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Micellar enhanced ultrafiltration (MEUF): activated carbon fiber (ACF) hybrid process using low surfactant concentration for zinc(II) removal from synthetic wastewater

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ABSTRACT

Zinc(II) removal from synthetic wastewater by micellar enhanced ultrafiltration (MEUF) using low sodium dodecyl sulfate (SDS) concentration and 300 kDa molecular weight cut-off (MWCO) membrane was studied. Effects of various parameters like initial permeate flux, retentate pressure, molar ratio of zinc(II) to SDS, initial zinc(II) concentration, MWCO, and solution pH were analyzed. Considering zinc(II) removal rate and permeate flux, initial permeate flux of 43.64 $L/m^2 h$, retentate pressure of 0.20 MPa, molar ratio of zinc(II) to SDS of 1:2, and neutral pH condition were found to be optimum operating parameters. Although initial SDS concentrations were far below critical micelle concentration, average zinc(II) removal rate was 77.29 and 75.93% whereas, absolute permeate flux was 39.64 and 10.45 $L/m^2 h$ at molar ratios of zinc(II) to SDS of 1:8 and 1:2, respectively. It was observed that concentration polarization played a crucial role in zinc(II) removal. In MEUF-ACF hybrid process at constant initial zinc (II) concentration (0.42 mM) and initial SDS concentration (0.21 mM), average zinc(II) removal rate was 8.65, 12.70, 10.84%, while SDS removal rate was 10.5, 12.6, 11.52% higher than MEUF alone in 30, 100, 300 kDa membranes, respectively.

Keywords: Micellar enhanced ultrafiltration; Sodium dodecyl sulfate; Activated carbon fiber; Concentration polarization

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1. Introduction

Heavy metal pollution is one of the major problems of this rapidly booming industrialized, modernized, and concretized society, resulted as the consequences of anthropogenic industrial and agricultural activities that can pose serious threat to all the living organisms. Among various heavy metals, zinc(II) is potentially toxic elements (PTEs) found abundantly in Earth's crust. In terms of application, zinc(II) is used in galvanization processes, brass and bronze alloy production, tyre, batteries, paints, plastics, rubber, fungicides, paper, textiles, taxidermy, building materials and special cements, dentistry, cosmetics, and pharmaceuticals [1]. Zinc(II) is considered by the United States Environmental Protection Agency (USEPA) as one of

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the 13 important toxic metals among a list of common organic and inorganic pollutants in wastewater. National secondary drinking water regulations had instituted secondary maximum contaminant level (SMCL) for zinc(II) of 5 mg/L [2]. EPA had set Land Disposal Restrictions; Universal Treatment Standards for zinc(II), i.e. wastewater standard—2.61 mg/L and non-wastewater standard—4.3 mg/L total characteristic leachate procedure (TCLP) [3]. Similarly, zinc(II) concentration level below 1.0 mg/L in both the specific water area and the water area has been instituted as the Korean Water Quality Standard for rivers, streams, lakes, and lagoons [4]. Therefore, the development and application of suitable and effective wastewater treatment technologies is of utmost importance either to prevent heavy metal pollution or to reduce it to very low level so as to meet the stringent environmental regulations.

Today membrane-based separation processes had become attractive and effective technique in the treatment of industrial wastewater containing toxic inorganic as well as organic pollutants. As surfactant-based pressure-driven membrane separation process, micellar enhanced ultrafiltration (MEUF) had shown to be an efficient technique for the removal of heavy metals laden wastewater as it combines both the high selectivity of reverse osmosis (RO) and high flux of ultrafiltration (UF) [5]. The advantages of this method are low energy requirements and high removal efficiency owing to the effective trapping or solubilization of desired pollutant solutes by the surfactant micelles [6]. Recent studies had reported that almost all metal ions can be separated via MEUF method, including Cd^{2+} [6,7], Co^{2+} [8], Cs^{+}, Sr^{2+}, Cr^{3+}, Mn^{2+} [9].

Although MEUF has been widely applied, to our best knowledge, most of the previous studies undertaken regarding heavy metals removal from aqueous solution via MEUF process were primarily based on use of surfactants higher than its critical micelle concentration (CMC) level, thereby resulting high concentration of surfactants in the retentate and also in permeate. As the economic viability of MEUF process greatly relies on the ability to recover the large portion of surfactants from the retentate, as a result aforementioned trend will ultimately enhance the operating costs. On the other hand, unbound surfactant monomers present in the permeate may result secondary pollution as well. Consequently, this has spurred the application of MEUF process using relatively low surfactant concentration. When the surfactant concentration is low, the efficient solute rejection is not expected in principle, but the concentration polarization (CP) effect can have beneficial effect in achieving desired metal rejection at low surfactant concentration [10]. Further, relatively few studies had reported application of MEUF coupled with activated carbon fibers (ACF) hybrid process regarding the removal of some heavy metals from aqueous solution [11–14]. As far as our concern, no work had been conducted using high molecular weight cut-off (MWCO) membrane, i.e. 300,000 Da (or) 300 kDa and relatively low anionic surfactant concentration throughout the entire MEUF process and MEUF coupled with ACF integrated processes regarding removal of zinc(II) ions and surfactant sodium dodecyl sulfate (SDS) from synthetic wastewater. Compared to powdered activated carbon (PAC) and granular activated carbon (GAC), ACF showed better adsorption capacity due to uniform micro-pore structure, faster adsorption kinetics, and lower pressure drop [15]. Adsorption of unbound surfactant monomers and metal ions from permeate generated from MEUF process using ACF can be viable promising technique to enhance metal as well as surfactant removal due to aforementioned peculiar features of ACF.

2. Materials and methods

The primary aim of this present study was to evaluate the performance of MEUF process in zinc(II) removal from synthetic wastewater using SDS at relatively low concentration and high MWCO membrane, and also to figure out the optimum operational conditions for MEUF-ACF hybrid process. The process was conducted in a cross-flow UF mode using polyacrylonitrile (PAN) hollow fiber membrane. In this study, the effects of various important parameters viz initial permeate flux, retentate pressure, solution pH, (initial SDS concentration) molar ratio of zinc(II) to SDS, molar ratio of SDS to zinc(II) (initial zinc concentration), and different MWCOs membrane on metal and SDS removal percentage, absolute permeate flux, relative or normalized flux, specific flux or filtration coefficient (or permeability of membrane), transmembrane pressure (TMP), recovery %, volume concentration ratio and system conversion, hydraulic membrane resistance, and secondary resistance of membrane were investigated. Analysis of aforementioned performance parameters can be beneficial to acquire the practical application of this integrated technique.

2.1. Chemicals

In this study, zinc(II) sulfate heptahydrate ($ZnSO_4{\cdot}7H_2O$) (molecular weight 287.56), 99% assay was obtained from Daejung Chemicals & Metals Co. Ltd, Korea. Similarly, anionic surfactant i.e. SDS

Table 1
Properties of surfactant SDS

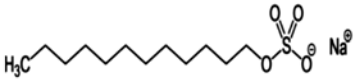
Chemical name	Type	CMC (mM)	Molecular weight	Molecular structure	Density (g/cm)	Assay (%)
Sodium dodecyl sulphate	Anionic	8.3	288.38		1.01	99.0

Table 2
Characteristics of UF membrane

Membrane material	Polyacrylonitrile (PAN)
Membrane type	Hollow fiber
Flow direction	Inside to outside
Flow type	Cross flow (or tangential flow)
Effective membrane area, m^2	4.8
Module dimension (D × L), mm × mm	89 × 1,126
Membrane diameter (inside/outside), mm	0.8/1.4
Molecular weight cut-off (MWCO), Dalton, Da	300,000; 100,000; 30,000
Maximum operating temperature,℃	40
pH range	1–13
Housing material	Polyvinylchloride (PVC)
Potting material	High performance epoxy resin

(molecular weight 288.38) of 99% assay was procured from Acros Organics, Ltd, USA. The surfactant was used without further treatment. The characteristics of SDS are given in Table 1. The distilled water was used throughout entire experiment. Hydrochloric acid (conc. HCl) (molecular weight 36.46) of above 35.0% assay, and sodium hydroxide (NaOH) (molecular weight of 40.0) of above 97.0% assay were obtained from Daejung Chemicals & Metals Co. Ltd, Korea.

