

*Article*

## Evaluation of Micellar-Enhanced Ultrafiltration (MEUF) Membrane for Dye Removal of Synthetic Remazol Dye Wastewater

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**Abstract.** Micellar-Enhanced Ultrafiltration Membrane (MEUF) is one of promising technology to separate low molecular weight substances such as dyes. The MEUF process involves a combination of ultrafiltration and surfactant at a concentration higher than surfactant's Critical Micelle Concentration (CMC). This research was focused on the application of MEUF for synthetic Remazol dye wastewater containing Remazol Yellow/Black B/ Turquoise Blue at different dye concentration. In the MEUF system, a PolyEther Sulphone Ultrafiltration membrane and Cetyl Pyridinium Chloride (CPC) surfactant at concentration of 2 times of its Critical Micelle Concentration were used. Evaluation of MEUF performance was determined based on flux profile and rejection. It was confirmed that dye concentration did not change the flux profile significantly. Flux reduction was effected by the addition of surfactant in the MEUF system. Flux decline in the MEUF was due to the formation of surfactant micelle aggregates with dye generating a deposited layer on the membrane surface. As the CPC is an ionic surfactant, the formation of the micelle is attributed to a balance between the attraction of hydrocarbon chain (hydrophobic tail) and ionic repulsion. Scanning Electron Microscopy images verified that there was an additional layer on the membrane surface after MEUF was performed, indicating a deposited layer. The permeate rejections were more than 70% and 96% for rejection of Chemical Oxygen Demand and Remazol dye, respectively. The rejection of dye ion and Chemical Oxygen Demand were attributed due to anionic dye solubilization in the surfactant micelles. The solubilization was formed by adsorption through electrostatic attraction.

**Keywords:** Remazol dye, ultrafiltration, surfactant, micellar.

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## 1. Introduction

Batik has been known as an Indonesian Cultural Heritage by the UNESCO. Numerous batik varieties were designed and usually were produced in a home industry. After the UNESCO recognized batik as an Indonesian cultural heritage, the development of batik industries grown more rapidly. That event promoted the prestige of Batik in Indonesia and International World so that the demand of Batik increase significantly. Along with the development of batik industry in Indonesia, the batik processes generate wastewater which is an undesirable environmental impact. In batik production, reactive dyes such as Remazol, indigosol, naphtol and rapid are used in the dying process. As a consequence, the batik wastewater has the characteristic of high content of reactive dye as well as wax and oil, pigments, salts, and diazonium. Different methods have been proposed to remove dissolved organic dyes from waste water. The most studied technologies were chemical coagulation, biological treatment, electrochemical oxidation, ozonation, activated carbon adsorption and membrane process. All the technologies have been reviewed comprehensively [1-3]. Among the membrane separation technologies, reverse osmosis (RO) [4] and modified nanofiltration (NF) [5] are the best available techniques for the separation of several commercial dyes. However, their major disadvantage are low membrane permeabilities and to get the desired throughput (permeate flux), a high operating pressure is required [6].

Micellar-Enhanced Ultrafiltration (MEUF) is a modified separation of ultrafiltration (UF) process. Besides requires a low pressure, this process also involves the addition of a surfactant with a concentration above the Critical Micelle Concentration (CMC). In this concentration, the surfactant monomers will assemble and aggregate to form micelles having a hydrodynamic diameter larger than the pore diameter of ultrafiltration membrane. The contaminant molecules entrap and solubilize in the micelle. Therefore, the solubilized dye molecules in the micelle will be more difficult to penetrate into membrane pores. This allows greater rejection as well as lowers the permeate flux and hence makes it possible to separate the solute that could not be separated by the ultrafiltration membranes. In addition, this technique combines the high selectivity of Reverse Osmosis and the high flux of ultrafiltration. The separation principle of the MEUF is illustrated in Fig. 1.

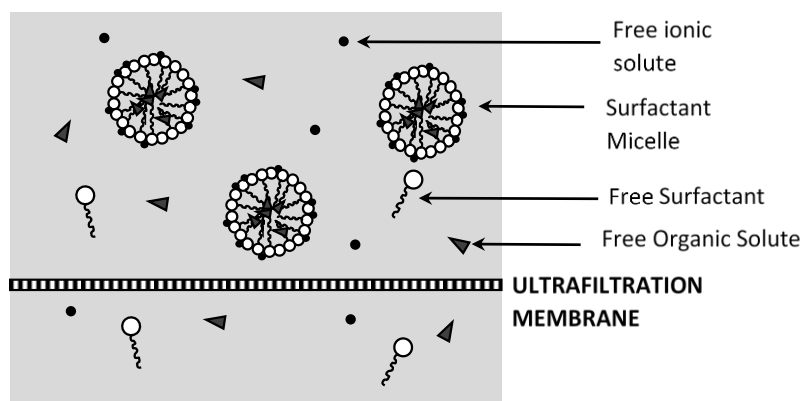


Fig. 1. Schematic illustration of MEUF separation principle

To our best knowledge, most of the previous studies merely focused on the effects of variables on the removal or separation of methylene blue [7-9], Eriochrome Blue Black [10], a mixture of Reactive Black and Orange [11] and eosin [12]. This study is focused on the application of MEUF for separation of Remazol reactive dyes such as Remazol Yellow, Remazol Black B, dan Remazol Turquoise Blue. The performance of MEUF was determined by the flux profile and the dyes rejection. When the MEUF is able to remove reactive dyes, then this technology can be applied for batik wastewater treatment.

