



## Removal of cadmium ion using micellar-enhanced ultrafiltration (MEUF) and activated carbon fiber (ACF) hybrid processes: adsorption isotherm study for micelle onto ACF

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### ABSTRACT

Micellar-enhanced ultrafiltration (MEUF) was applied to remove cadmium ion from wastewater using sodium dodecyl sulfate (SDS) as a micelle in this study. Investigations were carried out on operational parameters such as initial permeate flux, retentate pressure, initial cadmium concentration, pH solution, molecular weight cut-off (MWCO), and molar ratio of cadmium to SDS. Removal efficiency of cadmium increased with increase in retentate pressure, pH solution, and molar ratio of cadmium to SDS and decreased with increase in initial permeate flux. Higher removal efficiency of cadmium was achieved using lower MWCO. In optimized experimental condition, cadmium removal efficiency reached 75% within an hour. With MEUF–ACF hybrid process, removal efficiencies of cadmium and SDS were found to be over 99 and 90%, respectively. Freundlich isotherm equation fitted better with experimental results on adsorption of SDS by ACF than Langmuir isotherm equation. Overall SDS removal efficiency of ACF unit in series was 90%.

*Keywords:* Micellar-enhanced ultrafiltration; Activated carbon fiber; Cadmium; Sodium dodecyl sulfate; Adsorption isotherm

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### 1. Introduction

Heavy metals are recognized as dangerous anthropogenic environmental pollutants, for their toxicity, bioaccumulation, persistence in the environment, biomagnification in the food chain [1]. Due to wide usages of cadmium (Cd) in numerous industries, wastewater containing this metal can contaminate soil

and subsequently seep into groundwater. If directly discharged into the sewage system, such contaminated water may not only seriously damage the operation of biological treatment plants, but may also render the activated sludge generated unsuitable for the application to agricultural land [2]. The potential human health impacts of cadmium are: carcinogen, developmental toxicant, respiratory toxicant, reproductive toxicant, cardiovascular or blood toxicant, kidney toxicant, neurotoxicant, immunotoxicant, and endocrine toxicant

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[3]. At present, popular techniques for treating cadmium-bearing wastewater are chemical precipitation, adsorption, bleaching powder oxidation, ferrite process, ions exchange, and biotechnology. These techniques have their limitations such as secondary pollution of deposition, inconvenient operation, high cost, difficulty of recycling cadmium, and others [4].

Micellar-enhanced ultrafiltration (MEUF) has shown to be a promising technique for the removal of lower molecular weight substances, as it combines the efficiency of reverse osmosis and the high flux of surfactant-based ultrafiltration (UF) [5]. In this process, surfactant with a charge opposite to the target ion is added to the wastewater containing metal ions. The surfactant molecules will aggregate and form spherical micelles (around 50–150 of monomer molecule) when the surfactant concentration in the wastewater is at a concentration greater than critical micelle concentration (CMC) [6,7]. A large fraction of the metal ions is, therefore, electro-statically attached to the micelle surface. The wastewater can then be ultrafiltered through an ultrafiltration membrane with a pore size smaller than the micelle size, in order to reject the micelles. At the same time, cadmium ions adsorbed onto the micelles are rejected and the permeation quality is adequate for reuse or direct discharge to the environment.

Addition of surfactant to the MEUF system results in release of some of the surfactant in permeate. One of the major drawbacks of the MEUF process is the production of surfactant-rich effluent, which needs to be treated before discharge to the environment, as this may otherwise cause secondary pollution. Adsorption technology is commonly used for the removal of surfactants [8] as well for the removal of trace heavy metals from an aqueous solution. Commonly used media for adsorption processes are powdered activated carbon, granular activated carbon, and activated carbon fiber (ACF). Among these, ACF has faster adsorption kinetics, a uniform micro-pore structure, and lower pressure drop [9,10]. In the MEUF-ACF hybrid process, heavy metals are effectively removed by the MEUF unit, and surfactant-rich MEUF effluent containing trace heavy metals is treated with the ACF unit. The main objective of this study is to investigate the optimal operational condition for the MEUF process for the removal of cadmium from wastewater using sodium dodecyl sulfate (SDS), and to investigate the performance of the MEUF-ACF hybrid process for SDS removal, as well as for the removal of trace cadmium from the MEUF effluent. Moreover, another objective of this research is to investigate the adsorption isotherm models of SDS by ACF with different concentrations of SDS and the removal efficiency of SDS by dual ACF unit.

