Competitive removal of Reactive Black 5/Reactive Orange 16 from aqueous solution via Micellar-enhanced Ultrafiltration

Puasa S. W., Ruzitah M. S., and Sharifah A. S. A. K.

***Abstract*—This study was designed to investigate the removal of reactive dyes mxiture, C.I Reactive Black 5 (RB5) and C.I Reactive Orange 16 (RO16) using Micellar-enhanced ultrafiltration (MEUF). A cationic surfactant, cetylpyridinium chloride (CPC) was used for the MEUF and its effectiveness was investigated. The critical micelle concentration of CPC was obtained at 0.38 g/L via conductivity method. The diameter of micelles (CPC: 0.6 g/L) obtained at range of 2.3 nm to 6.5 nm diameter. The MEUF process was performed using cellulose acetate membrane with 20,000 MWCO and the surfactant concentration was varied from 0.5 g/L to 1.0 g/L at 400 kPa operating pressure. The results show that the highest dye rejection achieved was 78.0% and 79.2% of RB5 and RO16 for single dye removal while 76.4% and 81.6% of RB5 and RO16 for RB5-RO16 mixture removal from aqueous solution.**

***Index Terms*—Cationic surfactant, critical micelle concentration, micellar-enhanced ultrafiltration, reactive dye.**

I. Introduction

Production of colored textiles is listed as one of essential technology worldwide. The growth of Malaysia's textiles and apparel industry started in the early 1970s when the country entered on export oriented industrialization. This industrial sector has contributed unquestionably to Malaysia's economic development especially in East the Coast of Peninsular Malaysia and Sarawak [1].

Dye as the main key player in the textile industry can be described generally as organic or inorganic substances which can absorb light and reflect some light to show color. The goods were immerged into the dye solution which called dye bath for a certain period of time thereby creating bonding between the dye and the goods [1]. Reactive dyes are water soluble dyes which are extensively used by the industries. It is a soluble anionic dye that contain one or more reactive groups which are designed to bond covalently with hydroxyl group in cellulosic fibers [2]. These dyes have wide variety of color shades, high wet fastness profiles which ease the application, brilliant colors, and require minimal energy consumption for dyeing process [3]. Reactive dyes are also used for dyeing of wool and polyamide [3], [4].

The development of textile industry and improvement of human life from day to day has led to high demand for dyes. Currently, textile industry are facing a number of complex environmental problems, due to the substantial amount of dye released in the wastewater [5]. In the textile industry, dyeing process uses the largest volume of water mainly for dyeing, fixing and washing process. The presence of even small amount of dye in the water is highly visible which affects the water transparency and the aquatic life by blocking the passage of sunlight through the water [6]. According to N. K. Lazaridis, T. D. Karapantsios, and D. Georgantas [7], the concentration of dyes present in textile effluent are normally between 10 to 50 mg/L and the existence of dye in wastewater is visible at dye concentration above 1 mg/L.

There are several current alternative methods that have employed by textile industry to treat the textile wastewater. Some of the example are adsorption, coagulation/flocculation, and polyelectrolyte [8]. Generally, dye with low solubility can be easily removed by physical means such as flocculation and coagulation but not efficient for water soluble dyes such as reactive dyes. The conventional biological treatment processes also unable to achieve adequate color removal [9]. Furthermore, there is problem with these methods where the biological treatment methods are more focus to reduce the COD but insufficient for color removal [10]. Fixed bed adsorption would be the most economical process for this problem, however the process is neither selective nor energy efficient. For that reason, an advanced treatment method was introduced where membrane separation seem to be one of the promising method to solve this problem [10].

Membrane filtration process is recognized as one of the treatment method used in the textile wastewater treatment [11]. Reverse Osmosis (RO) and Nanofiltration (NF) are the examples of this technique that treat colored effluent for several commercial dyes. Nevertheless, these processes have setbacks where the permeability of these membranes is low. Therefore, a high operating pressure is required to obtain the desired throughput [12]. Thus, a modified membrane separation known as Micellar-enhanced Ultrafiltration (MEUF) can be used as an alternative method, where it required low operating pressure and high membrane permeability can be used in this process.

MEUF is a combination of high selectivity of reverse osmosis and high flux of ultrafiltration [13].The main feature of this process in MEUF is that the surfactant added to an aqueous stream at a concentration higher than its critical micelle concentration (CMC) forming aggregates called micelle. Then, the solutes with opposite micelle's charge tend to solubilize in the interior of the micelles. The size of the solutes increase and the solutes can be retained after trapped with the micelles whereby the untrapped solutes freely pass

Manuscript received August 4, 2012; revised September 17, 2012.