## 2. Materials and Method

### 2.1. Materials

Remazol Yellow (RY), Remazol Black (RBA) and Remazol Blue (RBU) produced by Hoechst were used as model dyes. Chemical structures of Remazol dyes are shown in Fig. 2. Cetyl Pyridinium Chloride,  $C_{21}H_{38}ClN$  (Sigma Aldrich) having Molecular Weight (MW) of 358 with Critical Micelle Concentration (CMC) of 0.90 mM [13] was selected as a surfactant. Polyethersulphone (PES), PolyEthylene Glycol (PEG) and N-Methyl Pyrrolidone (NMP) were materials for membrane preparation. The PES material was Veradel3100P from Solvay.

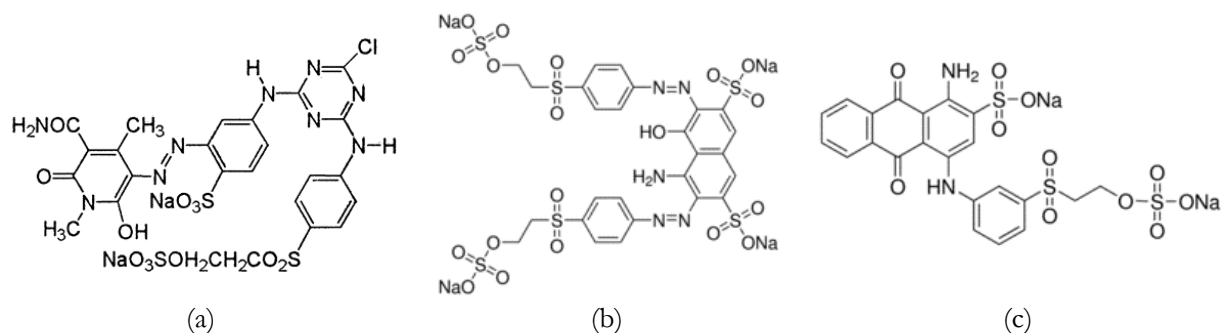


Fig. 2. Chemical structure of Remazol dyes: (a) Remazol yellow; (b) Remazol black; (c) Remazol blue.

### 2.2. Methods

#### 2.2.1. Membrane preparation

Flat sheet ultrafiltration membrane was prepared through a non-solvent induced phase separation method. The PolyEthylene Glycol (PEG) and N-Methyl Pyrrolidone (NMP) were the additive and solvent, respectively [14]. The membrane was prepared by dissolving 12% w of PES into 83% w of NMP. Then, 5% of PEG was added into a solution containing PES and NMP. The solution was homogenized by stirring using a magnetic stirrer. Further, the solution was left to complete release of bubbles. The solution was cast into casting machine at a thickness of 200  $\mu\text{m}$  and then being immersed into a coagulation bath filled with distilled water for 1 hour. When the membranes were formed, they were then taken out from the coagulation bath and being immersed into the next coagulation bath for 24 hours. After 24 hours, the membranes were dried in a 40  $^{\circ}\text{C}$  oven for 24 hours and then ready for membrane characterization.

#### 2.2.2. Micellar-enhanced ultrafiltration membrane

Experiments were carried out by a laboratory-scale MEUF system. Schematic diagram of the MEUF is illustrated in Fig. 3.

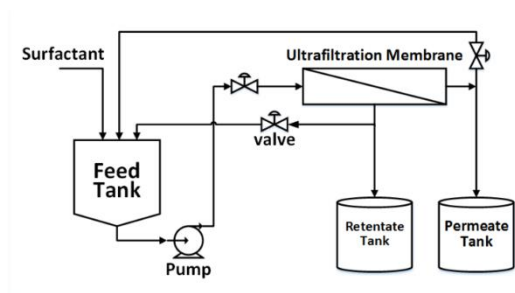


Fig. 3. Schematic diagram of micellar-enhanced ultrafiltration membrane system.

All experimental runs were conducted at room temperature ( $29 \pm 2^\circ\text{C}$ ). Before starting the experiments, membranes were first compacted by filtering water through the membrane at a pressure of 1 bar for 30 minutes. For each run, a new circular membrane sheet with an area of  $13.85 \text{ cm}^2$  was used. Pure water flux ( $J_0$ ) was obtained by weighing analytically permeate collected at certain recorded time. In the MEUF system, the feed was a solution comprising a mixture of surfactant, dye, and water at a certain concentration. In this study, the concentration of Cetyl Pyridinium Chloride (CPC) surfactant was 2 times of its CMC concentration and concentration of dyes were  $90 \text{ mg/L}$  and  $120 \text{ mg/L}$ . The MEUF was operated by filtering feed solution at a temperature about  $24 - 25^\circ\text{C}$  and 1 bar operating pressure. Permeate fluxes ( $J$ ) were determined by measuring the volume of permeate collected at 5 minute intervals for 120 minutes and calculated by using Eq. (1).

$$J = \frac{W}{A.t} \quad (1)$$

where  $W$  representing total weight of permeate,  $A$  is the membrane area and  $t$  is a time interval.