2.2. Membrane

Hollow fiber UF membrane of various MWCOs, i.e. 300,000 Da (300 kDa), 100,000 Da (100 kDa), 30,000 Da (30 kDa) were obtained from Chemicore, Ltd, Korea, and used without further treatment for entire experiment. The characteristics of membrane are listed in Table 2.

2.3. Activated carbon fiber

ACF was supplied by ACF, Korea. It has BET surface area of 1,000 m^2/g. Likewise, the weight of ACF is 30 g and that of the cartridge is 40 g. Schematic diagram of ACF used in this study is shown in Fig. 1. Similarly, the characteristics of the ACF are given in Table 3.

2.4. Experimental instrumentation and procedure

A laboratory-scale membrane system was used in all UF experiments carried out in this research study. All the solutions were prepared by mixing stoichiome-

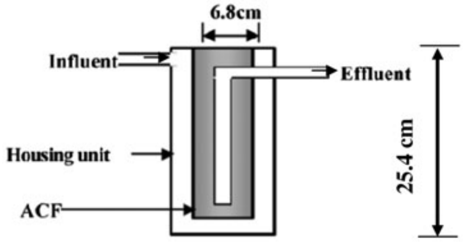


Fig. 1. Detail apparatus of activated ACF filter (Model FMC—250 A; Mfg: Korea, ACF).

Table 3
Characteristics of ACF

No. of fibers	103
ACF BET surface area, m^2/g	1,000
Weight of ACF, g/cartridge	30
Bulk density, kg/m^3	0.2
Iodine number, mg/g	1,500

tric amounts of SDS and zinc(II) sulfate heptahydrate in six liters of distilled water and kept on mixing for 3–4 h before the feed solution was fed into the UF membrane. The mixing speed was maintained such that there is proper mixing of solution and no vortex was formed.

The MEUF experimental unit consists of a feed tank, UF membrane, backwash tank, and permeate tank as shown in Fig. 2. The membrane filtration mode was cross-flow (tangential flow) filtration in which the feed solution passes parallel to the membrane. Similarly, the mode of UF membrane operation is of batch type in which retentate was re-circulated to the feed tank, while permeate was collected in the separate tank as shown in Fig. 2. At the beginning of an experiment, an initial flow rate was measured using distilled water. Permeate flux was also monitored at a regular time interval during the MEUF operation. Initial retentate pressure was also maintained using retentate pressure gage mounted on the UF unit. After fixing the initial flow rate, feed solution was passed to the UF membrane for an hour. Samples were collected at specified time interval, i.e. 10, 20, 30, 40, 50, and 60 min. Flux was also measured at the aforementioned time. After each experiment, the membrane was cleaned successively with distilled water, 0.05 N NaOH, distilled water, 0.25% HCl (conc.), and finally in distilled water. The details of the experimental conditions are summarized in Table 4.

MEUF-ACF experiments were carried out for 4 h. In this case, cartridge filter (CF) followed by two ACF filters unit were added in series after the MEUF unit (Fig. 2). Experimental module consists: influent tank, a CF followed by two sets of ACFs and effluent tank. The main function of CFin the system was to enhance the life span of ACFs. The ACFs used were 25.4 cm (10 inch) of length, 6.8 and 2.8 cm of outside and inside diameters, respectively, and 2 cm of thickness. Total mass of ACF used per filter was 120 mg (ACF, Korea) (Table 3).

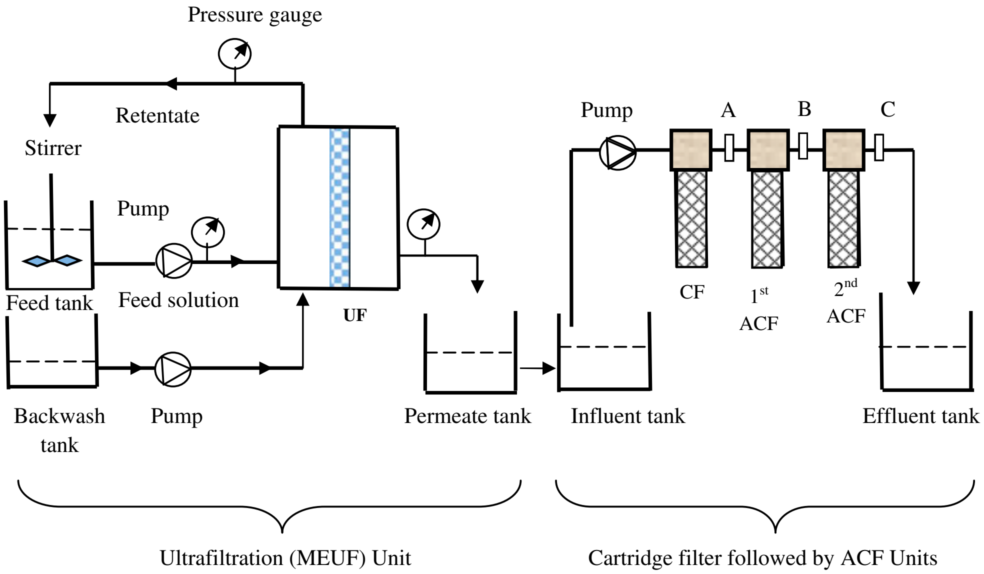


Fig. 2. Schematic representation MEUF-ACF hybrid process for zinc(II) removal from synthetic wastewater.

Table 4
Details of MEUF experimental conditions

Retentate pressure, MPa	0.14, 0.20, 0.22
Initial permeate flow, mL/min	30, 40, 50, 60, 64, 80
Initial permeate flux, L/m^{2}h	32.73, 43.64, 54.55, 65.45, 69.82, 87.27
Sampling time, min	10, 20, 30, 40, 50, 60
pH	2.2, 4.02, 7.0, 8.2, 11.7
Molar ratio of zinc(II) to SDS	1:5, 1:8, 1:10, 1:120, 1:200
Initial SDS concentration, mM	0.348, 0.557, 0.696, 8.352, 13.92
Molar ratio of SDS to zinc(II)	1:2, 1:4, 1:8, 1:10
Initial zinc(II) concentration, mM	0.42, 0.84, 1.68, 2.10

In this experiment, MEUF permeate was collected and passed to the ACF unit. Samples were collected every half an hour time interval from sampling point A, B, and C. Flow in ACF unit was maintained 40 mL/min. After each experiment, the MEUF unit was cleaned as mentioned in the previous experimental part, while ACF unit was cleaned by soaking it in distilled water and then subsequently soaking in 0.1 M NaOH solution, distilled water, 2% HCl solution, and distilled water for 12 h each.

