



Micellar enhanced ultrafiltration (MEUF) and activated carbon fibre (ACF) hybrid processes for nickel removal from an aqueous solution

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Received 10 November 2011; Accepted 6 February 2012

ABSTRACT

In the present study, nickel removal from an aqueous solution using micellar enhanced ultrafiltration at various operating parameters such as initial permeate flux, retentate pressure, initial nickel concentration, pH, molecular weight cut-off and molar ratio of nickel to sodium dodecyl sulphate (SDS) was investigated. The SDS surfactant removal from an aqueous solution using activated carbon fibre (ACF) was also monitored. The removal efficiency of nickel from an aqueous solution increased with an increase in the retentate pressure, initial permeate flux, pH and molar ratio of nickel to SDS, while the specific and relative fluxes declined. Considering the nickel removal efficiency and the permeate flux, initial permeate flux of $1.05 \text{ m}^3/\text{m}^2/\text{day}$, nickel to SDS molar ratio of 1:10 and operating retentate pressure of 1.4 bar were found to be the optimum operating parameters within the experimental condition for 0.5 mM or less initial nickel concentration. At the optimized experimental condition, the nickel removal efficiency was 98% and the corresponding permeate nickel concentration was less than 1 mg/L. Similarly, two ACF cartridge units in series have removed the SDS up to 85% and the adsorptive capacity of ACF for SDS was 170 mg/g. The Langmuir isotherm equation fitted better with the experimental results than the Freundlich isotherm equation.

Keywords: Micellar enhanced ultrafiltration (MEUF); Nickel; Sodium dodecyl sulphate (SDS); Activated carbon fibre (ACF)

1. Introduction

Water contamination by heavy metals in ecosystem is a major environmental problem encountered in many industrial areas. When the wastewater contaminated with heavy metals is disposed and used for irrigation purpose, it will adversely affect the plants. However, if the heavy metal contaminated wastewater gets mixed with the municipal wastewater, it will also inhibit the biological treatment process [1,2]. These

heavy metals are found in various concentrations in any natural resource of water and have detrimental health effects on human beings. Most of the heavy metals are carcinogenic in nature affect the and lungs, skin and cause many related diseases. Nickel is a toxic heavy metal that can cause adverse effects on both human beings and the environment. Nickel released into the environment contaminates the soil and then seeps into groundwater. The most common adverse health effect of nickel on human beings is an allergic dermatitis. Overexposure to nickel may lead to acute

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effects such as an organ iron content, blood and enzymatic changes, and a decrease in the body weight [3]. Similarly, nickel exceeding its critical level can bring about serious lung and kidney problems aside from gastrointestinal distress, pulmonary fibrosis and skin dermatitis [4].

Membrane technology is advancing rapidly as a powerful tool to abate the looming water crisis and reduce quality degradation of water resources. Among the various pressure-driven membranes, ultrafiltration (UF) is the one which has a high flux and low energy requirement compared to nanofiltration and reverse osmosis (RO). And micellar enhanced ultrafiltration (MEUF) has been proved as a promising technique to remove lower molecular weight substances as it combines the efficiency of RO and high flux of UF [5]. In the MEUF process, the surfactant having charge opposite to the target ion is added to the effluent stream containing the metal ions at a concentration greater than the critical micelle concentration (CMC), so that they form aggregates of around 50–150 of monomer molecule called micelles [6]. Therefore, a large fraction of the metal ions get electrostatically attached to the micelle surface. The retention of such metal ions attached to the micelles is possible if the resulting solution is passed through an ultrafilter membrane, having a pore size smaller than the micelle diameter [7]. The main problem in the MEUF process is “membrane fouling”, mainly caused by concentration polarization (CP) and pore blocking [8]. Membrane fouling can be minimized using optimum initial permeate flux, retentate pressure and initial feed concentration of the pollutant and surfactant. And it can be regenerated by frequent backwashing with clean water or cleaning with chemicals like NaOH and HCl [9].