## 2. Materials and methods

In this study, cadmium nitrate tetrahydrate ( $\text{CdN}_2\text{O}_6 \cdot 4\text{H}_2\text{O}$ ) of 99% purity was procured from Sigma-Aldrich Co., USA (molecular weight of 308.48) and SDS of 99% purity was procured from Acros Organic Ltd., USA (molecular weight of 288.38). These were respectively used as the source of cadmium and surfactant for the preparation of the feed solution. The surfactant was used without any further treatment. Details of MEUF experimental operating conditions are summarized in Table 1.

All solutions were prepared using distilled water. Solutions were prepared by mixing stoichiometric amounts of SDS surfactant and cadmium in 8 L of distilled water for an hour with 100 rpm. Hollow fiber membrane (Chemicore Ltd., Korea), having two kinds of molecular weight cut-off (MWCO) sizes, were used for the experiment. Ultrafiltration is a cross-flow type of filtration process, in which the rejected permeate is re-circulated into the feed tank and permeate water is collected at the separation tank.

The experimental module consisted of a feed tank, ultrafiltration membrane, wash-out tank, and permeate tank as shown in Fig. 1. The ACF unit comprised a cartridge filter (CF) connected with a feed tank to prolong the life span of the ACF. The characteristics of the membrane used in this process are presented in Table 2. In addition to the CF, two sets of ACF cartridge units were connected in series. ACF was purchased from ACF Korea Ltd., whose cartridge code no. is FC-B. Bulk density and iodine number of ACF were  $0.2 \text{ kg/m}^3$  and  $1,500 \text{ mg/g}$ , respectively. After each series of experiments, UF membrane was flushed and backwashed with the distilled water and cleaned with 0.1 M NaOH and 0.5% HCl. CF and ACF unit was cleaned with distilled water before soaking in 0.1 M of NaOH and 2% of HCl for a day. Cadmium concentration was measured using inductively coupled plasma (ICP, Varian OES-720) with a wavelength of 214.439 nm. Samples of MEUF were pretreated

Table 1  
Details of MEUF experimental operating conditions

Retentate pressure (MPa)	0.2, 0.24, 0.28
pH	3, 5, 7, 8.6
Initial cadmium concentration (mM)	0.065, 0.1621, 0.1784, 0.3242
Molar ratio of cadmium to SDS	1:2, 1:5, 1:10, 1:12
Sampling time (min)	5, 10, 20, 30, 40, 50, 60
Initial flux (distilled water) ( $\text{L/m}^2 \text{ h}$ )	31.3, 42.9, 55.3, 65.5
Membrane pore size (Da)	100,000–300,000
ACF flux ( $\text{m}^3/\text{d}$ )	0.06

