes. To pH = 6.4 and 5, the rate of discoloration has been obtained respectively in the values 63.4 % and

75.15 % vis-a-vis the RC3 and BD7 dyes. To pH = 9, the rate of discoloration has been obtained respectively in the values of 81.52 % and 72.32 % vis-a-vis the RC3 and BD7 dyes [34]. To pH = 12, the rate of discoloration has reached the maximum percentages respectively 97.43 % and 100 % compared to the effluent loaded with RC3 and BD7 dyes. Analysis of the results reveals that the discoloration coagulation with lime emulsion at 3 ml has generated precipitates that have embraced and adsorbed effluent dye compounds exploited in this study causing them to precipitate, this is more noticed very basic pH [53]. Moreover, an ultrafiltration membrane by asymmetric polysulfone has in turn increase process performance while retaining the colloidal and precipitates. This may go back to the very small pore of size and the asymmetric structure of the membranes used. Therefore, the molecular size of RC3 and BD7 dyes becomes very small relative to that of the membrane pores. The increase in the rate of fading in very alkaline medium (pH = 12), can be explained by the asymmetric structure of the membranes used, the uneven distribution of the diameter of these pores, the quasi-solubility of the dye BD7 (100 %) in water and the completing solubility of the dye RC3 (97.43 %) in the presence of a basic medium.

Conclusions

In the light of this work, the fading of models wastewater loaded with dyes of red cibacron 3 (RC3)and blue dispersed 7 (BD7)requires the effect of pH study of each colored effluent and optimization. And the performance of the discoloration of this type of effluent by the combined process of coagulation-ultrafiltration (CO-UF) is much greater when using the coagulating lime at the concentration of 800 mg/l, the permeable and asymmetric membranes synthesized by PSU UDEL P1700 polysulfone. According to the study, the rate of fading by the process used has reached the maximum percentages of 97.43 % and 100 % from the effluent loaded respectively with RC3 and BD7 dyes, at optimal pH of 12 and at a dose 3 ml of emulsion of the lime mass concentration 800 mg/l. We will study in a future study, the effect of different doses of the coagulant used on the rate of bleaching effluents loaded by RC3 and BD7 dyes by the combined process CO-UF.

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