2.5. Measurement and analysis

Zinc(II) concentrations were measured using Inductively Coupled Plasma (Varian-720, ICP-OES) at a wavelength of 213.857 nm. Samples were pre-treated according to the Standard Methods for the Examination of Water and Wastewater. SDS concentration was measured using chemical oxygen demand (COD) according to the Standard Methods. There was a significant close correlation between COD and SDS concentration (R^2 = 0.998). Zinc (II) and SDS removal rate were calculated using following Eq. (1):

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

where R = removal rate; C_p = permeate concentration (mg/L); C_f = feed concentration (mg/L). The TMP was also calculated using following Eq. (2) [13]:

$$\text{TMP} = (P_F - P_R)/2 - P_P \tag{2}$$

where TMP is transmembrane pressure (MPa); P_F, P_R, and P_P are the feed pressure(MPa), retentate pressure (MPa), and permeate pressure (MPa), respectively. The absolute permeate flux (J_v) was calculated by following Eq. (3) [16]:

$$J_v = \frac{V}{t \times A} \tag{3}$$

where J_v is the absolute permeate flux ($\text{L/m}^2\,\text{h}$); V is the volume of the permeate sample collected (L), t is the time needed for collecting the permeate sample (h); A is the membrane effective area (m^2). In addition, normalized flux or relative flux was also calculated as ratio of absolute permeate flux (J_v) to pure water flux (J_w), which is expressed as depicted in Eq. (4) [17]:

$$\text{Normalized flux} = \frac{J_v}{J_w} \tag{4}$$

Similarly, specific flux, J_{sp} (or permeability of membrane) also known as filtration coefficient, L_p or hydraulic conductivity of membrane is defined as ratio of absolute permeate flux, J_v to TMP, which is expressed as shown in Eq. (5) [3]:

$$J_{sp} = \frac{J_v}{\text{TMP}} \tag{5}$$

The ultimate aim of UF is to maximize the recovery of solutes of interest. Recovery of an UF system is defined as the percentage of the feed water that is converted into the permeate. It was calculated using following Eq. (6) [18]:

$$R\,(\%) = \frac{P}{F} \times 100 \tag{6}$$

where R is the recovery %; P is the permeate volume; and F is the feed volume.

3. Results and discussion

3.1. Effect of initial permeate flux

Series of experiments were conducted to investigate the effect of initial permeate flux. The zinc(II) removal rates were observed to vary with time as a function of initial permeate flux, but did not show much of distinct trend (Fig. 3a). Thus, average removal rate values were plotted against varying initial permeate fluxes in Fig. 3b. It was found that the average zinc(II) removal rate was 74.84% at initial permeate flux of 43.64 $\text{L/m}^2\,\text{h}$, while it was 69.09 and

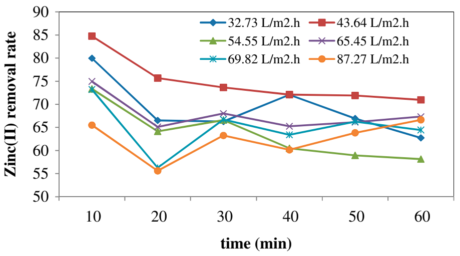


Fig. 3a. Effect of initial permeate flux on zinc(II) removal rate with time.
Conditions: initial zinc concentration = 20 mg/L; molar ratio of zinc to SDS = 1:2; initial retentate pressure = 0.14 MPa; MWCO of the membrane = 300,000 Da.

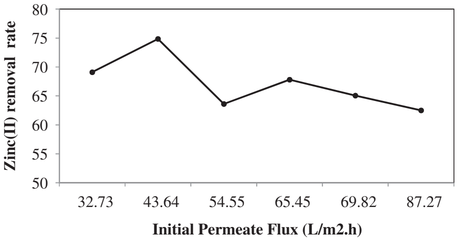


Fig. 3b. Average zinc(II) removal rate with initial permeate flux.

62.51% for initial operating permeate fluxes of 32.73 and 87.27 L/m^2 h, respectively (Fig. 3b). It implies that the zinc(II) removal rate decreased with the initial operating permeate flux. This is because SDS concentration on membrane surface gets increased with the reduction of initial operating permeate flux, enhancing higher zinc(II) removal from the feed solution. In surfactant-based membrane separation process, flux decline is the major bottleneck of the process. It is caused by the CP, fouling and adsorption. It has been reported that at the higher flux, CP and secondary gel layer formation get accelerated. The decline in the removal rate with the increase in initial permeate flux might be due to secondary resistance exerted to the passage of metal–micelle complexes by the CP/gel layer (in extreme case) [19].

Moreover, the mean pore diameter of the 300 kDa (300,000 Da) is larger, i.e. ranges from 8.6 to 24.5 nm [20]. Due to larger pore diameter, it has more surfactant permeation and less sub-micelles (smaller in size than micelle) retention [21]. With the increasing initial operating permeate flux, the surfactant and sub-micelle/hemi-micelle permeation gets more enhanced, thereby resulting less zinc(II) removal rate from the feed solution. Considering higher zinc(II) removal rate, the initial permeate flux of 43.64 L/m^2 h was found to be the optimum permeate flux in the experimental condition.

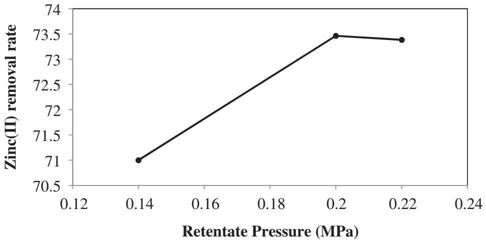


Fig. 4a. Average zinc(II) removal rate vs. retentate pressure.
Conditions: initial permeate flux = 43.64 L/m^2 h; molar ratio of zinc to SDS = 1:2; initial zinc concentration = 20 mg/L; MWCO of the membrane = 300,000 Da (300 kDa); pH 7.

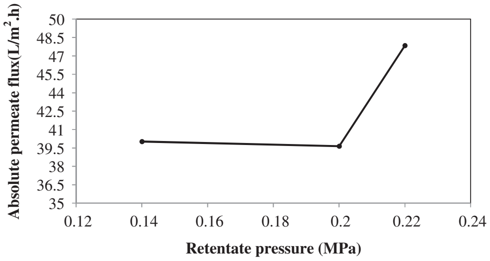


Fig. 4b. Variation of absolute permeate flux with retentate pressure.

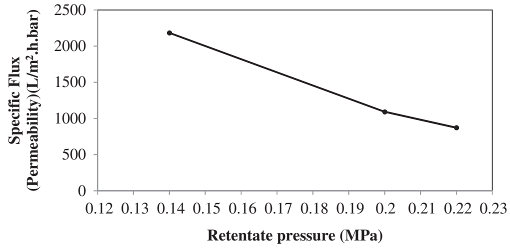


Fig. 4c. Variation of specific flux (or permeability) with retentate pressure.

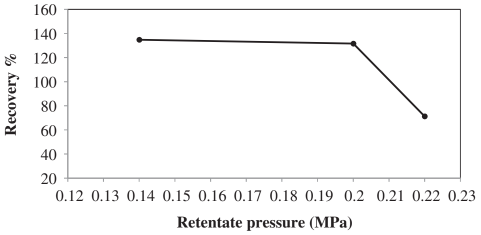


Fig. 4d. Variation of recovery % with the retentate pressure.