This work was supported by Fundamental Research Grant Scheme, from Ministry of Higher Education, Malaysia and Universiti Teknologi MARA, Malaysia.

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DOI: 10.7763/IJCEA.2012.V3.217

through the membrane [14]. The permeate stream contains water, free solute and surfactant.

The MEUF processes on dyes removal have been explored by many researchers. A. L. Ahmad and S. W. Puasa [12] had done research on removal of Reactive Orange 16 and Reactive Black 5 while N. Zaghbani, A. Hafiane, and M. Dhahbi [15], [16] had studied the removal of Eriochrome Blue Black R and Direct Blue 71 via MEUF process from an aqueous solution. Their works show that MEUF is a promising technique for dye removal. However, in actual condition the textile wastewater containing several mixtures of reactive dyes, therefore it is necessary to investigate on the competitive removal of reactive dyes mixture via MEUF.

This research focused on the removal of dyes mixture from an aqueous solution using MEUF. The dyes that have been chosen in this study were C.I Reactive black 5 (RB5) and C.I Reactive orange 16 (RO16) while cetylpyridium chloride (CPC) was selected as the cationic surfactant. The surfactant was characterized based on its critical micelle concentration (CMC) and micelle particle size. The investigation on the effect of surfactant concentration on MEUF of RB5, RO16 and their mixtures is based on the percentage of dye removal and permeate flux of the separation.

II. Materials and Methods

A. Experimental Materials

The surfactant CPC (MW 358.01), reactive dyes; RB5 (MW 991.82) and RO16 (MW 617.54) were purchased from Sigma Aldrich (M) Sdn. Bhd. The chemical structures for these materials are presented in Fig. 2, Fig. 3 and Fig. 4.

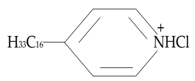


Fig. 2. Cetylpyridinium Chloride (CPC)

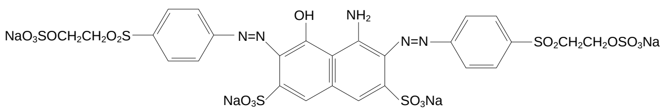


Fig. 3. C.I Reactive Black 5 (RB5)

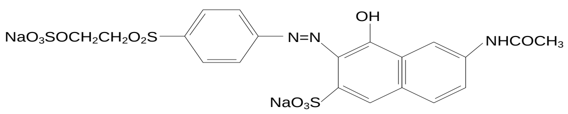


Fig. 4. C.I Reactive Orange 16 (RO16).

B. Membrane

A Cellulose Acetate membrane of molecular weight cut-off 20,000 obtained from Osmonics was used for the experiments.

C. Critical Micelle Concentration

The Ciritical micelle concentration (CMC) was determined from the concentration dependence of the specific conductivity of aqueous solution at 299 K via WTW 3420 Multimeter. The conductivity measurements were conducted by placing a known volume of micellar surfactant solution into a beaker, and successive injection of deionized water was added respective to the selected surfactant concentration [17]. The conductivity was measured where the solution was stirred via MR Hei-Tech Digital Hotplate stirrer whereby EKT Hei-Con temperature control was used to maintain the temperature of surfactant solution. The conductivity value was obtained after 5 minutes of injection to ensure the attainment of equilibrium in the system [18].

D. Micellar-Enhanced Ultrafiltration

All experiments were carried out at room temperature (25°C ± 2°C) in a batch stirred cell (model Sterlitech™ HP4750, USA). The MEUF experiments were conducted using unstirred dead end filtration. The dyes and CPC surfactant was mixed for 10 minutes with stirring speed of 300 rpm before it was loaded in the stirred cell. The operating pressure was controlled at 400 kPa (58 psig). The membrane was sonicated in deionized water (DI) water for 10 minutes at the end of each experiment. If the clean water flux through the membrane deviated within 5% of pure water flux, then it will be reused.

For single dye removal, the MEUF experiments used either RB5 or RO16 only while equal mass of RB5 and RO16 were used as the mixture. The dyes concentration were kept constant at 0.05 g/L throughout the experiment and the CPC concentration was varied at 0.5 g/L, 0.75 g/L and 1.0 g/L.