The surfactant holds the major operating cost in MEUF process and the recovery of a surfactant is of utmost importance in this process from economical and environmental points of view. One of the major drawbacks of the MEUF process is the production of a surfactant-rich effluent, which needs to be removed before discharging to the environment as it may cause secondary pollution. And the remedial measure for solving this problem is the adsorption process, which has proven to be an economical and efficient methods for removing trace heavy metal and organics from wastewater [10]. And the commonly used media for adsorption for this process are Powdered Activated Carbon (PAC), Granular Activated Carbon (GAC) and Activated Carbon Fibre (ACF). Compared to PAC and GAC, ACF has a uniform micro-pore structure, faster adsorption kinetics and lower pressure drop [11]. In the MEUF-ACF hybrid process, MEUF can be effectively used for heavy metals removal and the ACF for

the surfactant removal from the surfactant-rich MEUF effluent containing trace heavy metals. In the present study, the main objectives are to investigate the performance of an MEUF process in nickel removal from wastewater using sodium dodecyl sulphate (SDS) at various operating parameters and to validate the effectiveness of ACF on SDS removal as a coupling unit with MEUF process.

2. Materials and methods

In the present study, nickel of 97% purity (molecular weight of 248.85) and SDS of 99% purity (molecular weight of 288.38) were used as the source of nickel and surfactant, respectively. Characteristics of the membrane and the ACF used in the experiment are given in Table 1.

All the solutions were prepared using distilled water. It was prepared by mixing stoichiometric amounts of the SDS surfactant and nickel in eight litres of distilled water for 2 h. The mixing speed was kept at 500 rpm. Hollow fibre membrane having three kinds of molecular weight cut-off (MWCO) sizes produced from Chemicore Ltd., Korea was used for the entire experiment. UF is a cross-flow type in which the rejection of permeate is re-circulated into the feed tank and permeate water is collected at the separation tank.

The experimental module consists of a feed tank, UF membrane, washout and permeate tank as shown in Fig. 1. The ACF unit comprises of a cartridge filter (CF) which was connected with a feed tank to prolong the life span of the ACF. The deviation of the adsorption capacity of this ACF was found to be 1.97 mg/g and 2.72 mg/g for the Langmuir and Freundlich isotherms, respectively. Based on the isotherm equilibrium, the correlation coefficients for Langmuir and Freundlich equations were 0.99 and 0.98 respectively. Characteristics of the membrane used in this process are presented in Table 2.

Following the CF, two sets of ACF cartridge units in series were connected. The ACF was purchased from ACF Korea Ltd., whose cartridge code number is FC-B. Bulk density and iodine number of the ACF were 0.2kg/m³ and 1,500mg/g, respectively. After

Table 1
Details of the MEUF experimental operating conditions

Retentate/pressure, bar	1.4
pH	5, 7, 9, 10
Initial nickel concentration, mg/L	20
Molar ratio of the nickel to SDS	1:0.5 to 1:30
Sampling time, min.	0, 10, 20, 30, 40, 50, 60
Initial flux (distilled water), L/m ² h	43.6

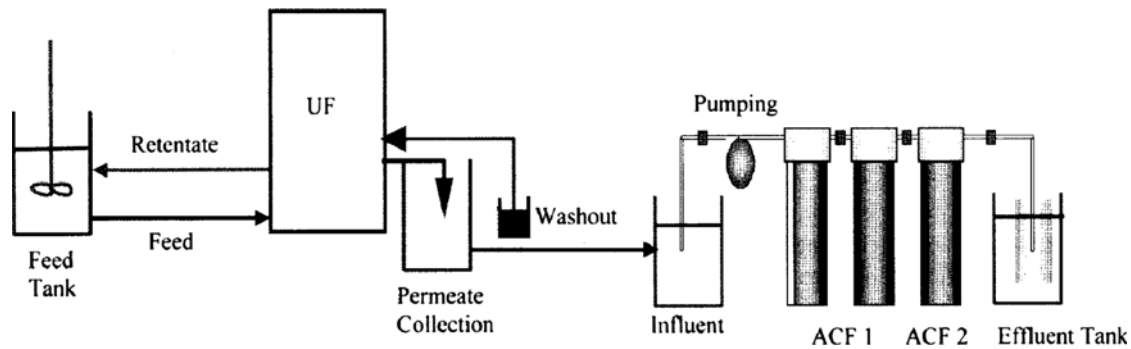


Fig. 1. Experimental set-up for MEUF-ACF hybrid processes.

Table 2
Characteristics of an MEUF membrane and an ACF unit

Membrane material	Polyacrylonitrile
Membrane type	Hollow fibre
Flow direction	Inside to outside
Flow type	Cross-flow
Effective surface area, m ²	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 ² /g	1,000
Weight of ACF, g/cartridge	30

each series of experiments, The UF membrane was flushed and backwashed with the distilled water, and cleaned with 0.1M NaOH and 0.5% HCl. the CF and ACF were cleaned with distilled water before soaking mixture into 0.1M of NaOH and 2% of HCl for a day.