3.2. *Effect of retentate pressure*

Fig. 4a revealed average zinc(II) removal rate with the varying operating retentate pressure. Average zinc (II) removal rate was 71.0% at the retentate pressure of 0.14 MPa (1.4 bar), whereas it was 73.46 and 73.38% for retentate pressure of 0.20 and 0.22 MPa, respectively. Similar results were presented on chromate and copper removal in a lower MWCO membrane (10 kDa) in the previous studies [12,14]. In addition, as anticipated with the increase in retentate pressure, TMP also increased. It was found that for the retentate pressure of 0.14, 0.20 and 0.22 MPa, the TMP were found to be 0.02, 0.04, and 0.05 MPa, respectively. The operation of membrane processes at low TMP is an important issue in terms of minimizing the operating costs. Actually increase of pressure increases the gel layer thickness that in turn increases the rejection of metal–micelle complex. At higher pressures, resistance of micelle aggregation layer (MAL) increases due to the compaction that in turn increases the retention of surfactant micelles, thereby enhancing more metal rejection [22]. Thus, it was found that zinc(II) removal rate was enhanced with the increase in the retentate pressure. Similar result was also presented by previous authors [8].

As shown in Fig. 4b, absolute permeate flux depicted the fluctuating trend with the increase of operating retentate pressure. Absolute permeate flux (J_v) was 40.018 L/m^{2}h for 0.14 MPa retentate pressure, while it was 39.64 and 47.84 L/m^{2}h for 0.20 and 0.22 MPa retentate pressure, respectively. As shown in Fig. 4c, the specific flux, J_{sp} decreased with the increase of the operating initial retentate pressure. The J_{sp} were 218.20, 109.10, 87.28 L/m^{2}h bar, for the retentate pressure of 1.4, 2.0, 2.2 bar, respectively. This can be ascribed as increased CP at a higher initial applied retentate pressure caused a faster reduction in the permeate flux. Thus, a lower operating retentate pressure should be chosen to get a higher specific flux [14].

The ultimate aim of UF process is to maximize recovery of solutes of interest. It is said that for economic reasons, the recovery rate should always be high as possible [23]. However, retentate pressure and TMP both have negative impact on recovery % (Fig. 4d). Recovery % were found to be 134.77% (recovery rate = 1.35), 131.60% (recovery rate = 1.32), 71.25% (recovery rate = 0.71) for the retentate pressure of 0.14, 0.20, 0.22 MPa, respectively. Considering all above results, the retentate pressure of 0.20 MPa (2.0 bar) was the optimized retentate pressure in experimental condition of the present study.

3.3. Effect of solution pH

In order to investigate the effect of solution pH on zinc(II) removal rate, the pH of the feed solution was varied from 2.2 to 11.7, and the constant concentrations of zinc(II) and SDS were maintained at 0.0696 and 0.1392 mM, respectively. Fig. 5a depicted the average zinc(II) removal rate with pH of feed solution, while Fig. 5b depicted the zinc(II) removal rate with operational time as a function of solution pH. Zinc(II) removal rate was higher at the beginning and showed a marginal reduction in removal rate with the span of time as shown in Fig. 5b. Similarly, it was observed that the zinc(II) removal rate was 45.86% for feed

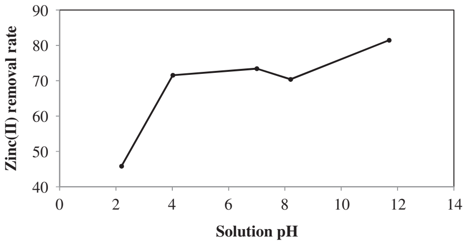


Fig. 5a. Effect of solution pH on zinc(II) removal rate. Conditions: initial permeate flow = 40 mL/min (43.64 L/m^{2}h); retentate pressure = 0.20 MPa (2.0 bar); MWCO of membrane = 300,000 Da (300 kDa); constant SDS concentration = 40.54 mg/L (0.1392 mM); constant initial zinc(II) feed concentration = 20.20 mg/L (0.0696 mM).

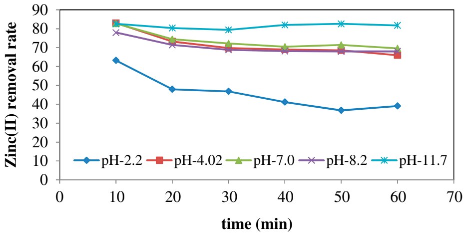


Fig. 5b. Effect of solution pH on zinc(II) removal rate with operational time.

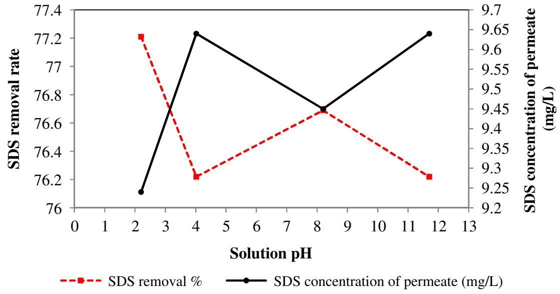


Fig. 5c. Effect of solution pH on permeate SDS concentration and SDS removal rate.

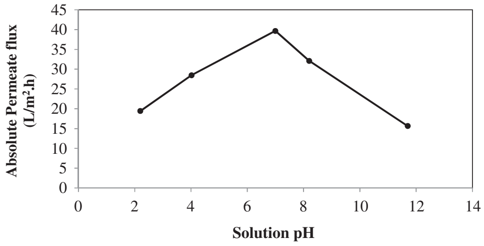


Fig. 5d. Effect of solution pH on absolute permeate flux, *Jv*.

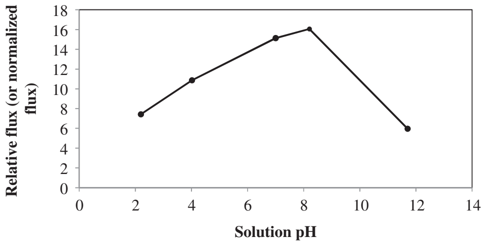


Fig. 5e. Effect of solution pH on relative flux.

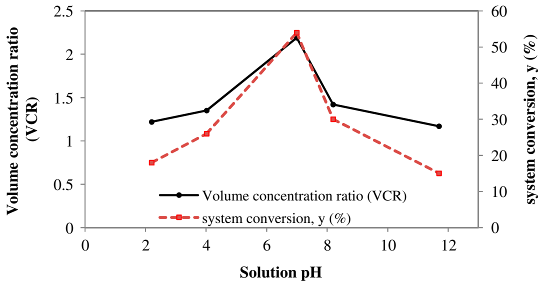


Fig. 5f. Effect of solution pH on volume concentration ratio and system conversion.

solution pH of 2.2, and 73.46% for solution pH of 7.0, while it was 81.47% for the pH value of 11.7. The least zinc(II) removal rate was obtained at highly acidic condition (pH 2.2). This is because at low pH, the concentration of H^+ ions becomes greater than the Zn^{2+} ions concentration, therefore Zn^{2+} ions now have to compete with Na^+ as well as H^+ ions for their binding with SDS micelles. The H^+ ions possessing the smallest ionic radius in comparison to Na^+ and Zn^{2+} ions, more active H^+ binds to SDS micelles more selectively and this is reflected in the corresponding decrease in zinc(II) removal rate in this study [8].

Similarly, under the highly basic condition (pH 11.7), zinc(II) removal rate was the highest. This can be corroborated as under this condition, Zn^{2+} ions does not have to compete with highly active H^+ ions for the binding sites with SDS micelles. Thus, it can be deduced that the effect of pH relies upon the type of metal used in the solution, whether H^+ ions compete with metal or not during the electrostatic adsorption on surfactant micelle as the recovery of metal ions via MEUF process is primarily due to electrostatic attraction between the metal ions and oppositely charged micellar surface.