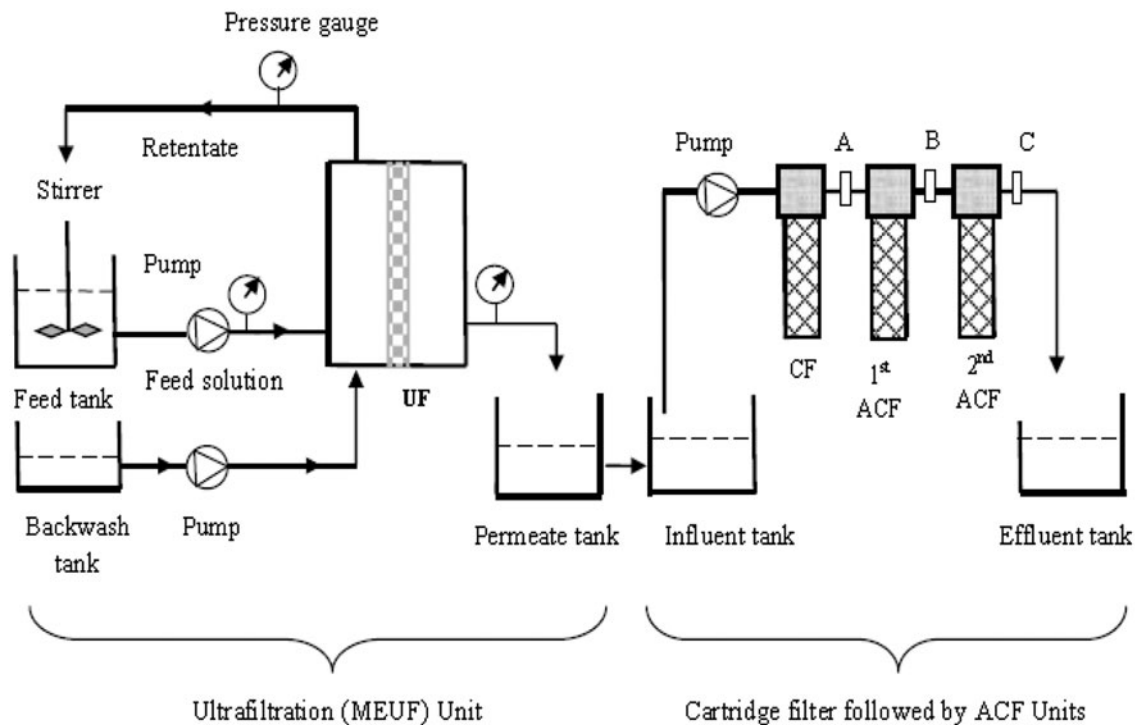


Fig. 1. Experimental setup for MEUF-ACF hybrid processes.

Table 2  
Characteristics of UF membrane and ACF unit

Membrane material	Polyacrylonitrile
Membrane type	Hollow fiber
Flow direction	Inside to outside
Flow type	Cross flow
Effective surface area (m <sup>2</sup> )	0.055
Membrane diameter (inside/outside) (mm)	0.8/1.4
Molecular weight cut-off (MWCO)	100,000, 300,000
ACF BET surface area (m <sup>2</sup> /g)	1,000
Weight of ACF (g/cartridge)	30

according to standard methods for the examination of water and wastewater [11]. SDS was measured using chemical oxygen demand as per Standard Methods. Cadmium and SDS removal efficiencies were calculated using Eq. (1).

$$R = (1 - C_p/C_i) \times 100 \quad (1)$$

where,  $R$  = rejection (%);  $C_p$  = permeate concentration (mg/L);  $C_i$  = influent concentration (mg/L). Similarly, adsorption of SDS on ACF was conducted and adsorptive capacity of ACF was calculated using Freundlich

[12] and Langmuir [13] isotherm equations as indicated by Eqs. (2) and (3), respectively.

$$\log q = \log k + 1/n \times \log C_e \quad (2)$$

where,  $q$  = adsorbate adsorbed per unit weight of adsorbent (mg/g);  $C_e$  = final concentration of adsorbate in solution (mg/L);  $n$  = constant representing the adsorption intensity (dimensionless);  $k$  = Freundlich capacity factor (mg adsorbate per g activated carbon (L water per mg adsorbate)<sup>1/n</sup>).

$$1/q = 1/q_m + 1/bq_m \times 1/C_e \quad (3)$$

where,  $q_m$  = maximum adsorption at monolayer coverage (mg/g);  $b$  = adsorption equilibrium constant related (mg/g) to energy of adsorption (L/mg).

### 3. Results and discussion

#### 3.1. Effect of permeate flux on cadmium removal

A series of experiments were conducted using various permeate fluxes. Fig. 2(a) shows the removal characteristics of cadmium at various permeate fluxes. Average cadmium removal efficiency was 43.0% with

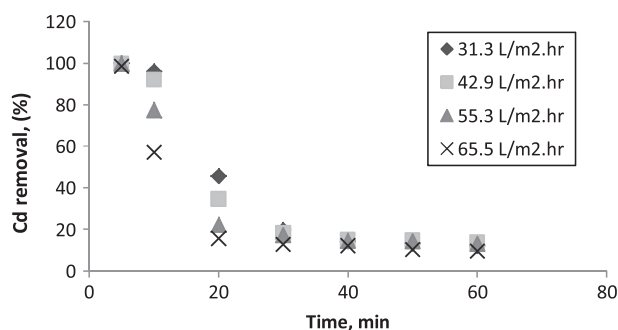


Fig. 2a. Effect of permeate flux on cadmium removal efficiency (retentate pressure = 0.2 MPa, initial concentration of cadmium = 0.1621 mM, molar ratio of cadmium to SDS = 1:5, MWCO of membrane = 100,000 Da).