3. Results and discussion

3.1. Effect of initial permeate flux on nickel removal

A series of experiments was carried out varying the initial permeate flux. The removal characteristics of nickel are shown in Fig. 2. The average nickel removal efficiency was 55% at the permeate flux of 1.31 m³/m²/day, while it was 51 and 54% for the initial permeate flux of 0.79 and 1.05 m³/m²/day, respectively. It implies that the nickel removal efficiency increased with an increase in the flux within the operational experiment range.

With the increase in permeate flux, CP on the membrane surface increased. As a result, bulk SDS concentration at the surface becomes higher and enhances the nickel removal [12]. In a surfactant-based UF process, flux decline is the main bottleneck of the process, which is mainly caused by CP, fouling and adsorption [13].

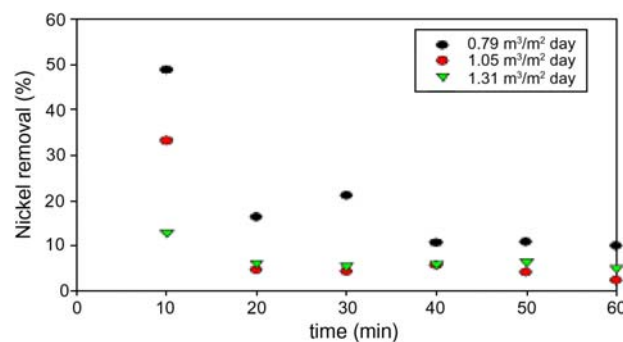


Fig. 2. Effect of initial permeate flux on the nickel removal efficiency (retentate pressure = 1.4 bar, molar ratio of nickel to SDS = 1:2, initial concentration of nickel = 20 mg/L, MWCO of membrane = 100,000 Da).

3.2. Effect of retentate pressure

Another series of experiments were carried out at various initial retentate pressures. Fig. 3 shows the nickel removal efficiency as a function of retentate pressure. The average nickel removal efficiency was found to be 54% for 1.4 bar retentate pressure, whereas it was found to be 61 and 63% for 1.8 and

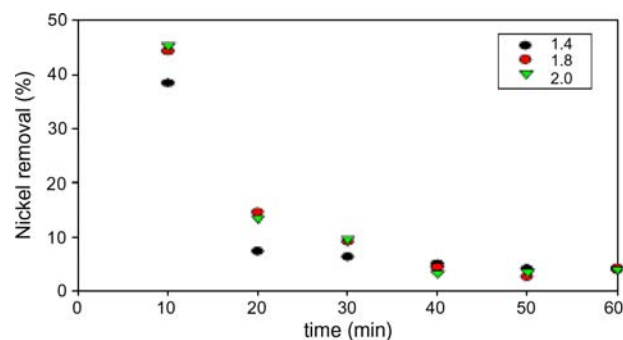


Fig. 3. Effect of retentate pressure on nickel removal efficiency (permeate flux = 1.05 m³/m²/day, molar ratio of nickel to SDS = 1:2, initial concentration of nickel = 20 mg/L, MWCO of membrane = 100,000 Da).

2.0 bar retentate pressures, respectively. The nickel removal increased with an increase in the initial retentate pressure. A similar result was achieved on copper [14]. The increase of pressure increased the gel layer thickness, which results in an increase in the rejection of the nickel–micelle complex.

3.3. Effect of molar ratio of nickel to SDS

Another series of experiment were conducted at various molar ratios of nickel to SDS to observe the influence of molar ratio of nickel to SDS concentration on nickel removal efficiency. Fig. 4 shows the nickel removal efficiency during 60 min of operational time at the nickel to SDS molar ratios of 1:0.5–1:30. The average nickel removal efficiency was 98.8% at a molar ratio of 1:30. The nickel removal efficiency was constant at 99.5 and 98.6% at the molar ratios of 1:20 and 1:10, respectively. The corresponding nickel removal efficiency for the molar ratios 1:5 and 1:2.5 was 89.6 and 63.4%, respectively. The nickel removal increased with an increase in the molar ratio. It means that the nickel removal efficiency was higher for a higher initial SDS concentration, which produces more micelles. After the surfactant concentration reaches the CMC at 8.2 mM, all the surfactant added gets converted to micelle. It then provides more surface area available for an electrostatic attraction. Surfactant monomers cannot form micelles unless they reach the CMC. Monomers pass through the membrane together with the pollutant, which results in a larger micelle surface area available for an electrostatic attraction of the nickel ions. As a result, a higher quantity of nickel was removed together with the micelles. As regards the nickel removal efficiency, the molar ratio of 1:10 was found to be the most optimum condition.