Fig. 5c demonstrated the variation of permeate SDS concentration and SDS removal rate as a function of solution pH. It was found that highest SDS removal rate (77.21%) was obtained at highly acidic condition (pH 2.2) and the least removal rate (76.22%) was obtained at slightly acidic and highly basic condition (pH 4.02 and 11.7). But the SDS removal rate was 76.69% in case of slightly basic condition (pH 8.2). Above all, the differences in SDS removal rate within the range of various pH values thus studied were found to be marginal. This may be due to use of constant influent surfactant SDS concentration below its CMC level. As the pH decreases to 2.2, the charge density of SDS molecules becomes less negative [24], causing the SDS molecules to repel one another less, i.e. decline in repulsion among SDS molecules. This favors their passage through the membrane in lower pH value [25].

Regarding surfactant retention, in all cases, the lowest retention i.e. least permeate SDS concentration (highest removal rate) was observed at pH value of 2.2 (as shown in Fig. 5c). Highly acidic condition (pH 2.2) gives the membrane a less negative zeta potential. This leads to a greater repulsion of free zinc(II) cations and as a result, the zinc(II) cations are forced into the bulk solution. This explains why the zinc(II) removal rate was least at highly acidic condition (pH 2.2) in comparison to other pH values [25].

It was observed that absolute permeate flux, J_v increased rapidly with the increment in solution pH from 2.2 to 7.0, while J_v demonstrated rapid reduction with further increment in pH value as shown in Fig. 5d. On the other hand, it was figured out that the relative or normalized flux also followed similar trend as that of J_v (Fig. 5e), but the value of relative flux was highest in basic condition, i.e. 16.05 (pH 8.2), followed by relative flux value in neutral condition, i.e. 15.13 (pH 7.0). In this study, the pure water flux was found to be 2.2 L/m^2 h. Thus, phenomenon of CP was dominant in this MEUF process because the J_v is substantially higher than that of pure water flux, J_w [9]. This can be ascribed by the reason that the shape and aggregation number of surfactant micelles changes with the increase in feed solution pH and also pore blocking can be more intense in the higher feed solution pH [26].

Volume concentration factor, C_v (VCF or X), also known as volume concentration ratio (VCR) is the amount that the feed stream has been reduced in volume from the initial volume [23]. It was figured out that volume concentration factor (C_v) and also system conversion, y(%) rapidly increased with the increase in feed solution pH (2.2–7.0), while the C_v and y(%) both reduced rapidly with the further rise in feed solution pH (7.0–11.7) as shown in Fig. 5f.

From this result, it could be concluded that the system conversion, y(%), and volume concentration factor, (C_v), would generate low values when the feed solution pH is highly acidic or basic in UF process. Considering aforementioned results, it could be stated that at neutral condition of feed solution (pH 7.0), a higher zinc(II) removal rate and also higher permeate flux can be achieved.

3.4. Effect of molar ratio of zinc(II) to SDS

Series of experiments were conducted to investigate the effect of molar ratio of zinc (II) to SDS, i.e. initial SDS concentration on zinc(II) removal rate. Fig. 6a showed the variation of zinc(II) removal rate with the initial SDS concentrations ranging from 0.1392 to 13.92 mM (molar ratio of zinc to SDS ranging from 1:2 to 1:200), at a constant initial zinc(II) feed concentration of 0.0696 mM (20.20 mg/L). Fig. 6b showed the variation of zinc(II) removal rate with time as a function of initial SDS concentration. As reported, the CMC of SDS is 8.3 mM from conductivity measurement [9].

As depicted in Fig. 6a, the marginal increase in removal rate was observed, i.e. from 75.93 to 77.29% when the initial SDS concentration increased from 0.1392 to 0.557 mM (molar ratio of zinc to SDS of 1:2–1:8). Although these SDS concentrations are far below its CMC, zinc(II) ions were still removed up to 77.29%. Further, increase in the SDS concentration from 0.696 to 13.92 mM (above CMC) (molar ratio of zinc to SDS from 1:10 to 1:200) resulted in reduction of removal rate, i.e. 76.33–67.93%.

There may be two main reasons for metal rejection at very low SDS concentration—Firstly, the presence of bivalent zinc(II) ions enhances the surface activity of DS$^-$, thereby resulting decline in surfactant CMC [27] and secondly, the surfactant concentration in the layer adjacent to the membrane surface was higher than that in the bulk solution due to CP [28]. Thus, it can be deduced that CP is an important characteristic of all UF systems and some level of CP is a valuable practical aspect of the MEUF process, in terms of the low surfactant concentration required to achieve high solute rejections [10].

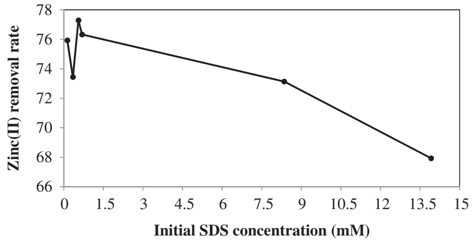


Fig. 6a. Effect of initial SDS concentration on zinc(II) removal rate.
Conditions: initial permeate flow = 40 mL/min (43.64 L/m^2 h); retentate pressure = 0.20 MPa (2.0 bar); pH = neutral (7.0); MWCO of membrane = 300,000 Da (300 kDa); constant initial zinc(II) concentration = 20.20 mg/L (0.0696 mM).

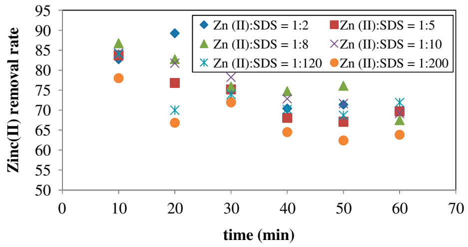


Fig. 6b. Zinc(II) removal rate with time as a function of molar ratio of zinc(II) to SDS (or initial SDS concentration).

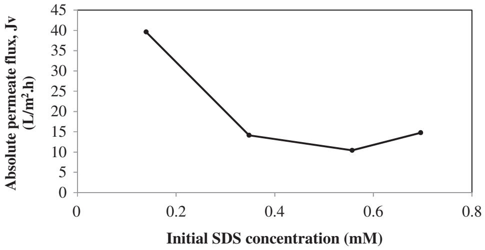


Fig. 6d. Variation of absolute permeate flux with initial SDS concentration or molar ratio of zinc(II) to SDS.

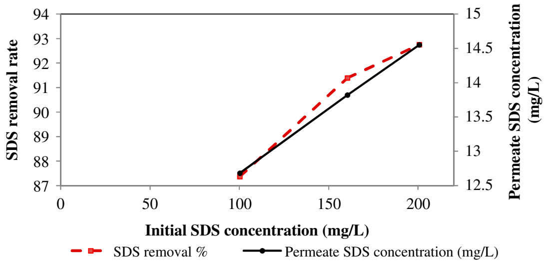


Fig. 6c. Variation of permeate SDS concentration and SDS removal rate with initial SDS concentration (molar ratio of zinc to SDS).

However, the marginal decrease in removal rate was obtained at high SDS concentration and above CMC. This may be attributed to the fact that the increase of feed SDS concentration has two opposite effects [29]. The advantageous effect is the formation of more micelles and then more zinc(II) ions get entrapped. On the other hand, there is a competition between Na$^+$ and Zn^{2+} ions to bind with the micelles, and the competition depends on both the electrical charge of the ion and their concentration. The bivalent Zn^{2+} ions get preferentially bound with the micelle originally. However, the high concentration of Na$^+$

inverted the tendency. At the low SDS concentration (below CMC), the first effect is predominant and the retention of Zn^{2+} ions increases; however, at the high SDS concentration and above CMC, the competition between two counter-ions is more advantageous to Na^{+}, thereby causing the marginal decrease in zinc removal rate [19].