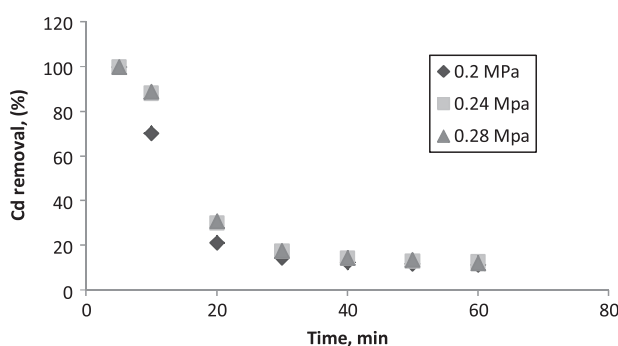


Fig. 2b. Effect of retentate pressure on cadmium removal efficiency (permeate flux = 42.91 L/m<sup>2</sup> h, initial concentration of cadmium = 0.1621 mM, molar ratio of cadmium to SDS = 1:5, MWCO of membrane = 100,000 Da).

permeate flux of 31.3 L/m<sup>2</sup> h, while it was 41.1, 36.9, and 30.9% with the initial permeate fluxes of 42.9, 55.3, and 65.5 L/m<sup>2</sup> h, respectively. This implied that cadmium removal efficiency decreased with an increase of permeate flux within the operational experimental range. In UF process, an increase in permeate flux led to an increase in concentration polarization (CP) on the membrane surface [14]. Flux decline is the main bottle-neck of this process. This is mainly caused by CP, fouling, and adsorption [15]. Considering the higher removal efficiency of cadmium (43.0%) achieved at this value, 31.3 L/m<sup>2</sup> h was found to be the optimum initial permeate flux in this study.

### 3.2. Effect of retentate pressure

The effect of initial retentate pressure on cadmium removal was investigated under various initial retentate pressures. As shown in Fig. 2(b), average

cadmium removal was 34.4% with initial retentate pressure of 0.2 MPa, whereas it was 39.2 and 39.6% at the initial pressures of 0.24 and 0.28 MPa, respectively. Cadmium removal increased with an increase in initial retentate pressure similar to that obtained in previous studies [14]. With an increase in retentate pressure, transmembrane pressure (TMP) also increased. The operation of membrane process at low TMP is an important issue in terms of minimizing operating costs. An increase in pressure actually increases the gel layer thickness, in turn, increasing the rejection of the metal–micelle complex. Moreover, after 40 min of operation (Fig. 2(b)), the cadmium removal became linear for all the three cases. For these reasons, the optimum retentate pressure was found to be 0.2 MPa.

### 3.3. Effect of molar ratio of cadmium to SDS

To find the effect of molar ratio of cadmium to SDS, another series of experiments were conducted at different molar ratios of cadmium to SDS. Fig. 3(a) shows that average cadmium removal was 74.6% for a molar ratio of 1:10. Removal efficiency decreased to 72.9 and 69.5%, at molar ratios of 1:5 and 1:2, respectively. Cadmium removal increased with increase in molar ratio. Cadmium removal efficiency was higher with higher initial SDS concentration that produced more micelles, making more micelle surface area available for electrostatic attraction of cadmium ions. Cadmium ions were retained on the membrane surface along with the micelles [2]. After surfactant concentration reaches CMC, all the surfactant added is converted to micelles. It then provides more available surface area for electrostatic attraction. Surfactant monomers cannot form micelles unless they reach CMC, and monomers pass through the membrane

