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Removal of Direct Blue 71 from wastewater using micellar enhanced ultrafiltration

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ABSTRACT

Micellar-enhanced ultrafiltration (MEUF) represents a potentially attractive tool for the removal of different contaminants from wastewaters. In this study, MEUF has been carried out to investigate the retention of Direct Blue 71 (DB71 MW 965.94), an azo dye with a high worldwide consumption providing toxic effluents, from aqueous stream. The efficiency of MEUF on the removal of DB71 was studied as a function of dye and surfactant concentrations, type of surfactant, ionic strength and pH.

The experiments showed that the highest dye rejection was about 98% for cationic surfactants due to the high electrostatic interaction between this surfactant and dye. The retention depended slightly on dye and surfactant concentration, ionic strength and pH. However, permeate flux decreases when surfactant and electrolyte concentrations increases which was mainly attributed to the concentration polarisation and osmotic pressure.

Keywords: Direct Blue71; Micelles; Ultrafiltration; Surfactant

1. Introduction

Various types of dyes are manufactured for printing and dyeing industries from coal tar based hydrocarbons such as benzene, naphthalene, toluene, etc. Most of these dyes are harmful, when brought in contact with living tissues for a long time. The discharge of these to the river stream without proper treatment causes damage to the crops and living beings, both aquatic and terrestrial. Azo dyes constitute the largest group of colorants used in industry. There are aromatic rings in its molecular structures which cause these effluents to be toxic and mostly non-biodegradable; therefore, becoming an important source of environmental pollution. Due to their high molecular weighs, their complex structures and especially their high solubility in water, they persist once discharged into a natural environment [1]. Direct Blue 71is used in dyeing of silk, wool, paper and of pulp, also used in organic paint. Removal of the unused dye from the effluent is a difficult requirement faced by the textile finishing, dye manufacturing, pulp and paper industries.

Several techniques for removal of colored dye from wastewater, i.e., coagulation/flocculation [2], various advance oxidation processes [3,4] and adsorption on to: (i) sludge of wastewater treatment plant [5]; (ii) different bentonites [6,7]; (iii) different types of activated carbon [8]; and (iv) surfactant impregnated montmorillonite [9], etc., are available in the literature. Among these, adsorption is the most common technique. But it is inherently a slow process and its performance is limited by equilibrium. Recently membrane separation

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of dyes [10,11]. Among membrane processes, the nanofiltration (NF) is the most suitable for the decolourisation of effluent textile [11]. However, its major disadvantage is the decline in permeate flux due to adsorption of organic compounds on the membrane surface [12].

Micellar-enhanced ultrafiltration (MEUF) is one of the possible methods to remove organic dyes from water [13-16].

The basic idea for MEUF is that the surfactant forms large amphiphilic aggregate micelles when it is added to aqueous stream at a concentration higher than its critical micelle concentration (CMC). The ions and dissolved organic compounds (solute) can be mostly trapped by the micelles if they tend to be strongly attracted by the micelle surface and will solubilize in the micelle interior, respectively. The micelles containing solubilized solute are larger in size which makes it easier to be filtered by an ultrafiltration membrane, leaving only water and a small amount of unsolubilized solutes with free surfactants in the permeate stream [15].

In the last decade, increasing interest on the use of aqueous micellar solution has been found in the field of separation [17-28]. A review of the literature related to MEUF reveals that a number of studies have been carried out to understand the mechanism of separation and solubilization of the solute in the micelles to optimize the operating conditions.

MEUF can successfully be used to separate different metal ions [17-23]. Karate and Marathe [18] carried out MEUF studies on nickel and cobalt. These ions were simultaneously removed from aqueous feed using sodium dodecyl sulfate (SDS). Likewise, Chandan et al. [20] used SDS as anionic surfactant to remove Cu^{2+} and Ca^{2+}. Also, Fang et al. [19] employed nonionic surfactants Triton X-100, Brij 35 and anionic surfactant SDS to MEUF for separating cadmium ion. Federico [21] studied the colloidal stability of SDS surfactant micelles with bound mixtures of Al^{3+} and Zn^{2+} cations through fouling membrane ultrafiltration.