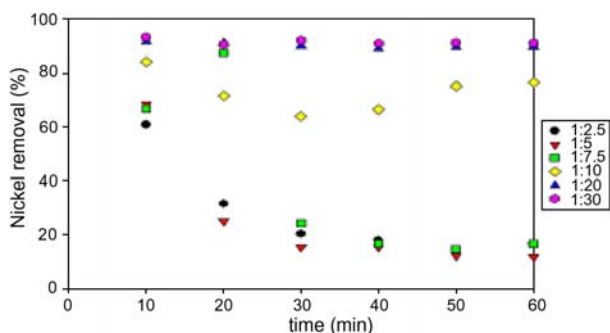


Fig. 4. Effect of molar ratio of nickel to SDS on nickel removal efficiency (permeate flux = $1.05 \text{ m}^3/\text{m}^2/\text{day}$, retentate pressure = 1.4 bar, initial concentration of nickel = 20 mg/L, MWCO of membrane = 100,000 Da).

3.4. Effect of pH

The other series of experiments were conducted to find the effect of pH. Fig. 5 shows the nickel removal with the respect to pH of feed solution. Nickel removal was below 10 and 16% for feed solution pH of 5 and 7, while it was up to 70 and 99% for pH of 9 and 10 respectively. Nickel removal increased with an increase in the pH of the feed solution. At a lower pH, nickel needs to compete with H^+ ions to get the micelle surface. In acidic condition due to the competition with H^+ ions, less nickel was adsorbed on the micelle surface, which leads to a reduction in nickel removal. Flux increased with an increase in the pH feed solution as shown in Fig. 5. Previous researchers have shown that copper removal increased with an increase in acidity in the feed solution [14]. The effect of pH depends on the type of metals used in the solution, whether H^+ ions compete with metal or not during the electrostatic adsorption on micelle.

3.5. Effect of initial concentration of nickel

Another series of experiments were performed with an SDS concentration of 9.44 mM at various initial nickel concentrations in feed solution as shown in Fig. 6. Nickel removal efficiency was 98% for 1 mM concentration. For initial nickel concentrations of 0.31 mM and 0.5 mM, the permeate nickel concentration was less than 1 mg/L, while 1.4 and 2.7 mg/L for nickel concentration of 0.75 and 1 mM, respectively. Nickel concentration in permeate increased with the increase of initial concentration mainly due to less micelle surface area available for electrostatic adsorption of a higher concentration. The average permeate flux remained almost the same for a given surfactant concentration. The charge surface available for nickel on the micelle surface remained constant for a constant initial surfactant concentration, which in turn

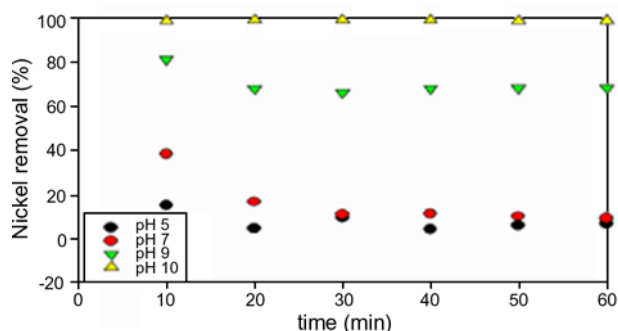


Fig. 5. Effect of pH on nickel removal efficiency (initial nickel concentration = 20 mg/L, initial retentate pressure = 1.4 bar, MWCO of the membrane = 100,000 Da).

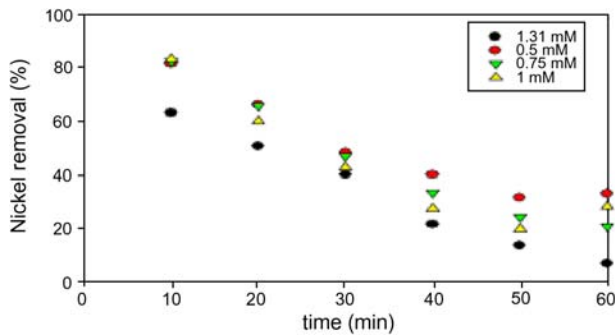


Fig. 6. Effect of initial concentration of nickel (Permeate flux = 1.05m³/m²/day, SDS concentration = 9.44 mM, retentate pressure = 1.4 bar, MWCO of membrane = 100,000 Da).

results in a lower removal of nickel at its higher concentration.