E. Analysis

Particle size analysis of CPC micelle formation was performed using Zetananosizer ZS90. The analysis was performed at 0.60 g/L of CPC. Concentrations of dyes in the feed and the permeate were measured by UV-Vis Spectrophotometer (UV-2100PC spectrophotometer). The wavelength at which maximum absorption occur was 598 nm for RB5 and 493 nm for RO16. Dyes rejection (R_d) and permeate flux (v_w) was calculated using equations (1) and (2):

$$R_d = \left(1 - \frac{C_{p_d}}{C_{0d}}\right) \times 100\% \quad (1)$$

$$v_w = \frac{(\Delta V)}{\Delta t . A} \quad (2)$$

where C_{0d} is feed dye concentration, C_{pd} is permeate concentration of dye, ΔV is cumulative volume difference, Δt is time difference, and A is membrane area [1].

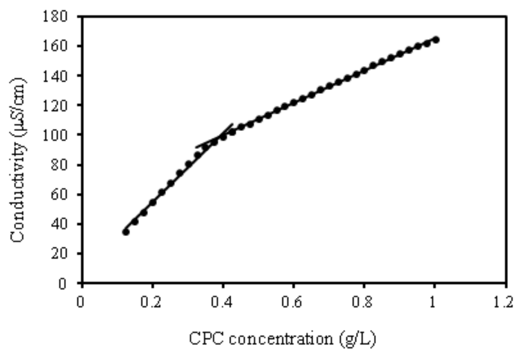


Fig. 5. Critical micelle concentration of CPC via conductivity method.

III. Results and Discussion

A. Critical Micelle Concentration

A representative plot of specific conductivity as a function of CPC concentration is shown in Fig. 5. From this figure, it demonstrates that the conductivity value increase rapidly at CPC concentration less than 0.38 g/L but then gradually increase with increase of CPC concentration.

This phenomenon can be explained where before the critical micelle concentration (CMC), the increment of the monomer surfactant conductivity increased with increment of CPC concentration. However, when it exceed the CMC value, the surfactant monomers form large amphiphilic aggregate micelles [15]. As micelle formed, some of the hydrophobic tail will bind together to form micelle where the increment of specific conductivity with addition of CPC concentration increases in different manner hence changing the slope of the graph (the straight line corresponds to the surfactant above CMC) [19], [20]. As a result, the change of slope is noticeable. This observation notifies the formation of micelles in surfactant solution. The change in slope at certain surfactant concentration permits the determination of CMC [20]. The CMC of CPC obtained from this study is 0.38 g/L which is consistent with the CMC value available in the literature (0.322 g/L) [21].

B. Particle Size Analysis

The particle size analysis was performed at CPC concentration of 0.6 g/L. The result obtained is shown in Fig. 6. It indicates the diameter of surfactant micelles varies from 2.3 nm to 6.5 nm. The highest percentage number of particles obtained was 23.9% at of 4.2 nm. According to M. K. Purkait, S. DasGupta, and S. De [21] the typical spherical diameters of CPC micelles are 5 nm, which in similar agreement with results obtained from this study.

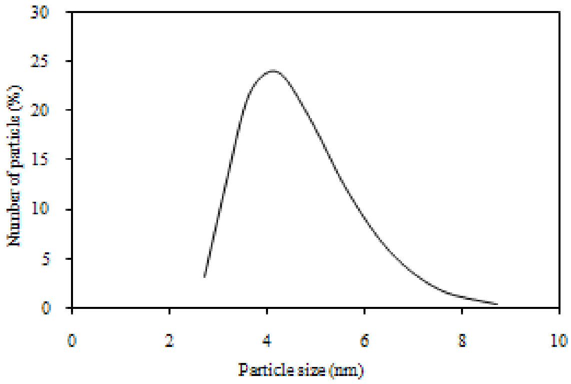
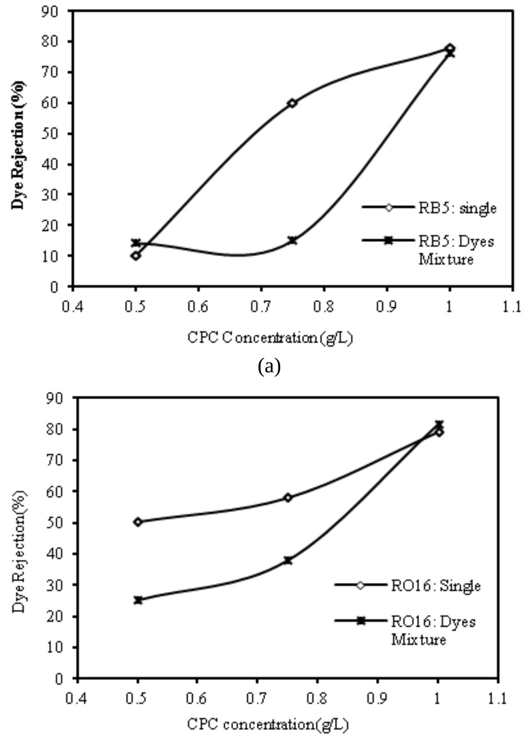