The toxic dyes have been investigated in many studies [25-28]. It is reported that many dyes were removed successfully from wastewater. Purkait et al. have studied the performance of MEUF to remove toxic eosin dye from aqueous phase using cetyl(hexadecyl) pyridinium chloride (CPC) as a cationic surfactant [27]. Bielska et al. [25,26] carried out both MEUF as well as solubilization studies on dyes and micelles in aqueous medium. Their research was investigated the effects of surfactant and membrane types upon the retention of methylene blue, mordant black 11 and mordant black 17 and they determined parameters such as the micelle loading (L_m), the micelle binding constant (log K_P) and the distribution coefficient (D), typical in colloid and extraction studies. The same authors had shown [26] that the interaction of four ionic dyes: C.I. Mordant Black 11, C.I. Mordant Black 17, C.I. Direct Yellow 50 and C.I. Basic Blue 9, with cationic and anionic surfactants by absorption spectroscopy. They concluded that the dyes interact strongly with oppositely charged surfactant in the premicellar concentration range and they estimated appropriate values of constant of dye–surfactant complex formation.

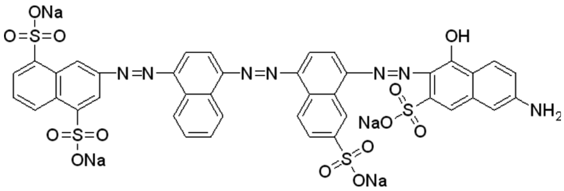

Fig. 1. The structure of Direct Blue 71.

The aim of this study was to evaluate the effect of chemical parameters such as ionic strength, chain length of surfactant and pH on the removal of Direct Blue 71, an anionic industrial dye, byMEUF.

2. Experimental

2.1. Materials

The different chemicals used are as follows: (i) dye Direct Blue 71 $C_{40}H_{23}N_7Na_4O_{13}S_4$ (MW 965.94), its structure is shown in Fig. 1. (ii) Sodium chloride NaCl (99.5% purity), (iii) surfactants: three *n*-alkyltrimethylammonium bromide (C_{14}TAB, C_{16}TAB and C_{18}TAB), SDS and Triton X-100 (TX-100). The CMCs of the studied surfactants in distilled water were shown in Table 1 [29]. All the chemicals products were supplied by Fluka and used as received (Table 2).

2.2. Membrane cell and membranes

Ultrafiltration was carried out at room temperature as previous paper [14]. Cross-flow membrane filtration was carried out with a tangential cell system Minitan-S purchased from Millipore and the effective filtration area was 30 cm^2. Organic regenerated cellulose membrane of molecular weight cut-off (MWCO) 10 kDa obtained from Millipore was used for all the MEUF experiments.

The membrane was soaked in deionised water during 24 h in order to eliminate preservative products. Then pure water flux at various operating pressures is measured and the membrane permeability is determined from the slope of the flux versus pressure plot.

Table 1
Characteristics of surfactant used in this study

Surfactant	MW	Purity	CMC at 25 °C
Tetradecyltrimethylammonium bromide (C_{14}TAB)	336.41	>98%	3.7×10^{-4}M
Hexadecyltrimethylammonium bromide (C_{16}TAB)	364.46	≥99%	8.7×10^{-4}M
Octadecyltrimethylammonium bromide (C_{18}TAB)	392.52	>97%	3.0×10^{-4}M
Sodium dodecyl sulfate (SDS)	288.38	≥98%	8.0×10^{-3}M
Triton X-100 (TX-100)	624	$d = 1.065$	2.4×10^{-4}M

The value of the membrane permeability is found to be $2.47 \times 10{-}10$ mPa^{-1} s^{-1}.

2.3. Methods

All ultrafiltration experiments were carried out at room temperature 30 °C in a continuous manner under a pressure difference $\triangle P$ with retentate recirculation to the aqueous feed vessel and permeate collection. The feed tank was initially filled with 250 mL solution and a micro pump with a variable flow rates was used to feed the solution into the cell. The retentate as well as the permeate were recycled in to the feed tank. After nearly 20 min a steady-state was reached and a permeate of 10 mL was collected and analysed. After each run, the membrane was thoroughly washed by distilled water for at least 15 min and at a pressure of 1.4 bar. The membrane permeability was checked to ensure that the permeability remains almost constant between successive runs.

Solutions of dye with and without surfactant or/and electrolyte have been prepared by dissolving accurately weighed amount of surfactant, dye and electrolyte in distilled water at different concentrations.