The permeate SDS concentration should be considered to evaluate the performance of the MEUF process because the surfactant in permeate may induce the secondary pollution [10]. The variation in permeate SDS concentration and SDS removal rate with various SDS concentrations 0.348–0.696 mM, i.e. 1:5–1:10, in the feed solution is shown in Fig. 6c. It was figured out that permeate SDS concentration increased with increasing molar ratio of zinc to SDS (below CMC) (or increasing initial SDS concentration), i.e. it was 12.68, 13.82, 14.55 mg/L for initial SDS concentrations of 0.348 mM (1:5), 0.5568 mM (1:8), and 0.696 mM (1:10), respectively. Similarly, SDS removal rate also followed the similar trend, i.e. it was 87.37, 91.39, 92.75% for initial SDS concentration of 0.348, 0.5569, and 0.696 mM, respectively. As the surfactant concentration is much below its CMC level so the rejection can be attributed to the CP and adsorption of surfactant monomers at the membrane surface.

To observe the effect of initial SDS concentration on absolute permeate flux, J_v, another series of experiments were conducted at various molar ratios of zinc (II) to SDS as shown in Fig. 6d. It was observed that J_v declined with the increase in molar ratio from 1:2 to 1:8, but on further increment to 1:10 (0.696 mM), the average J_v showed increment. Absolute permeate flux was found to be 39.64, 14.18, 10.45, 14.78 L/m^2 h for initial SDS concentrations of 0.1392 mM (1:2), 0.348 mM (1:5), 0.5568 mM (1:8), 0.696 mM (1:10), respectively.

It was observed that J_v decreased with the initial SDS concentration within the concentration thus studied. This can be ascribed as with the increase in initial SDS concentration, solution viscosity, osmotic pressure, including pore blocking resistance (CP and secondary membrane resistance) also get enhanced. Although no micelles are supposed to be in the initial feed solution at SDS concentrations below its CMC level, a larger fraction of surfactants is present in micellar form in the vicinity of membrane surface. The micelles accumulate on the membrane surface continuously and some small micelles block the membrane pores. Further increase in initial SDS concentration may also result in higher secondary membrane resistance due to pronounced CP effect [10].

Even though the permeate flux decreased with the increase in initial SDS concentration due to feed solution viscosity and secondary membrane resistance caused by CP, the absolute permeate flux of 19.76 L/m^2 h in average was obtained within the range of different initial SDS concentrations thus studied. Thus, it indicates good potential application of MEUF process using PAN-based hollow fiber UF membrane to remove the metal ions from aqueous solution at the surfactant concentration below its CMC level [10]. Thus, the molar ratio of zinc(II) to SDS, i.e. 1:2 (initial SDS concentration of 0.1392 mM) was found to be the most appropriate molar ratio based on the higher zinc (II) removal rate and permeate flux under the experimental conditions.

3.5. Effect of initial zinc(II) concentration

To investigate the effect of initial zinc(II) concentration on zinc(II) removal rate, the concentrations of zinc(II) were varied from 0.42 to 2.10 mM, keeping the concentration of SDS constant, i.e. 0.21 mM (Figs. 7a and 7b). The zinc removal rate values were observed to vary with the time as a function of initial zinc(II) concentration, but did not show much of a trend (Fig. 7b). Therefore, average removal rate values were reported against varying initial zinc(II) concentrations in Fig. 7a. It was observed that the zinc(II) removal rate declined with the increasing initial zinc(II) concentration, although the reduction was marginal, i.e. zinc(II) removal rate decreased from 84.67 to 82.42% when the initial zinc(II) concentration increases from 0.42 to 2.10 mM. Thus, it can be concluded that smaller the zinc(II) feed concentration is, the higher removal rate of zinc(II) could be achieved.

On increasing the zinc(II) feed concentration, the zeta potential of the SDS micelles increases as the surface charge density decreases. Moreover, as the feed concentration increases, there is corresponding increase in the concentration of unbound Zn^{2+} ions, and hence, the permeate concentration also increases [8]. Therefore, the reduction in removal rate at higher

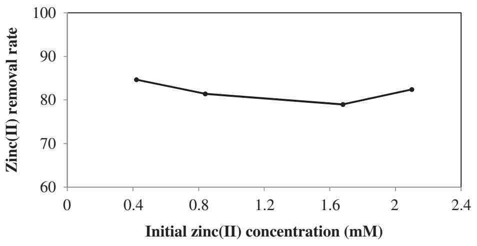


Fig. 7a. Effect of initial zinc(II) concentration on zinc(II) removal rate.
Conditions: initial permeate flow = 40 mL/min (43.64 L/m^2 h); retentate pressure = 0.20 MPa; pH = neutral (7.0); MWCO of membrane = 300,000 Da (300 kDa); constant SDS concentration = 60.56 mg/L (0.21 mM).

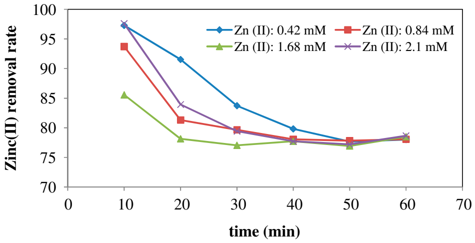


Fig. 7b. Zinc(II) removal rate with time as a function of initial zinc(II) concentration.

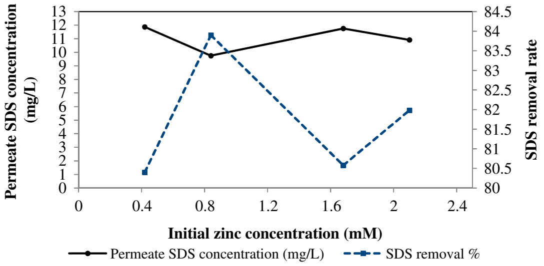


Fig. 7c. Variation of permeate SDS concentration and SDS removal rate with initial zinc(II) concentration (mM).

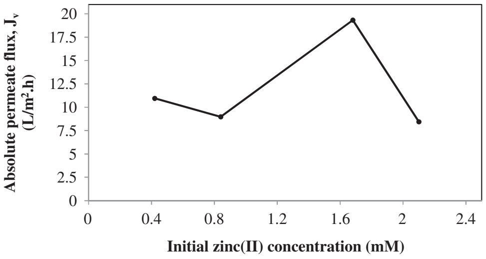


Fig. 7d. Effect of initial zinc(II) concentration on absolute permeate flux, *Jv*.

feed zinc(II) concentration might be due to the lack of available binding sites, which in turn can be explained by the increase in zeta potential [7].

Variation of permeate SDS concentration and SDS removal rate with initial zinc(II) concentration was also investigated (Fig. 7c). It was found that overall trend of SDS removal rate demonstrated marginal increment with the increase of initial zinc(II) concentration. However, SDS removal rate was highest, i.e. 83.90%, when the initial zinc concentration was 0.84 mM at constant SDS concentration 0.21 mM (below CMC). It can be attributed as the quantity of SDS micelle gets enhanced with increasing Zn^{2+} ions concentration in the feed solution. This is because in the presence of metal ions, positively charged metal ions bridge more than one negatively charged surfactant micelles leading to a formation of gel layer on the membrane surface at lower concentration of surfactants. Further, increment in cation concentration enhances the repulsive forces between the head groups, and the formation of micelles becomes easier, thereby resulting more zinc(II) removal and less permeate surfactant monomer concentration [30].