The filtrations were conducted at total recycle mode where both permeate and retentate were recycled back to the feed tank in order to maintain same concentration. After each flux determination, the collected permeate was returned back to the feed tank. Further, normalized fluxes are obtained by dividing fluxes ( $J$ ) with initial water fluxes ( $J_0$ ). After 120 minute operation, a permeate of 10 ml was collected and analyzed.

## 2.3. Analysis

### 2.3.1. Membrane characterization

Membrane morphology was visualized using a Scanning Electron Microscope (FIE-Inspect S50, USA) with a magnification of 5,000 and 10,000 times. The outer surface of the sample was coated with Gold or Palladium. Chemistry of the membrane surface was analyzed by FT-IR Prestige-21 (Shimadzu, Japan).

### 2.3.2. Membrane rejection

The efficiency of filtration for dye removal from the feed solution was evaluated through the dye and Chemical Oxygen Demand (COD) rejection. The rejection ( $R$ ) was calculated by Eq. (2).

$$R = \left( 1 - \frac{C_p}{C_f} \right) \times 100 \quad (2)$$

where  $C_p$  and  $C_f$  are a concentration of dye or COD value in the permeate and in the feed, respectively.

### 2.3.3. Determination of dye content and COD value

The concentration of RY, RBA and RBU were determined by UV-Vis Spectrophotometer at a wavelength of 416 nm, 640 nm, and 660 nm, respectively, with ultrapure water as the reference solution. COD values were measured by using COD Meter (HANA HI 839800).

## 3. Result and Discussion

### 3.1. Membrane Characteristics

#### 3.1.1. Membrane surface chemistry

Membrane surface chemistry was characterized by functional specific groups of membrane based on FT-IR spectrum as presented in Fig. 4. FT-IR spectra show peaks at  $1492.9$  and  $1589.3 \text{ cm}^{-1}$  which are characteristic of PES membrane [15, 16]. The peak of  $1492.9$  and  $1589.3 \text{ cm}^{-1}$  indicate the presence of aromatic components (C-C stretching) in the benzene ring. The peak of  $1219.01$  and  $1261.45 \text{ cm}^{-1}$  specify

aromatic ether compounds. Moreover, the peaks of 1161.15 and 1172.7 show a symmetric stretching sulfur  $\text{SO}_2$ . In addition, the peak of 842.9 and 862.2  $\text{cm}^{-1}$  are a sign of the para-substituted benzene, which is appropriate with the chemical structure of the PES. The peak observed around 1074.3, 1093.6 and 1114.8  $\text{cm}^{-1}$  are characteristic of  $(-\text{C}-\text{O}-\text{C}-)$  bonding. Moreover, the peak of 1492.9 and 1589.3  $\text{cm}^{-1}$  indicate the presence of  $(\text{C}-\text{C})$  bonding, while the peak of 2872.01 and 2926.01  $\text{cm}^{-1}$  exhibit C-H asymmetric bonding. Furthermore, the absorbance peak at 3375.43 and 3475.73  $\text{cm}^{-1}$  denote the O-H alcohol bonding. The dominant peak caused by bonding vibration O-H, C-H asymmetric, C-O-C, and C-C are the type of constituent bonds in the PEG polymer [17].

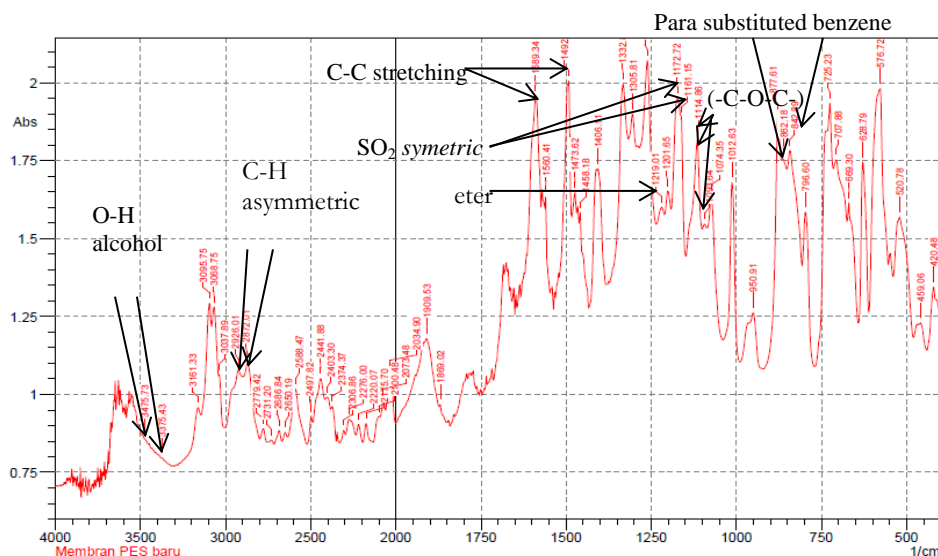


Fig. 4. FT-IR spectra of polyethersulphone membrane.