The filtration efficiency in removing the dye from the feed solution was evaluated through the dye rejection and permeate flux which were calculated using Eqs. (1) and (2) below,

$$R = \left[1 - \frac{C_p}{C_0}\right] \times 100 \quad (1)$$

$$J = \frac{V}{\Delta t \ .S} \quad (2)$$

where R is the percentage of observed dye rejection, C_p is the concentration dye in the permeate (mol L^{-1}), C_0 is the initial concentration of the dye in the feed solution (mol L^{-1}), J is the permeate flux (L h^{-1} m^{-2}), V is the volume of permeate (L), $\triangle t$ is the time difference (s) and S is the membrane area (m^2), respectively.

2.4. Analysis

Permeate concentrations of DB71 are measured by a Perkin-Elmer Lambda 20 spectrophotometer. The wavelengths at which maximum absorption occur are 581 nm for DB71 without surfactant and 583, 575, 572, 578 and 567 nm for DB71 with, respectively, C_{14}TAB, C_{16}TAB, C_{18}TAB, TX-100 and SDS at concentration 5 × CMC. The DB71 absorbance versus concentration plot follows Lambert-Beer law from 0 to 8 μM in all cases.

3. Results and discussion

3.1. Ultrafiltration of dye solution with and without C_{16}TAB

Fig. 2a and b shows, respectively, retention rate of DB71 and flux of permeate as a function of time. We observed from the Fig. 2a that in absence of surfactant the retention of DB71 is between 63% and 69% at feed dye concentration in the range of 10^{-3} to 10^{-4} M. This retention could be attributed to the adsorption of dye at the surface or in the pores of membrane. When surfactant is used, the retention of DB71 increases to about 98%. This clearly indicates that the dye is solubilized on the surfactant micelles, which are subsequently retained by the ultrafiltration membrane.

The variation of the permeate flux without surfactant (Fig. 2b) remains constant with time. However, with the presence of surfactant a slight decrease of flux is observed during initial stage of filtration, and thereafter, the flux attains a steady-state value. Lower flux is

Table 2
CAS numbers of the chemicals used

Chemical	DB71	NaCl	C_{14}TAB	C_{16}TAB	C_{18}TAB	SDS	TX-100
CAS number	4399-55-7	7647-14-5	1119-97-7	57-09-0	1120-02-1	151-21-3	9002-93-1

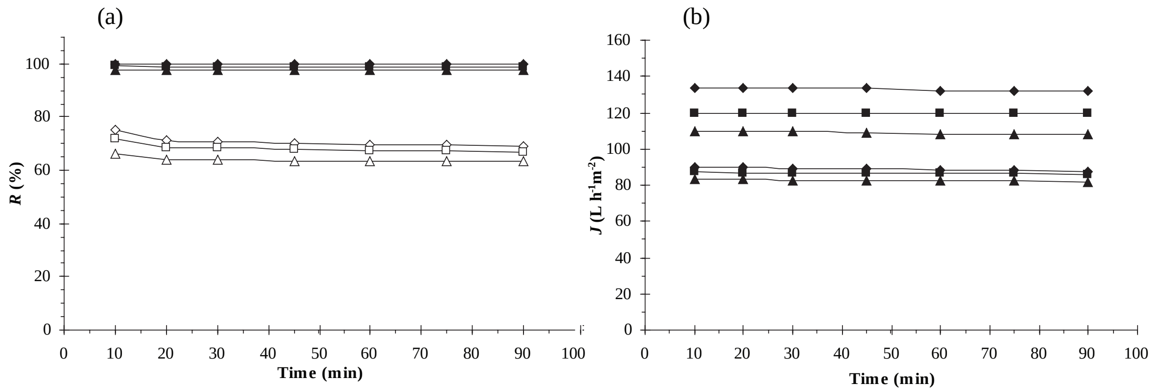

Fig. 2. Variation of dye retention and permeate flux during UF and MEUF with operating time at different DB71 concentrations of (◇, ◆) 10^{-4} M, (□, ■) 5 ×10^{-4} M and (△, ▲) 10^{-3} M at 1.4 bar and 30°C. Feed C_{16}TAB concentration is 2 × 10^{-3} M; empty (◇, □, △) and full points (◆, ■, ▲) correspond to the solutions in the absence and in the presence of C_{16}TAB, respectively.

observed during UF of dye with surfactant micelles. During the ultrafiltration, the micelles formed by surfactant molecules are rejected and accumulated near the membrane surface, resulting in a higher surfactant concentration near the membrane compared to the bulk surfactant concentration.