In addition, effect of initial zinc(II) concentration on absolute permeate flux, J_v was also investigated as depicted in Fig. 7d. It was found that J_v showed the fluctuating trend with the increasing initial feed zinc(II) concentration. For initial zinc(II) concentrations of 0.42, 0.84, 1.68, and 2.1 mM, the corresponding average J_v were found to be 10.95, 8.98, 19.33, and 8.45 L/m^2 h, respectively. However, the highest flux was obtained at initial zinc(II) concentration of 1.68 mM, i.e. 19.33 L/m^2h. It can be attributed as with the increment in initial zinc(II) concentration, thickness of SDS micelles layer, i.e. MAL, deposited on the membrane surface get reduced. This in turn results in reduction of resistance offered by the MAL of surfactant to permeate flow thereby causing higher permeate flux [30]. It was observed that within the whole range of different initial zinc(II) feed concentrations thus studied, the overall trend showed reduction in average J_v with the increase of initial zinc(II) concentration. Previous authors [31] had reported a significant decline in permeate flux when increasing the concentration of divalent metal counter ions at fixed feed surfactant concentration. They further reported that the increase in heavy metals cations causes the repulsive forces between the micelles head groups, and the formation of micelle becomes easier, i.e. the CMC decreases [30]. When decreasing the CMC of the surfactant, more surfactant monomers will be in the micellar form and therefore, surfactant concentration in the retentate will be increased enhancing the flux decline [6].

3.6. *Effect of MWCO of UF membrane (or NMWL of UF membrane)*

Another series of experiments were conducted to investigate the effect of MWCO or nominal molecular weight limit (NMWL) of UF membranes on zinc(II) removal rate and filtration coefficient, L_p or hydraulic conductivity of membrane at constant initial SDS concentration and initial zinc(II) concentration of 0.21 mM (60.56 mg/L) and 0.42 mM (120.78 mg/L), respectively.

As anticipated, it was observed that zinc(II) removal rate was higher for low MWCO UF membrane and vice versa as shown in Fig. 8a. During 2.5 h experimental operation, it was found that average zinc (II) removal rate was 79.06, 81.40, and 91.47% for 300, 100, and 30 kDa MWCO UF membrane, respectively.

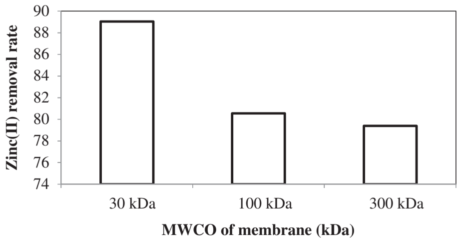


Fig. 8a. Comparative zinc(II) removal rate from MEUF unit with various MWCO UF membrane.
Conditions: molar ratio of SDS to zinc(II) = 1:2 (0.21:0.42 mM); initial retentate pressure = 0.20 MPa; initial permeate flux = 43.64 L/m^2 h; pH = neutral (7.0); operational time = 2.5 h; MWCO of membrane = 30 kDa, 100 kDa, 300 kDa.

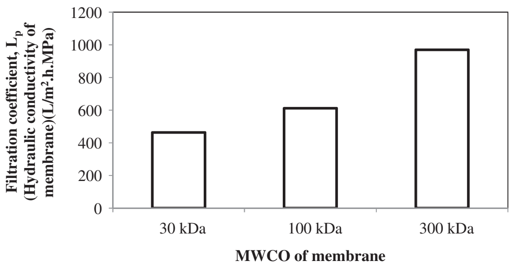


Fig. 8b. Effect of MWCO of membrane on filtration coefficient, *Lp*.

This can be attributed as influence of MWCO of membrane would be more pronounced at low surfactant concentration (as in this study) unlike in the condition of higher surfactant concentration in MEUF process [12]. Thus, it could be inferred that low MWCO membrane was more efficient in zinc(II)–SDS micelle complexes rejection. Previous author had presented that arsenate removal was slightly higher in case of 3 kDa MWCO compared to 10 kDa, especially at the lower concentration of surfactant [32]. Similar results had been also reported in previous research papers [11,12]. Nevertheless, filtration coefficient, L_p, was found to be higher for high MWCO UF membrane and vice versa as shown in Fig. 8b. It was observed that the values of L_p were 969.78, 612.36, and 463.89 L/m^2 h MPa for 300, 100, and 30 kDa MWCO UF membrane, respectively.

3.7. Zinc(II) removal from various units of MEUF-ACF hybrid process

Series of experiments were conducted to investigate the removal of excess zinc(II) ions present in the MEUF permeate by coupling with ACF as shown in Fig. 2. Further, the comparative analysis was carried out regarding the average zinc(II) removal rate from various units of MEUF-ACF hybrid process viz, MEUF unit, CF unit, two ACF units (1st ACF and 2nd ACF), at constant initial SDS concentration and initial zinc(II) concentration of 0.21 mM (60.56 mg/L) and 0.42 mM (120.78 mg/L), respectively (Figs. 9a and 9b).

As shown in Table 5, the 30 kDa UF membrane showed average zinc(II) removal rate of 90.76% for initial zinc(II) concentration of 0.42 mM during 2.0 h operation time. Average zinc(II) removal rate reached 99.41% when coupled with the ACF units. The 100 kDa UF membrane showed average zinc(II) removal rate of 81.75%, and the zinc(II) removal rate reached 94.45% after combining with the ACF units. Similarly, the 300 kDa UF membrane showed 79.37% zinc(II) removal rate, while the average zinc(II) removal rate reached 90.21% after coupling with the ACF units. In conclusion, it could be stated that average zinc(II) removal rate of MEUF-ACF hybrid process was 8.65, 12.70, 10.84% higher than MEUF alone in 30, 100, 300 kDa UF membranes, respectively. Further, average zinc(II) removal rate from MEUF unit, CF unit, and two ACF units (1st ACF, 2nd ACF) in various MWCOs UF membranes depicted the following decreasing trend:

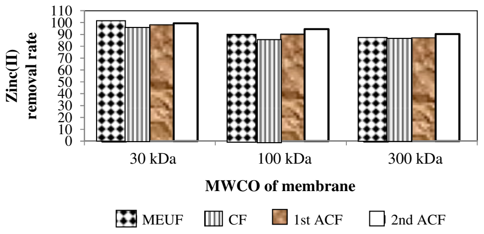


Fig. 9a. Comparative zinc(II) removal rate from various units of MEUF-ACF process.
Conditions: molar ratio of SDS to zinc(II) = 1:2; initial retentate pressure = 0.20 MPa; initial permeate flux = 43.64 L/m^2 h; pH = neutral (7.0); operational time = 2.0 h; MWCO = 30 kDa, 100 kDa, 300 kDa.

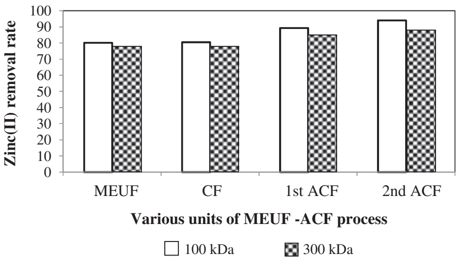


Fig. 9b. Comparative zinc(II) removal rate from various units of MEUF-ACF hybrid process.
Conditions: molar ratio of SDS to zinc(II) = 1:2; initial retentate pressure = 0.20 MPa; initial permeate flux = 43.64 L/m^2 h; pH = neutral (7.0); operational time = 4.0 h; MWCO = 100 kDa, 300 kDa.