3.6. Effect of MWCO

To identify the effect of membrane pore size, another series of experiments were conducted by varying the pH with UF membranes of MWCO 100,000 and 300,000 Da. The nickel removal efficiency for 100,000 Da MWCO membrane was 26.7, 76.2, 90.3 and 91.7% for the pH 5, 7, 9 and 10, respectively, while the nickel removal efficiency reduced to 19.2, 63.5, 86.4, and 90.4% for the same pH values respectively. Similar results were presented in the previous paper on the removal of anionic pollutant through an MWCO of 3,000 and 10,000 Da [15] (see Fig. 7).

3.7. SDS removal from an ACF filter

The MEUF process can remove nickel to a lower concentration, while the leakage of SDS in the permeate creates secondary pollution. A set of experiments was carried out to study the SDS removal in an ACF

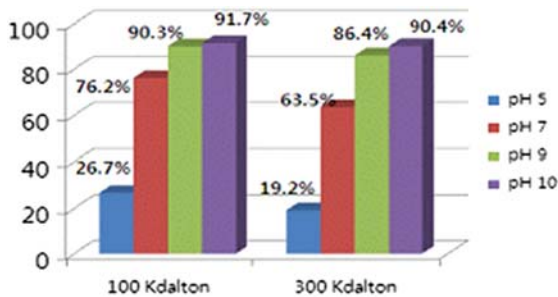


Fig. 7. Effect of MWCO of membrane on the nickel removal efficiency (retentate pressure = 1.4 bar, MWCO of membrane = 100,000/300,000 Da, Permeate flux = 1.05m³/m²/day).

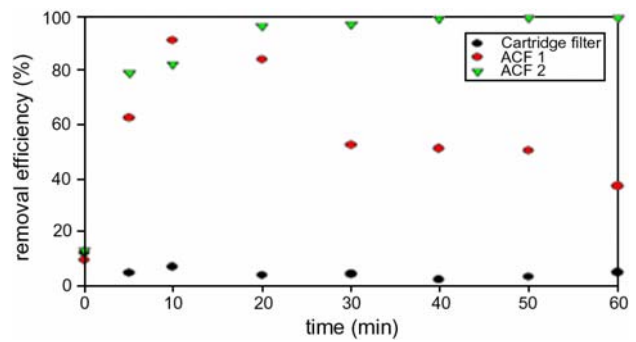


Fig. 8. SDS removal from ACF filter (Nickel:SDS = 1:2, initial nickel concentration = 20 mg/L, retentate pressure = 1.4 bar).

unit. As shown in Fig. 8, SDS removal was 9, 68 and 93% at the CF, ACF1 and ACF2, respectively. Two sets of ACF units in series were able to remove SDS efficiently from wastewater.

3.8. Adsorption isotherm constants for SDS on ACF

The adsorptive capacity of ACF on SDS was identified from a batch experiment. Freundlich and Langmuir isotherm equations were used to calculate the adsorptive capacity. The adsorption constants and adsorption rates are summarized in Table 3. The Adsorptive capacity of ACF on SDS was 170mg/g at an initial SDS concentration of 9.44 mM. The Langmuir isotherm equation gives a better fit compared to the Freundlich isotherm equation. Similar results were figured out in the previous research paper [16].

3.9. SDS removal from the ACF filter

A set of experiments was carried out to study the SDS removal in an ACF unit. As shown in Table 4, SDS removal was 88, 86.2 and 83% at an initial concentration of SDS of 100, 200 and 1,200 mg/L, respec-

Table 3
Adsorption rate of ACF at different equilibrium concentration

Equilibrium conc. (C _e) (mg/L)	Adsorption capacity (mg/g)		
	Experimental	Langmuir equation	Freundlich equation
10.7	170.6	169.3	215.8
124.9	204.6	216.5	219.2
187.2	242.6	218.3	222.2
400.4	217.6	220.4	220.2

Table 4
SDS removal from the ACF at various initial concentration

Average permeate surfactant concentration and removal efficiency			
1,200 mg/L C_e (mg/L)	200 mg/L C_e (mg/L)	100 mg/L C_e (mg/L)	
Removal efficiency (%)	Removal efficiency (%)	Removal efficiency (%)	Removal efficiency (%)
195.3	83.0	86.2	88.0

tively, with the corresponding permeate SDS concentration of 11.4, 26.5 and 195.3 mg/L. Two ACF units in series have removed SDS efficiently from the wastewater.