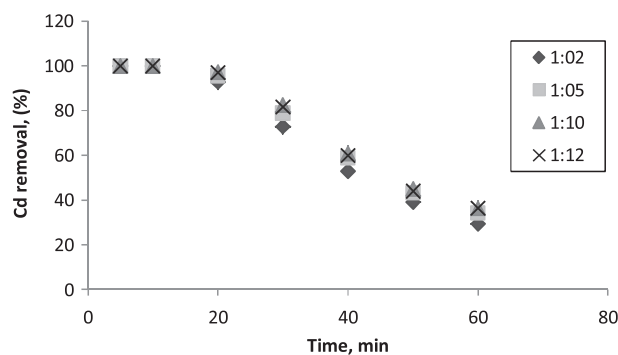


Fig. 3a. Effect of molar ratio of cadmium to SDS on cadmium removal efficiency (permeate flux = 42.91 L/m<sup>2</sup> h, retentate pressure = 0.2 MPa, initial concentration of cadmium = 0.1621 mM, MWCO of membrane = 100,000 Da).

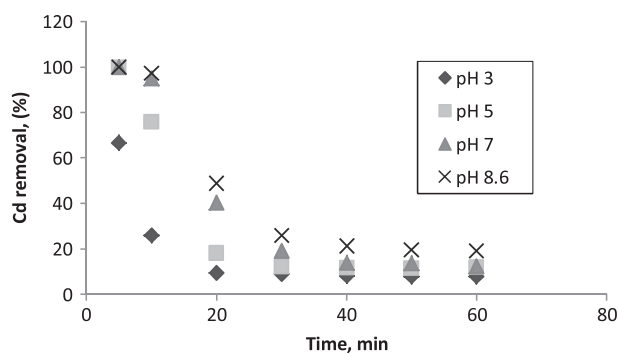


Fig. 3b. Effect of pH on cadmium removal efficiency (permeate flux = 42.91 L/m<sup>2</sup> h, retentate pressure = 0.2 MPa, initial concentration of cadmium = 0.1621 mM, molar ratio of cadmium to SDS = 1:5, MWCO of membrane = 100,000 Da).

together with pollutant [16]. This results in a large micelle surface area being available for electrostatic attraction of cadmium ions. As a result, a higher quantity of cadmium is removed together with the micelles.

### 3.4. Effect of pH

A series of experiments were performed to investigate the effect of pH on cadmium removal. As shown in Fig. 3(b), average cadmium removal efficiency was 19.3 and 34.4% for feed solution pH values of 3 and 5, respectively, while it was 42.1 and 47.4% for pH values of 7 and 8.6, respectively. Cadmium removal increased with an increase in pH of the feed solution. At lower pH, cadmium needs to compete with H<sup>+</sup> ions for the micelle surface. Under acidic conditions, due to competition with H<sup>+</sup> ions, less cadmium was adsorbed onto the micelle surface leading to a reduction in cadmium removal. On the contrary, at higher pH, H<sup>+</sup> bound with functional groups, can be dissociated easily, and the deprotonated functional groups can bind with cadmium ions [17]. Previous research has also shown that copper removal increased with the increased acidity of feed solution [18]. The effect of pH depends on the type of metals used in the solution, and specifically on, whether H<sup>+</sup> ions compete with the metal during electro-static adsorption on micelles.

### 3.5. Effect of initial concentration of cadmium

Another series of experiments were conducted with SDS concentration of 0.3242 mM at various initial cadmium concentrations in feed solution as shown in Fig. 4. Average cadmium removal efficiency was 58.7% for 0.065 mM concentration of cadmium. For

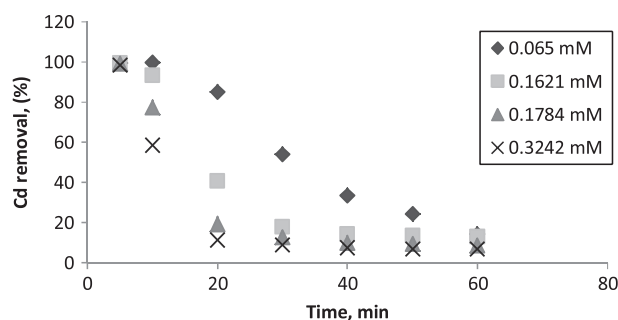


Fig. 4. Effect of initial concentration of cadmium on cadmium removal efficiency (permeate flux = 42.91 L/m<sup>2</sup> h, retentate pressure = 0.2 MPa, SDS concentration = 0.3242 mM, MWCO of membrane = 100,000 Da).

initial cadmium concentration of 0.1621, 0.1784, and 0.3242 mM, average cadmium removals were 41.7, 33.8, and 28.3%, respectively. Cadmium concentration in permeate increased with an increase in initial concentration, mainly due to less micelle surface area being available for electrostatic adsorption of higher concentrations [16]. Average permeate flux remained almost the same for the given surfactant concentration, and the charge surface available for cadmium on the micelle surface remained constant for constant initial surfactant concentration. This resulted in lower removal of cadmium at its higher concentration in feed solution [19].