### 3.1.2. Membrane morphology and structure

Morphology of membrane surface and cross section of membrane structure were displayed in Fig. 5.

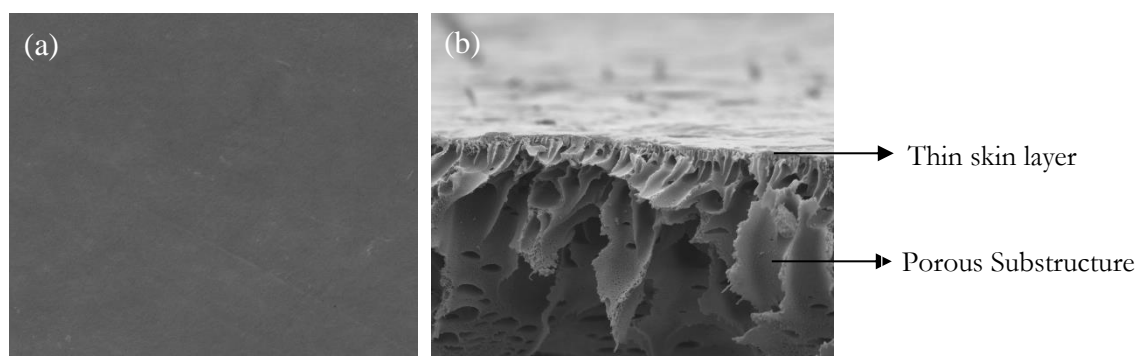


Fig. 5. SEM images of (a) Membrane surface at 10,000 x magnification; (b) Cross-sectional structure at 5,000 x magnification.

Figure 5(a) shows that the membrane pores are not visible on the membrane surface. In addition, the visualization of the membrane surface is spotless without any deposit layers. The cross-sectional structure of the membrane as displayed in Figure 5(b) shows an asymmetric or anisotropic membrane structure. The structure is identified by two layers comprising thin skin layer on the surface and porous substructure at the bottom. Selectivity and permeability of membrane were determined by the thin skin layer and typically having a thickness of 0.2 to 1  $\mu\text{m}$ . On the other hand, the porous substructure layer at the bottom has a function as a membrane support [18].

### 3.2. Flux Profiles

Figure 6 shows normalized flux profiles with time at various Remazol dyes. Figure 6 presents declining normalized fluxes over the time during MEUF and thereafter the fluxes remain constant. This is a usual phenomenon in membrane separation process since the accumulation of solute (dye) resulting in a flux decline. The flux decline was more pronounced for the MEUF since in the MEUF the surfactant micelle aggregates with dye generated a deposited layer on the membrane surface. The layer on the membrane surface was described as concentration polarization phenomenon and was found had a correlation with the MEUF [19]. The concentration polarization on the membrane surface increases with time. As a consequence, the osmotic pressure at the membrane solution interface enhances and has an effect on reduction of driving force for the permeating solution. Surfactant aggregates lead to a deposited layer on the membrane surface and rise the membrane resistance. As a consequence of higher membrane resistance, a decline of permeate flux was observed [20]. Huang et al. [21] also found an enhancement of additional assistance ( $R_f$ ) with the increase of surfactant concentration. Trends of declining flux with filtration time were also observed for ultrafiltration of CPC solution with phenolic derivatives [22] and eosin dye [23] as well as MEUF of Methylene Blue [24]. According to Fig. 6, it reveals that the RBA had the highest normalized flux compared to RBU and RY. This was assumed due to the different average molecular weight of dye solubilized into surfactant micelles, as reported by Puasa [11]. It was found that dye-micelle having higher average molecular weight generated a more deposited layer of the micelle aggregates.