3.2. Effect of feed surfactant concentration

Fig. 3 describes the effect of feed C16TAB concentration on the retention of DB71 and on permeate flux at different DB71 concentration. The retention remains almost constant with varying C16TAB concentration. For example, at 10−3 M of feed dye concentration, the retention of dye varies from 97% to 98% when C16TAB feed concentration increased from 1 to 40 mM. In these experiments the ratio of C16TAB to DB71 are high enough that solubilization capacity of the micelles is not reached.

On the other hand, the permeate flux decreases when the feed surfactant concentration increases. Purkait et al. reported similar trends of permeate flux during cross-flow ultrafiltration of CPC solution in presence of phenolic derivatives [30] and eosin dye [27]. Indeed, the increase of micelles concentration generates a deposited layer over the membrane surface (concentration polarisation) and consequently, increases the resistance against the solvent flux though the membrane. Also, the increases in the osmotic pressure difference across the membrane reduces the effective transmembrane pressure and consequently, decreases the permeate flux.

3.3. Effect of the nature of surfactant

To study the effect of the nature of the surfactant on the retention of DB71 was investigated by comparing the filtration of dye solutions in presence of anionic

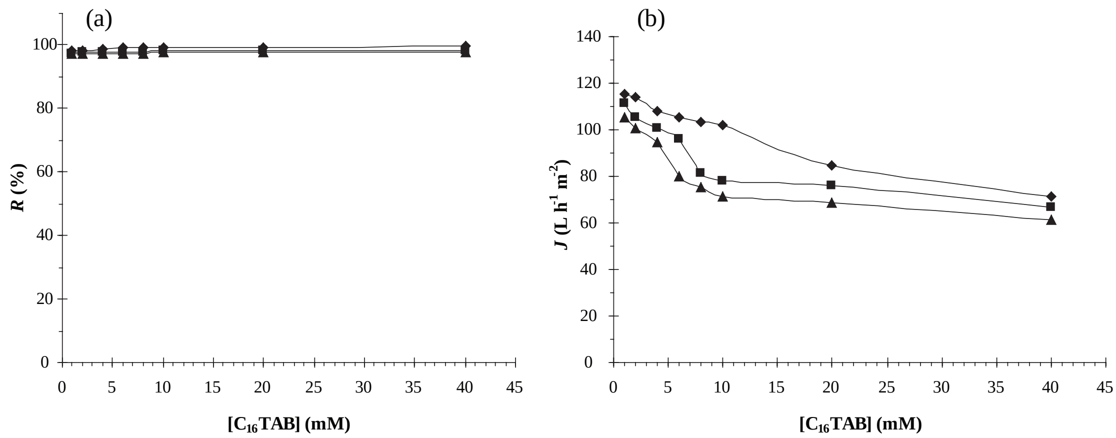

Fig. 3. Effect of feed C_{16}TAB concentration on dye retention and permeate flux at different DB71 concentrations (◆) 10^{-4} M, (■) 5 × 10^{-4} M and (▲) 10^{-3} M at 1.4 bar and 30°C.

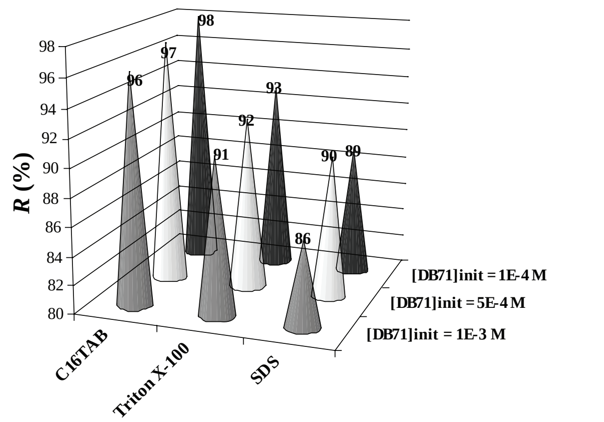

Fig. 4. Variation of DB71 retention in the presence of surfactants (TX-100, SDS and C_{16}TAB) for three feed DB71 concentration (10^{-4} M, 5 × 10^{-4} M and 10^{-3} M). Surfactants concentration is 5 × CMC.