### 3.6. Effect of MWCO

To investigate the effect of membrane pore size, another series of experiments were conducted with varying pH values and different molar ratios (cadmium to SDS) using ultrafiltration membranes of MWCO 100,000 and 300,000 Da. Cadmium removal efficiencies using 100,000 Da MWCO membrane were found to be 19.3, 34.4, 42.1, and 47.4%, with pH values of 3, 5, 7, and 8.6, respectively. Similarly cadmium removal efficiencies using 300,000 Da MWCO membrane were 14.6, 20.8, 21.8, and 32.9% for the same pH values, respectively (Fig. 5(a)). Previous research has also shown similar trend [19]. On the other hand with different molar ratios (cadmium to SDS) of 1:2, 1:5, 1:10, and 1:12, average cadmium removal efficiencies using 100,000 Da MWCO membrane were found to be 69.5, 72.9, 74.6, and 74.1%, respectively, while with the same molar ratios, removal efficiencies using the 300,000 Da MWCO membrane reduced to 26.1, 29.6, 31.9, and 32.9%, respectively (Fig. 5(b)). Similar results were obtained in previous studies on the removal of anionic pollutants through MWCO of 100,000 and 300,000 Da [20].

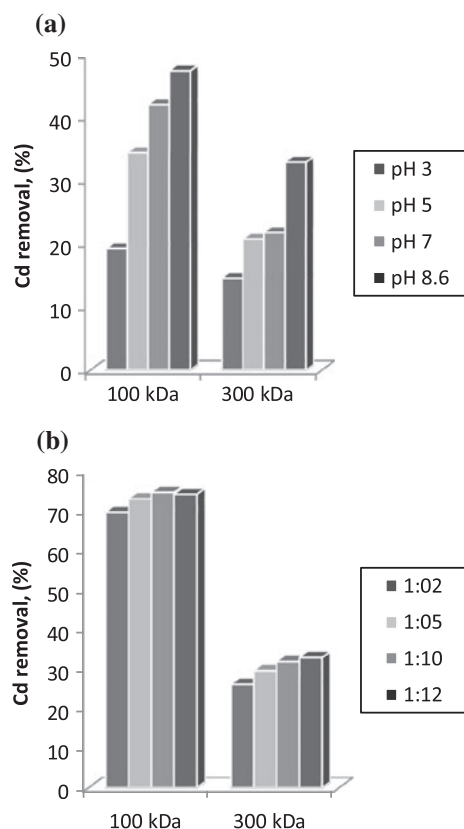


Fig. 5. Effect of MWCO of membrane on cadmium removal efficiency (a) in various pH (permeate flux = 42.91 L/m<sup>2</sup> h, retentate pressure = 0.2 MPa, MWCO of membrane = 100,000 and 300,000 Da) and (b) in various molar ratios of cadmium to SDS (permeate flux = 31.27 L/m<sup>2</sup> h, retentate pressure = 0.2 MPa, MWCO of membrane = 100,000 and 300,000 Da).

### 3.7. Removal of cadmium by MEUF without SDS

An experiment was conducted without surfactant (SDS), in feed solution containing initial cadmium concentration of 20 mg/L (0.065 mM). As shown in Table 3, cadmium removal percentage was 22.2% during the 60 min of operation. This strongly indicated inefficiency of UF alone in the removing of cadmium

ions from feed solution. In the MEUF process, the removal of pollutants is primarily due to the screening action of the UF membrane and adsorption of micelle-metal complexes on the membrane surface as well as inside the pore walls of the membrane [21].