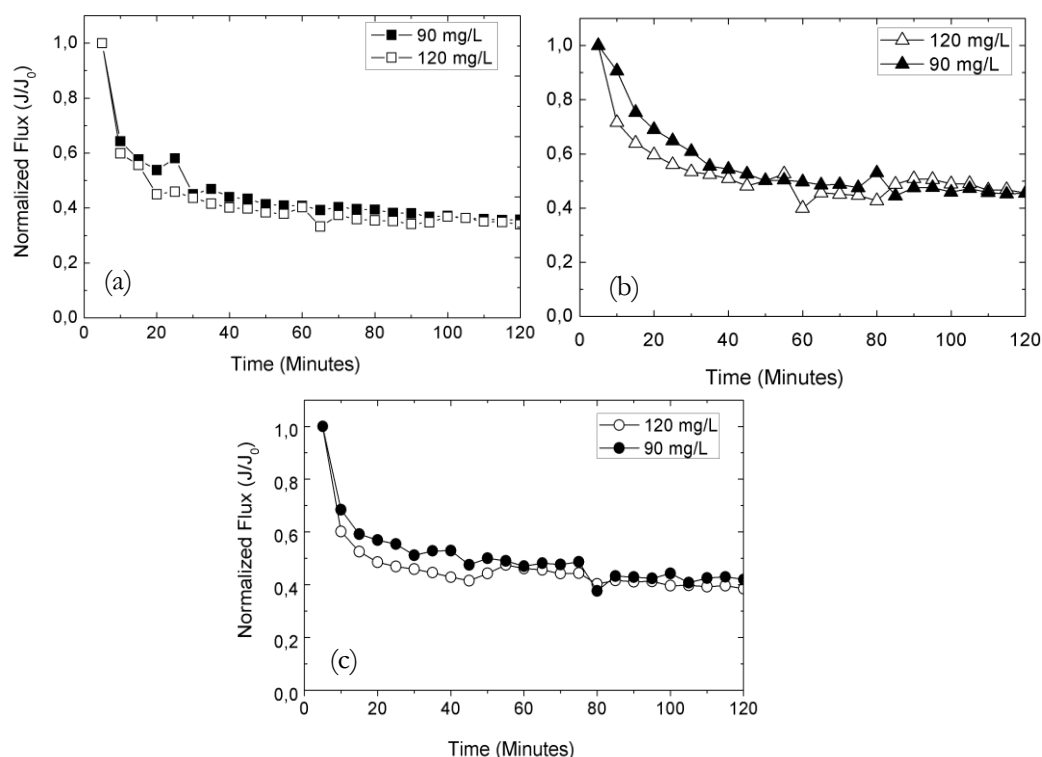


Fig. 6. Normalized flux profile of MEUF membrane for dye removal at dye concentration of 90 mg/l and 120 mg/l: (a) RY, (b) RBA (c) RBU

In addition, Fig. 6 illustrates that effect of dye concentration and type of Remazol dye were not significant. Both variables result in similar normalized flux for about 0.4 after 120 minute filtration. In the MEUF system, dye particles were bonded with surfactant providing large aggregates of surfactant micelles, and hence the surfactant characteristics (concentration, type) were taking more account than the type of solute (dye). Previous research using actual batik wastewater was published and found similar tendency [14]. Results presented in Fig. 4 were supported with SEM images of the membrane after MEUF as displayed in Fig. 7 (dye concentration of 90 mg/l) and Fig. 8 (dye concentration of 120 mg/l).

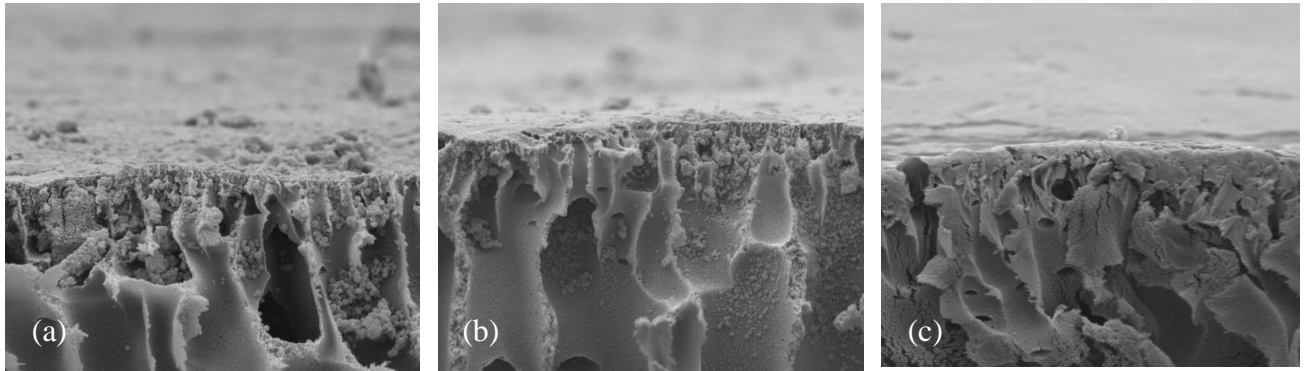


Fig. 7. Cross-sectional SEM images of membrane after MEUF (magnification of 5000x) at dye concentration of 90 mg/l: (a) RY, (b) RBA (c) RBU.

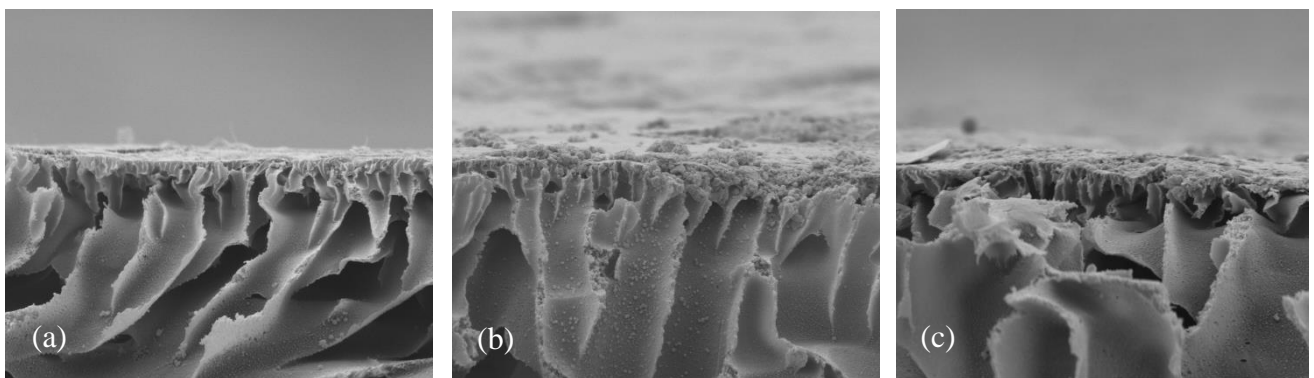


Fig. 8. Cross-sectional SEM images of membrane after MEUF (magnification of 5000x) at dye concentration of 120 mg/l: (a) RY, (b) RBA (c) RBU.