3.10. Comparison with other heavy metals

The metals are removed by the MEUF process due to the formation of complexes between the lower molecular weight metal pollutants and micelles that result in the rejection of complexes during UF. A comparison between a large and a small-pore sized membrane in the removal efficiencies has found that a large pore-sized membrane had more surfactant permeation than a small pore sized type. Moreover, the mean pore diameter of the 300 kDa membrane was larger than that of the pore diameter of a 100kDa membrane [17]. Therefore, the smaller pore-sized membrane (100 kDa MWCO) could retain more micelles and thus, lead to a better formation of SDS of the micelles and secondary membrane than that of a 300 kDa MWCO [18,19]. Consequently, the 100kDa MWCO membrane could reject more micelles and metal-micelle complexes than that of the 300 kDa MWCO. As a result, the use of 100 kDa MWCO membrane led to higher removal efficiencies in comparison with the 300 kDa MWCO membrane. Similar results were found by previous researchers [14,15].

The MEUF removal efficiency was found to be higher for the removal of heavy metals but it produces a surfactant-rich permeate, which needs to be treated before discharging. As a surfactant in the permeate of MEUF can cause secondary pollution to the environment, the surfactant concentration in the permeate should be considered during the MEUF process [20]. Bade and Lee [14] had reported that almost 98% of copper was removed using SDS but the process can remove only 50% of SDS. The MEUF process followed by the ACF was able to remove 98% of SDS. Similarly, Bade and Lee [21] reported that the MEUF-ACF hybrid process removed 99.5% of the chromate and over 99.5% of CPC. Similar results were also obtained in this experiment. The MEUF-ACF hybrid process in the present study was able to remove 98% of nickel and 85% of SDS. The effectiveness of the MEUF process greatly relies on the surfactant concentration in the feed solution. That is the reason why the nickel removal efficiency was found to be 98.8% at the 1:30 M ratio of nickel to SDS and 99.5, 98.5, 89.6, and 63.4% at the molar ratios of 1:20, 1:10, 1:5 and 1:2.5, respectively. Baek et al. [22] demonstrated that at the molar ratio of 1:5, 98% of the chromate removal was achieved but when the CPC concentration was too high, it decreased due to the increased Cl^- ions [23].

Nevertheless, there is a significant effect of pH on the heavy metal removal in the MEUF process. The nickel removal was below 10% and 16% for the feed solution pH of 5 and 7, respectively, while it was up to 70 and 99% for the pH of 9 and 10, respectively. Juang et al. [24] reported that cationic heavy metal (Mn^{2+} , Co^{2+} , Cu^{2+} , Zn^{2+} and Cr^{3+}) removal reached over 80% with SDS as the pH increased from 2 to 12. Similarly, cadmium rejection increased from 83 to 99% when the pH increased from 3 to 11 in the feed solution containing cadmium (100 mg/L) and SDS (8 mM) [25]. Moreover, the rejection percentage of the chromate increased from 95.4 to 99.7% when the pH of the feed solution changed from 4 to 5.5 [26].

4. Conclusions

The MEUF-ACF hybrid process was very effective and efficient in the removal of nickel from an aqueous solution at various initial retentate pressures, initial permeate flux, pH values and molar ratios of SDS to nickel. The average nickel removal efficiency at the optimum operating condition was found to be 98% and the corresponding permeate nickel concentration was found to be less than 1 mg/L. Based on the removal efficiency and permeate flux, initial permeate flux of $1.05 \text{ m}^3/\text{m}^2/\text{day}$, nickel to SDS molar ratio of 1:10 (9.44 mM SDS) and retentate pressure of 1.4 bar were found to be the optimum operating parameters. The adsorptive capacity of ACF for SDS was 170 mg/g and the overall SDS removal efficiency of the ACF units was found to be 85%. The Langmuir isotherm equation gives a better fit with over the Freundlich isotherm one.

Acknowledgements

This work was supported by the Korea Research Foundation Grant funded by the Korea Government (2011-218-011). The authors are also grateful for the provision of UF membrane unit from Chemicore Inc., Korea.

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