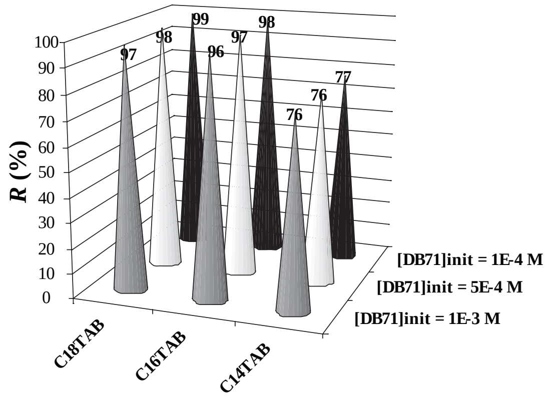

Fig. 5. Variation of DB71 retention of for three kind of *n*-alkyltrimethylammonium bromide (C_{14}TAB, C_{16}TAB and C_{18}TAB) at different feed DB71 concentration (10^{-4} M, 5 × 10^{-4} M and 10^{-3} M). Surfactants concentration is 5 × CMC.

(SDS), cationic (C_{16}TAB) and nonionic (TX-100) surfactants. The filtrated solutions contain different dye concentrations and fixed surfactant concentration (5 × CMC). From the Fig. 4, it may be observed that, at 10^{-3} M of dye concentration as example, the dye retention are 86%, 91% and 96% for the anionic, nonionic and cationic surfactant, respectively. The retention using cationic surfactant is the highest which is related to the electrostatic interaction between dye and surfactant with opposite charge. The non-negligible retention obtained by the two other surfactants indicates that hydrophobic interaction between DB71 and surfactants contributes in the retention of the dye.

3.4. Effect of the surfactant chain length

The retention of dye was also studied by using a series of *n*-alkyltrimethylammonium bromide surfactants (C_nTAB). The variation of retention rate of DB71 and the permeate flux are reported in Figs. 5 and 6. The dye retention rate is about 98% for C_{16} and 97% for C_{18} surfactant. The dye retention in presence of C_{14} is much lower and it is around 76%. This result confirms that the hydrophobic effect plays an important rule in the mechanism of dye solubilization by micelles. The permeate flux decreases when surfactant chain length decreases. In fact, surfactant with shorter chain length has the higher CMC value which induces more micelles near the membrane interface and accordingly the effect of concentration polarisation is enhanced.

3.5. Effect of ionic strength

The effluents from textile and dyeing industries contain a high concentration of salts which may affect the removal of dye. The presence of electrolyte can decrease the CMC of ionic surfactants because the electrolyte can weaken the repulsive forces between the head groups, which are normally fighting against the aggregation of surfactant monomers. Therefore, micelles can form comparatively easier in the presence of electrolyte [31,32]. Consequently, the addition of electrolyte to solutions of surfactant increases the extent of solubilization of hydrocarbons that are solubilized in the inner core of the micelle and decreases that of polar compounds that are solubilized in the outer portion of the palisade layer [33,34] In the case of DB71, it can be seen from Fig. 7a that DB71 retention decreases slightly upon increasing the sodium chloride concentrations from 0 to 500 mM. This result suggest that solubilization of DB71 is located in a region

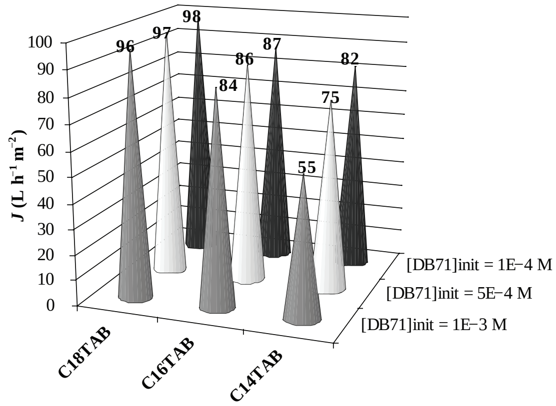

Fig. 6. Variation of permeate flux for three kind of *n*-alkyltrimethylammonium bromide (C_{14}TAB, C_{16}TAB and C_{18}TAB) at different feed DB71 concentration (10^{-4} M, 5 × 10^{-4} M and 10^{-3} M). Surfactants concentration is 5 × CMC.