Figures 7 and 8 illustrate that the thin skin layer of membrane covered with aggregates of surfactant micelle and some of the aggregates passed into membrane porous structures. However, different layer thickness for each dye type and concentration were not observed. This result supports the explanation of flux decline profiles in Fig. 6. In order to verify the effect of surfactant addition in lowering flux, dye solution only (without surfactant) was filtered by ultrafiltration membrane as presented in Fig. 9.

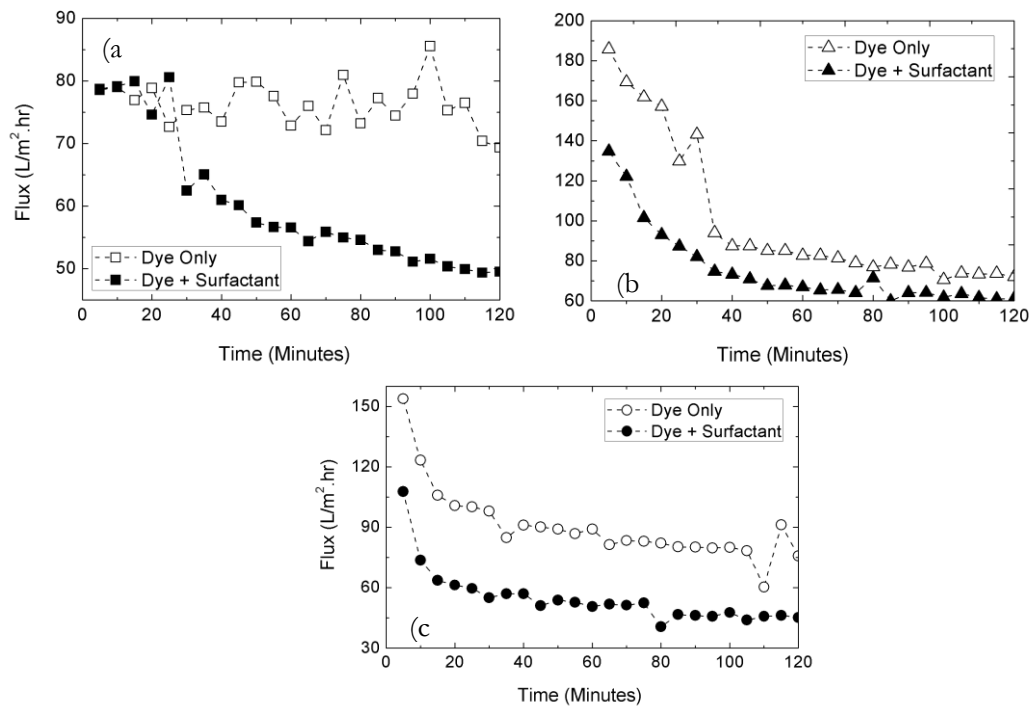


Fig. 9. Flux profile of dye and mixture of dye and surfactant ultrafiltration at dye concentration of 90 mg/l: (a) RY, (b) RBA (c) RBU

Based on Fig. 9, it was confirmed that addition of a surfactant decreased flux profile. Ultrafiltration of dye solution only generates a higher flux than ultrafiltration of dye-surfactant mixture. Similar results have also been explained by another study [23, 24, 25]. The surfactant has a noticeable effect in reducing membrane fluxes since it tends to be deposited on the membrane surface. Figure 10 displays SEM images of membrane surface after being used for filtering solution containing dye only, surfactant only and a mixture of dye and surfactant. The figure confirms that surfactant deposition onto the membrane surface appeared like a gel network. On the other hand, the dye was not retained by the membrane and hence dye deposition on the membrane surface was insignificant.