Table 5
Comparative zinc(II) removal percentage from various units of MEUF-ACF hybrid process

300 kDa MWCO				100 kDa MWCO				30 kDa MWCO			
MEUF	CF	1st ACF	2nd ACF	MEUF	CF	1st ACF	2nd ACF	MEUF	CF	1st ACF	2nd ACF
79.37	79.28	87.07	90.21	81.75	80.85	90.13	94.45	90.76	87.43	98.06	99.41

Table 6
Comparative zinc(II) removal percentage from various units of MEUF-ACF hybrid process

300 kDa MWCO				100 kDa MWCO			
MEUF	CF	1st ACF	2nd ACF	MEUF	CF	1st ACF	2nd ACF
78.55	78.06	85.48	88.18	80.05	80.47	89.31	94.03

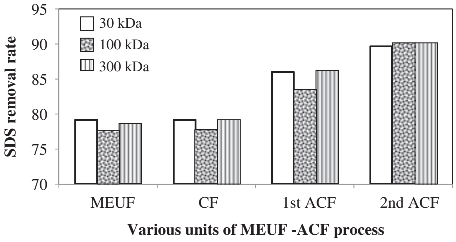


Fig. 10. Comparative SDS removal rate from various units of MEUF-ACF hybrid process.
Conditions: molar ratio of SDS to zinc(II) = 1:2; initial retentate pressure = 0.20 MPa; initial permeate flux = 43.64 L/m^2 h; pH = neutral (7.0); MWCO = 30 kDa, 100 kDa, 300 kDa.

30 > 100 > 300 kDa membranes (Figs. 9a and 9b) (Table 5).

In addition, average zinc(II) removal rate from various units of MEUF-ACF hybrid process depicted reduction by 0.83% (in case of 100 kDa membrane) and 1.42% (in case of 300 kDa membrane) in average when the experimental duration was extended to 4.0 h under the similar experimental condition (Tables 5 and 6). This might be due to release of some zinc(II) ions to the permeate with the enhancement in operational time.

3.8. SDS removal from various units of MEUF-ACF hybrid process

One of the major drawbacks of the MEUF process is the leakage of surfactant monomers in the filtrate or permeate that may induce the secondary pollution. Thus, series of experiments were conducted to investigate the removal of excess SDS monomers present in the MEUF permeate by coupling with ACF as shown in Fig. 2. Further, the comparative analysis was carried out regarding the SDS removal rate from various units of MEUF-ACF hybrid process viz. MEUF unit, CF unit, two ACF units (1st ACF and 2nd ACF), at constant initial SDS concentration and initial zinc(II) concentration of 0.21 mM (60.56 mg/L) and 0.42 mM (120.78 mg/L), respectively (Fig. 10).

As shown in Table 7, SDS removal rate was 79.18, 77.43, and 78.67% for 30, 100, and 300 kDa UF membrane, respectively. After coupling with the ACF units,

Table 7
Comparative SDS removal percentage from various units of MEUF-ACF hybrid process

300 kDa MWCO				100 kDa MWCO				30 kDa MWCO			
MEUF	CF	1st ACF	2nd ACF	MEUF	CF	1st ACF	2nd ACF	MEUF	CF	1st ACF	2nd ACF
78.67	79.0	86.36	90.19	77.43	77.79	83.55	90.03	79.18	79.18	86.0	89.68

SDS removal rate reached 89.68, 90.03, and 90.19% for 30, 100, and 300 kDa UF membrane, respectively. In conclusion, it can be stated that SDS removal rate of MEUF-ACF hybrid process was 10.5, 12.6, 11.52% higher than MEUF alone in 30, 100, 300 kDa UF membranes, respectively. As depicted in Table 7, SDS removal rate decreased with decrease in MWCO of UF membrane. This can be corroborated as larger pore-sized membranes caused earlier development of CP and reduced the release of surfactant in the permeate [33].

4. Conclusion

In MEUF process, zinc(II) removal from synthetic wastewater using relatively low surfactant concentration and high MWCO (or NMWL) UF membrane was investigated. Taking consideration primarily zinc(II) removal rate and permeate fluxes, the operating initial permeate flux of 43.64 $L/m^2 h$, operating retentate pressure of 0.20 MPa, molar ratio of zinc(II) to SDS of 1:2 (0.1392 mM of initial SDS concentration), and solution pH of neutral (i.e. 7.0) condition were found to be optimum operating parameters within the experimental conditions. Even though the initial SDS concentrations are far below CMC, average zinc(II) removal rate was 77.29 and 75.93% whereas the values of J_v was 39.64 and 10.45 $L/m^2 h$ at molar ratios of zinc(II) to SDS of 1:8 and 1:2, respectively. At molar ratio of SDS to zinc(II) 1:2 and during 2.5 h experimental operation, average zinc(II) removal rate was 79.06, 81.40, and 91.47% for 300, 100, and 30 kDa MWCO UF membrane, respectively.

In MEUF-ACF hybrid process at constant initial zinc(II) concentration (0.42 mM) and fixed initial SDS concentration (0.21 mM) as a function of varying MWCOs, average zinc(II) removal rate was 8.65, 12.70, and 10.84% higher than MEUF alone in 30, 100, and 300 kDa UF membranes, respectively. Similarly, SDS removal rate of MEUF-ACF hybrid process was 10.5, 12.6, and 11.52% higher than MEUF alone in 30, 100, and 300 kDa UF membranes, respectively. In conclusion, it can be stated that coupling of surfactant-based UF process and ACF adsorption technique, i.e. MEUF coupled with ACF integrated approach proved to be an effective remedial measure for metal and surfactant removal from wastewater.

Acknowledgements

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Abbreviations

PTEs	—	potentially toxic elements
USEPA	—	United States Environmental Protection Agency
SMCL	—	secondary maximum contaminant level
TCLP	—	total characteristics leachate procedure
MEUF	—	micellar enhanced ultrafiltration
UF	—	ultrafiltration
RO	—	reverse osmosis
CMC	—	critical micelle concentration (mM)
CP	—	concentration polarization
ACF	—	activated carbon fiber
MWCO	—	molecular weight cut-off (Dalton)
SDS	—	sodium dodecyl sulfate
PAC	—	powdered activated carbon
GAC	—	granular activated carbon
PAN	—	polyacrylonitrile
TMP	—	transmembrane pressure (MPa)
BET	—	Brunauer–Emmett–Teller
COD	—	chemical oxygen demand
CF	—	cartridge filter
MAL	—	micelle aggregation layer
NMWL	—	nominal molecular weight limit

Symbols

R^2	—	coefficient of determination
R	—	removal rate
C_p	—	permeate concentration (mg/L)
C_f	—	feed concentration (mg/L)
P_F	—	feed pressure (MPa)
P_R	—	retentate pressure (MPa)
P_P	—	permeate pressure (MPa)
J_v	—	absolute permeate flux ($L/m^2 h$)
V	—	volume of the permeate sample collected (L)
t	—	time needed for collecting the permeate sample (h)
A	—	membrane effective area (m^2)
J_w	—	pure water flux ($L/m^2 h$)
J_{sp}	—	specific flux (or permeability of membrane) ($L/m^2 h$ MPa)
L_p	—	filtration coefficient or hydraulic conductivity of membrane ($L/m^2 h$ MPa)
R (%)	—	recovery %
P	—	permeate volume (mL)
F	—	feed volume (mL)

Da — dalton
X — volume concentration factor (VCF)
C_v — volume concentration ratio (VCR)
y (%) — system conversion

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