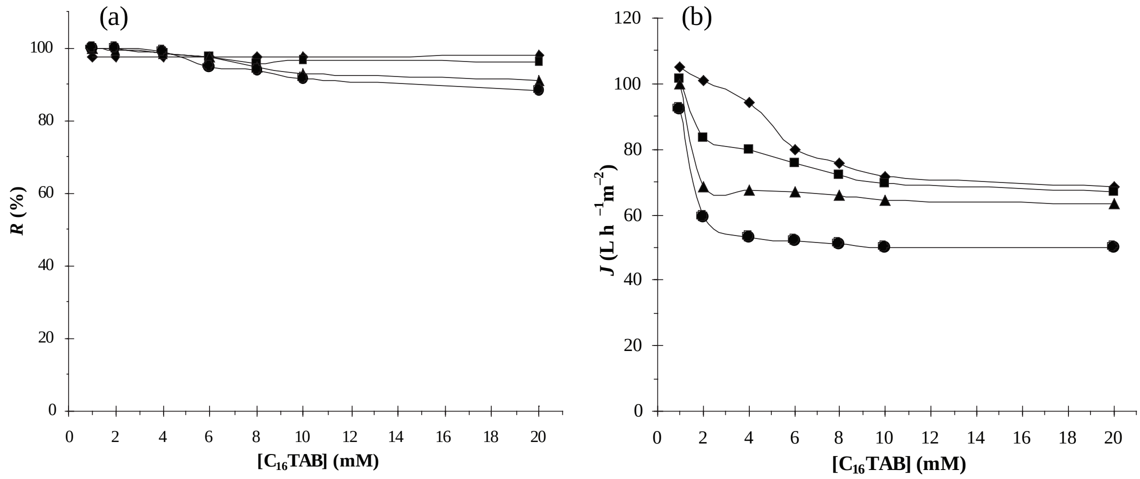

Fig. 7. Variation of DB71 retention and permeate flux with feed C_{16}TAB concentration at different NaCl concentrations (◆) 0 mM, (■) 10 mM, (▲) 100 mM and (●) 500 mM at 1.4 bar and 30°C. Feed DB71 concentration is 10^{-3} M.

between the core and the surface of micelle. Large polar molecules, such as polar dyestuffs, are believed to be solubilized, in aqueous medium, mainly between the individual molecules of surfactant in the palisade layer with the polar groups of the solubilizate oriented toward the polar groups of the surfactants and the non-polar portions oriented toward the interior of the micelle. Interaction here is presumably by H bonding or dipole–dipole attraction between the polar groups of solubilizate and surfactant [35,36].

Fig. 7b presenting the variation of permeate flux as a function of feed concentration of C_{16}TAB, indicates that the flux of permeate decreased when the concentration of NaCl increased. As ionic strength increased CMC of surfactant decreased and larger fraction of C_{16}TAB micelles are formed. This enhanced the effect of polarisation concentration.

3.6. Effect of pH

To study the effect of pH on the retention of dye by MEUF process only cationic surfactant C_{16}TAB was used. The filtered solutions contain 10^{-3} M and 2×10^{-3} M of dye and C_{16}TAB concentrations, respectively. The initial pH value ranging from 1.8 to 12.4 was controlled by the addition of dilute chloride acid (HCl) or sodium hydroxide (NaOH) solutions. Fig. 8 shows that the pH did not have any significant effect on retention of DB71 by MEUF. The retention is near 98% in the whole range of pH. The effect of pH on the permeate flux has been also studied. The flux increased progressively when pH increased from 1.8 to 12.4. This can be attributed to the fact that the membrane becomes more hydrophilic as a result of deprotonation of carboxylic group within the membrane active layer.

4. Conclusion

The MEUF has allowed the removal of Direct Blue 71 from an aqueous stream. The best retention rate in the order of 98% was obtained by using C_{16}TAB. The retention depends slightly on chemical parameters such as surfactant and dye concentrations, ionic strength, chain length and pH. This is due to the high interaction between surfactant and dye. However the flux decreased when the surfactant and dye concentration or ionic strength increased. This study demonstrated the potential of the MEUF for effective removal of organic dye.

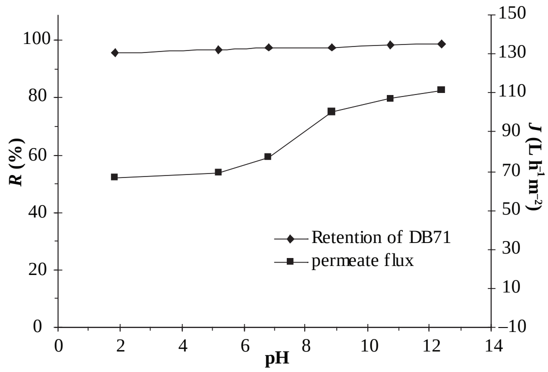

Fig. 8. Effect of pH on the retention of DB71 and on the permeate flux at 1.4 bar and 30°C. [DB71] = 10^{-3} M and [C_{16}TAB] = 2×10^{-3} M.

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