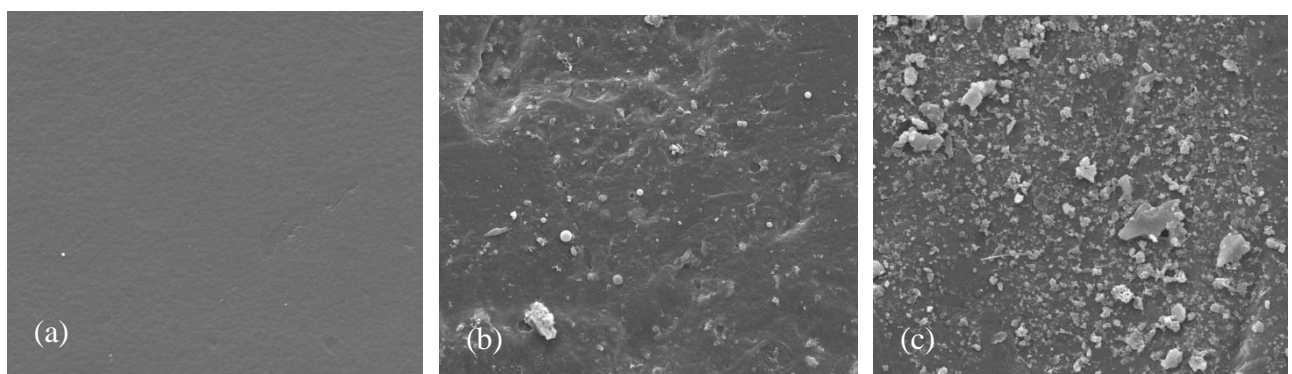


Fig. 10. SEM images of membrane surface after filtration of a solution containing: (a) Dye only (RY at a dye concentration of 90 mg/L), (b) Surfactant only, (c) Mixture of dye and surfactant (RY at a dye concentration of 90 mg/L). Images were visualized at magnification of 5,000 times.

### 3.3. Membrane Rejection

Membrane rejection is a usual parameter to evaluate membrane selectivity. The membrane selectivity determines membrane ability to resist a substance or to let pass other substance. Membrane selectivity is



subject to some variables such as membrane-substance interaction, particle or substance size, and membrane pore size. When feed comprises more than one component, the rejection of each component might be different.

### 3.3.1. Rejection of dye

Figure 11 depicts dye rejection of MEUF at various type of Remazol dye.

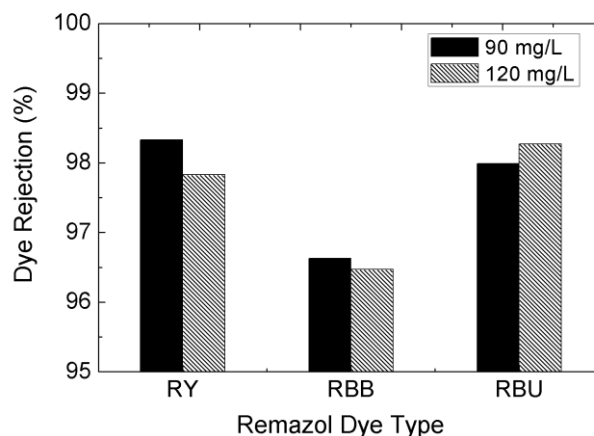


Fig. 11. Dye rejection of MEUF (Dye concentration in feed are 90 mg/l and 120 mg/l at various Remazol dye)

Figure 11 shows that dye rejection for all Remazol dye and concentration were more than 96%. High rejection of Remazol dye by the MEUF, representing that the concentration of dyes in the permeate were significantly reduced (Table 1).

Table 1. Concentration of dye in the permeate at various dye concentration in feed corresponding to Fig. 11.

Dye	Average Dye Concentration in permeate (mg/l)	
	90 mg/l	120 mg/l
RY	1,77	2,895
RBA	1,905	2,165
RBU	3,04	4,26

High rejection in correlation with the low concentration of dye in the permeate indicates that Remazol dye was entrapped on the surfactant micelles and retained by the membrane. In addition, Fig. 11 illustrates that comparing to other Remazol dye, the RBA was found to have the smallest rejection. This is probably due to the effective solubilization of RBA by the CPC micelles was lesser than that of other Remazol dye. The degree of solubilization of solute in surfactant micelle is characterized by the solubility of the solute in water, dipole moment and octanol-water partition coefficient [26].

Formation of ionic micelle surfactant is attributed to a balance between the attraction of hydrocarbon chain (hydrophobic tail) and ionic repulsion. At the micellar surface, a stern layer is formed as a result of counterions large fraction associated with the micelle. The associated counter ion and the micelle indicate the net charge of the micelle is a smaller amount than the degree of micellar aggregation [27]. Micelle formation can have different sizes, aggregation numbers, and shape. The micelles size in a surfactant solution was found in the range of 10-100 Å. The shape of micelles in aqueous ionic surfactant solution is changed from spherical-cylindrical-hexagonal and lamellar as concentration increase in surfactant solution [28]. However, specific information regarding the size and structures of micelles at different concentration for CPC surfactant was not found. Variation of micelle's aggregation number is subjected to the type of surfactant. In ionic surfactant, a cluster of surfactant for micelle aggregation is generated from 10-100

molecules. This is a less value compared to the nonionic surfactant (more than 1000 molecules) due to the effect of ionic charge [29]. The ionic charge produces an electrostatic repulsion among head-groups which have a tendency to break down the particles. The aggregate of ionic surfactant micelles involves a compressive core surrounded by a less compressive surface structure [27].

Proposed rejection mechanism of metal ions and organic solutes was described by Li et al. [30]. It was explained that the solubilization of organic solutes takes place in a different location in the micelles (surface or palisade region). When the surfactant is cationic, it consists of a surface with cationic charge and a hydrophobic core. Remazol dye is an anionic organic solute and the solubilization of its dye is by an adsorption on the surface of oppositely charged micelle [10, 30, 31]. Mechanism of anionic dye solubilization in surfactant micelle is illustrated in Fig. 12.