### 3.8. Performance of MEUF–ACF hybrid process

A series of experiments were conducted to investigate the removal of excess cadmium ions present in the MEUF permeate by coupling with ACF. Further, comparative analysis was carried out regarding the average cadmium removal percentage from various units of MEUF–ACF hybrid process such as MEUF, CF, and two ACF units (ACF1 and ACF2), at constant initial cadmium concentration and initial SDS concentration of 0.065 mM (20 mg/L) and 0.3242 mM (93.48 mg/L), respectively.

As shown in Table 4(a), the 100,000 Da UF membrane has shown an average cadmium removal percentage of 68.5% for initial cadmium concentration of 0.065 mM during 1 h operational time. Average cadmium removal percentage reached 99.6% when coupled with ACF units. On the other hand, the 300,000 Da UF membrane produced an average cadmium removal percentage of 36.4%, and the cadmium removal percentage reached 99.5% after being combined with the ACF units. Furthermore, average cadmium removal percentages from the MEUF, CF, and two ACF units were higher when using 100,000 Da UF membrane rather than 300,000 Da UF membrane.

One of the major drawbacks of the MEUF process is the leakage of surfactant monomers in the filtrate or permeate, possibly inducing secondary pollution. Thus, a series of experiments were conducted to investigate the removal of excess SDS monomers present in the MEUF permeate by coupling with ACF. Furthermore, comparative analysis was carried out regarding SDS removal percentage from various units of MEUF–ACF hybrid process such as MEUF, CF, and two ACF units (ACF1 and ACF2), at constant initial cadmium concentration and initial SDS concentration

Table 3  
Cadmium removal without using SDS

Time (min)	Permeate conc. (ppm)	Removal efficiency (%)	Flux rate (L/m <sup>2</sup> h)
30	3.66	68.2	31.3
40	6.36	44.8	31.3
50	8.0	30.6	31.3
60	8.96	22.2	31.3

Notes: Initial cadmium concentration = 20 mg/L, initial retentate pressure = 0.2 MPa, initial permeate flux = 31.27 L/m<sup>2</sup> h, and MWCO of the membrane = 100,000 Da.

Table 4a  
Comparative cadmium removal percentage from various units of MEUF–ACF hybrid process

100,000 Da				300,000 Da			
MEUF	CF	ACF1	ACF2	MEUF	CF	ACF1	ACF2
68.5	69.7	78.5	99.6	36.4	41.3	64.6	99.5

Notes: Molar ratio of SDS to cadmium = 1:5, initial retentate pressure = 0.2 MPa, initial permeate flux = 31.27 L/m<sup>2</sup> h, pH = neutral, operation time = 5 h, and MWCO = 100,000 and 300,000 Da.

Table 4b  
Comparative SDS removal percentage from various units of MEUF–ACF hybrid process

100,000 Da				300,000 Da			
MEUF	CF	ACF1	ACF2	MEUF	CF	ACF1	ACF2
34.6	41.5	70.8	91.0	31.3	40.4	63.2	89.9

Notes: Molar ratio of SDS to cadmium = 1:5, initial retentate pressure = 0.2 MPa, initial permeate flux = 31.27 L/m<sup>2</sup> h, pH = neutral, operation time = 5 h, and MWCO = 100,000 and 300,000 Da.

of 0.065 mM (20 mg/L) and 0.3242 mM (93.48 mg/L), respectively.

As shown in Table 4(b), SDS removal percentages were 34.6 and 31.3% by MEUF for 100,000 and 300,000 Da UF membranes, respectively. After coupling with the ACF units, SDS removal percentages reached to 91.0 and 89.9% for 100,000 and 300,000 Da UF membranes, respectively. In conclusion, it can be stated that the SDS removal percentage was much higher in the MEUF–ACF hybrid process as compared to results obtained when using MEUF alone. This was the case with both 100,000 and 300,000 Da membranes. As seen in Table 4(a), SDS removal percentage decreased with an increase 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 [22].