Fig. 6. Size distribution by number with particle diameter for CPC at 0.6 g/L

C. Micellar-Enhanced Ultrafiltration

The variation of dye rejection between single reactive dye (RB5 and RO16) and dyes mixture (mixed RB5 and RO16) at CPC concentration range between 0.5 g/L to 1.0 g/L are shown in Fig. 7. It can be observed that dyes rejection (RB5 and RO16) for MEUF both single and dyes mixture increased as CPC concentration increased. This phenomenon is due to the increased number of micelles in the solution as the feed of CPC concentration increased, whereby it enhances the overall dye solubilization. Dyes and surfactant with opposite charge has tendency to be absorbed in micelle. Consequently, the dyes trapped in the micelle which in turn will increase in size and retained on membrane surface thus leaving only unsolubilized dyes and surfactant pass through the membrane to the permeate side [15]. The highest RB5 dye rejections for single and mixed dyes obtained were 78% and 76.35% respectively while the highest RO16 dye rejections for single and mixed dyes were 79.18% and 81.64% respectively at CPC concentration of 1.0 g/L.

In the case of MEUF with single dye, it was found that the dye rejection of RO16 is higher as compared to RB5. It can be observed that dye rejection for RB5 and RO16 at CPC concentration of 0.5 g/L were 10.1% and 50.2%, respectively. The higher rejection of RO16 dye is due to the effect of tendency of surfactant micelles which have more tendency to solubilizes with RO16. A. L. Ahmad and S. W. Puasa [12] reported that the solubility differences of the dyes are based on chemical structures where the structure of RB5 and RO16, each have four sulphonic acid (SO^{3-}) and two sulphonic group respectively. Therefore, RB5 dye has higher water solubility compared to RO16. In micellar solution, the solute with a higher solubility shows a lower solubilization constant than the solute with a lower water solubility. This statement proved that RO16 have greater tendency to solubilizes in the micelles than RB5, hence resulted in higher dye rejection of RO16 dye as compared to that of RB5 dye.



(a)

(b)

Fig. 7. Dyes rejection (RB5 and RO16) for MEUF of single dye and dyes mixture after 1 hour of experiments at CPC concentration range between 0.5 g/L to 1.0 g/L.

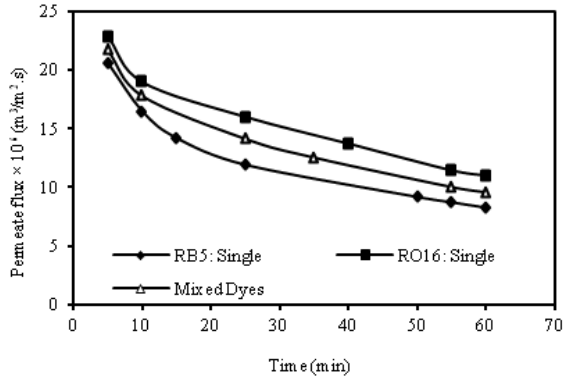


Fig. 8. Variation of permeate flux with time for MEUF of single dye and mixed dyes for RB5 and RO16

Fig. 7(a) shows that the dye rejection for MEUF of RB5 only increased rapidly from 10.1% to 60% as the CPC concentration increased from 0.5 g/L to 0.75 g/L, then the increased gradually to 78% (CPC concentration of 1.0 g/L). As CPC concentration increased, more RB5 dyes will be solubilized in micelles. Micelles with solubilized RB5 will retain on the membrane surface, hence resulting in increment of RB5 rejection. However, dye rejection of MEUF of single RO16 increased gradually from 50.2 % to 58% as the CPC concentration increased from 0.5 g/L to 0.75 g/L; which is slightly different as compared to MEUF of RB5. The comparison between these two trends can be explained based on the different in molecular weight of RB5 (MW 991.82) and RO16 (MW 617.54). As the concentration of CPC increased from 0.5 g/L to 0.75 g/L, more RB5 solubilized in CPC micelles, hence increased the average molecular weight of RB5-CPC micelles. This result in sharp increment of RB5 rejection.