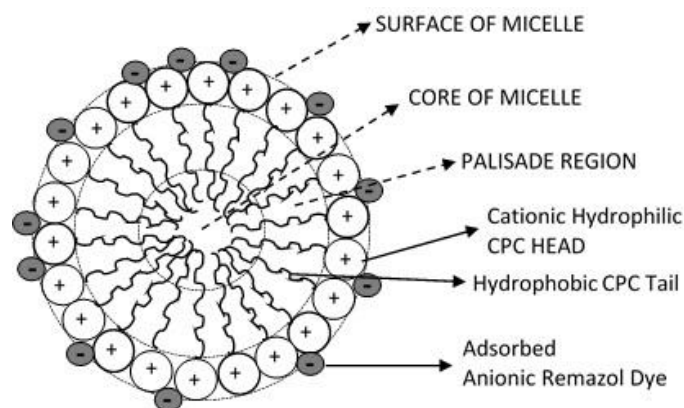


Fig. 12. Schematic illustration of Remazol Dye solubilized in surfactant micelle.

The adsorption of anionic dye on the head of cationic micelle was formed through electrostatic attraction [32, 33]. Ions are attached to the micelle near the micellar surface and do not penetrate into the non-polar part (hydrophobic tail) of the micelle [27]. In more detail, charged micelles interaction were usually explained in the framework of the DLVO Theory. It was elucidated that creation of electric double layer close to the charge surfaces determined the effective interaction between the micelles. The electric double layer of the micelle is generated as a result of anionic adsorption on the micelle surface. Theoretically, the layer was caused by interplay entropic effects supported by the homogenous distribution of the mobile charges and the electrostatic attraction between surface charge and their counterions in the aqueous environment. Moreover, the form of effective interaction adopted a Yukawa-type pair potential [29].

### 3.3.2. Rejection of chemical oxygen demand (COD)

COD is one parameter to determine wastewater quality. The COD corresponds to an Oxygen requirement to degrade biodegradable and non-biodegradable organic compounds. Figure 13 presents COD Rejection of MEUF and concentration of COD in permeate at dye concentration of 90 mg/l and 120 mg/l at various Remazol Dyes.

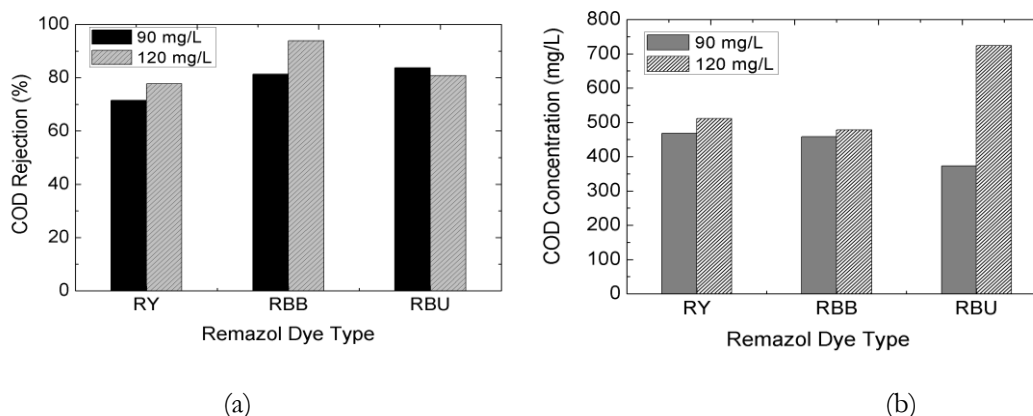


Fig. 13. (a) COD Rejection of MEUF, and (b) Representation of COD concentration in the permeate, dye concentration in the feed: 90 mg/l and 120 mg/l at various type of Remazol dyes. Initial COD in the feed were in the range of 6553-9992,8 mg/l and 9337,8-20636 mg/l for feed containing surfactant and dye concentration of 90 mg/l and 120 mg/l, respectively.

Figure 13 presents a decrease of the COD level when filtering mixture of the dye solution and surfactant. Low level of COD in the permeate indicates that the dye molecules have been solubilized or bounded by the surfactant micelles. Interaction of surfactant micelle-dye was further retained by the membrane. Consequently, a smaller concentration of dye and surfactant were found, which were characterized by low levels of COD [19, 26]. The MEUF has been applied for treatment of soil washing solution and it was found that the COD level was around 80% reduced [34].

#### 4. Conclusion

Micellar-Enhanced Ultrafiltration Membrane (MEUF) has been applied for removing synthetic Remazol dye waste water. The synthetic wastewater was a mixture of Remazol Black B/Yellow/Turquoise Blue at concentration of 90 mg/L and 120 mg/L. As generally found in membrane filtration, permeate fluxes decreased with the increase of time filtration. However, variation in dye concentration did not change the flux profile significantly. In this research, further flux decrease was mainly due to the addition of surfactant in the MEUF system. Evaluation of Remazol dye concentration and Chemical Oxygen Demand (COD) level in the permeate showed a reduction of dye concentration and COD level. Permeate rejection of COD level and Remazol dye concentration were found as more than 70% and 96%, respectively.

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