Table 5  
Freundlich and Langmuir isotherm parameters

Freundlich isotherm model			Langmuir isotherm model		
<i>n</i>	<i>k</i>	<i>R</i> <sup>2</sup>	<i>b</i>	<i>q</i> <sub>m</sub>	<i>R</i> <sup>2</sup>
0.831	88.308	0.997	2.57	142.86	0.986

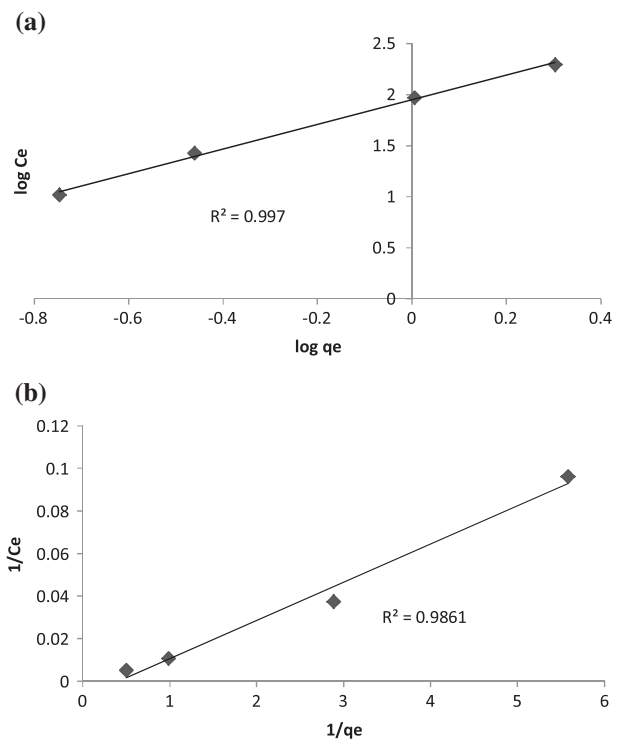


Fig. 6. Freundlich isotherm model (a) and Langmuir isotherm model (b) on the adsorption of SDS by ACF unit.

### 3.9. Investigate adsorption isotherm study for SDS on ACF

Adsorptive capacity of ACF for SDS was identified from batch experimentation. Freundlich and Langmuir isotherm equations were used to calculate the adsorptive capacity. Adsorption parameters are summarized in Table 5. The linearized form of the Freundlich isotherm equation and Langmuir equation are expressed in Fig. 6(a) and (b), respectively. The value of the regression coefficient (*R*<sup>2</sup>) obtained, 0.997, showed that Freundlich isotherm gave a better fit to the experimental data than the Langmuir isotherm (*R*<sup>2</sup> = 0.986). The similar results were figured out in the previous research papers [23,24].

### 3.10. SDS removal by ACF filter

MEUF process can remove SDS only to a lower concentration, so the leakage of SDS in the permeate can create secondary pollutants. A set of experiments were carried out to study the SDS removal in ACF filtration unit. As shown in Table 6, SDS removals were 89.6, 86.7, 84.4, and 83.8% at the initial concentration of SDS 100, 200, 600, and 1,200 mg/L, respectively. Two ACF units in series have removed SDS efficiently from the wastewater.

Table 6  
SDS removal by ACF filtration unit at various initial concentrations

Average permeate SDS concentration and removal efficiency							
1,200 mg/L		600 mg/L		200 mg/L		100 mg/L	
$C_e$ (mg/L)	Removal (%)	$C_e$ (mg/L)	Removal (%)	$C_e$ (mg/L)	Removal (%)	$C_e$ (mg/L)	Removal (%)
194.9	83.8	93.8	84.4	26.7	86.7	10.4	89.6

#### 4 Conclusion

In the MEUF process, the average cadmium removal efficiency under optimum operating conditions was 74.6% at neutral pH. Furthermore, the 100,000 Da MWCO membrane was better choice for higher cadmium removal efficiency over 300,000 Da MWCO membrane. During 60 min of ultrafiltration without SDS, the cadmium removal percentage was 22.2%. In the MEUF-ACF hybrid process, removal efficiencies of cadmium and SDS at the end effluent were 99.6 and 91.0% with 100,000 Da membrane whereas 99.5 and 89.9% with 300,000 Da membrane, respectively. Adsorption isotherm for SDS on ACF showed, Freundlich isotherm equation was obeyed better than Langmuir isotherm equation with experimental results. The SDS removal efficiency of ACF unit was 90%.

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