In MEUF with dyes mixture, the dyes rejection for RB5 and RO16 was quite low as compared to MEUF of single dye. For MEUF of dyes mixture, the increment of dye rejection for RB5 is too small (from 14.25% to 15.2%) while dye rejection of RO16 increased gradually (from 25.2% to 38.0%) at CPC concentration 0.5 g/L to 0.75 g/L. This observation is due to the competitive solubilization of RB5 and RO16 in CPC micelles at limited number of micelles formed. However, as CPC concentration increased to 1.0 g/L, the dye rejection for both RB5 and RO16 increased rapidly which is similar to those dye rejection obtained from MEUF of single dye. This observation confirmed that the adequate number of micelles formed was achieved at CPC concentration of 1.0 g/L.

Fig. 8 depicted the permeate flux profile with time for MEUF of single and dyes mixture for RB5 and RO16 at operating pressure of 400 kPa and CPC concentration of 1.0 g/L. It can be seen that the permeate flux declined over the time of operation where a layer of surfactant micelles has formed over the membrane surface thus reducing the flow of the solvent. However, the comparison between single dye and mixed dye clearly indicate that the permeate flux for MEUF of RO16 only > RB5-RO16 > RB5 only. This is due to the different of average molecular weight obtained from solubilization of dyes into CPC micelles. Dyes-CPC micelles with higher average molecular weight will create more deposited layer of the micelles aggregates over the membrane surface, hence resulting in the decrement of permeate flux. Theoretically, the average molecular weight of RB5-CPC micelles > RB5-RO16-CPC micelles > RO16-CPC micelles. A. L. Ahmad, S. W. Puasa, and M. M. D. Zulkali [1] reported that dyes with lower molecular weight can easily pass through the membrane. Meanwhile dyes with higher molecular weight will be retained on the membrane surface. Therefore, it is an evident that the RB5-CPC micelles had lowest permeate flux as compared to others because of the molecular weight factor.

From Fig. 9, it is clearly shown that the permeate flux for MEUF in all cases decreases with increasing feed CPC concentration. The flux decline trend is due to the increase of permeate flux resistance since most of the micelles were retained on the membrane surface in MEUF process. As discussed previously, the surfactant micelles formed large aggregates and generates a deposited layer on the membrane surface which increased the resistance against the permeate flux through the membrane. Channarong et.al. [22] reported that the reduction of flux is caused by concentration polarization and formation of micelles sludge cake at the membrane surface that bring negative effect on the MEUF process.

It was found that permeate flux for MEUF of RB5 only is higher at CPC concentration of 0.5 g/L as compared to both MEUF of RO16 only and RB5-RO16 mixture. As discussed earlier, when less amount of RB5 solubilized in CPC micelles, less amount of gel layer formed hence resulting in high permeate flux but low in RB5 rejection (10.1%).

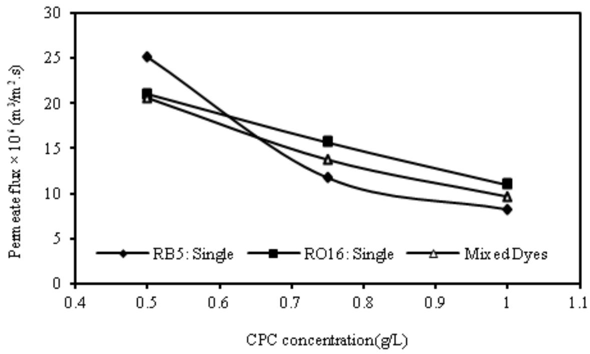


Fig. 9. Permeate flux for MEUF of single dye and dyes mixture for RB5 and RO16 after 1 hour of experiment at CPC concentration range between 0.5 g/L to 1.0 g/L

IV. Conclusion

The critical micelle concentration of CPC obtained was 0.38 g/L measured by conductivity method. Based on particle size analysis, the diameter of micelles formed ranges from 2.3 nm to 6.5 nm. The MEUF process was performed at constant operating pressure of 400 kPa with surfactant concentration varies from 0.5 g/L to 1.0 g/L. Results obtained in MEUF study shows that the highest dye rejections obtained was 78.0% and 79.2% of RB5 and RO16 for single dye removal while 76.4% and 81.6% of RB5 and RO16 for RB5-RO16 mixture removal from aqueous solution. This study reveals the existence of competitive removal between RB5 and RO16 dyes from aqueous solution via MEUF process.

Acknowledgment

This work was financially supported by the Fundamental Research Grant Scheme, from Ministry of Higher Education, Malaysia and Universiti Teknologi MARA, Malaysia.

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