

Application of micellar enhanced ultrafiltration (MEUF) and activated carbon fiber (ACF) hybrid processes for the removal of nickel from an aqueous solution

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Abstract- Micellar enhanced ultrafiltration (MEUF) was used to remove nickel from an aqueous solution using sodium dodecyl sulfate (SDS) as a surfactant in this study. 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 were investigated. Removal efficiency of nickel from aqueous solutions increased with the increase of retentate pressure, pH and molar ratio of nickel to SDS but decreased with increase of initial permeate flux. Higher removal efficiency of nickel from the aqueous solution was achieved using lower MWCO (smaller membrane pore size). At optimized experimental condition, nickel removal efficiency was 71.8% within an hour in MEUF process. Using MEUF-ACF hybrid (micellar enhanced ultrafiltration-MEUF combined with activated carbon fiber-ACF) process the removal efficiencies of nickel and SDS were found to be 96.2% and 82.2% respectively. The overall SDS removal efficiency of two sets of ACF unit was 89.6%.

Keywords – Micellar enhanced ultrafiltration, Activated carbon fiber, Nickel, Sodium dodecyl sulfate, heavy metal removal

I. INTRODUCTION

Heavy metals are amongst the most dangerous anthropogenic environmental pollutants, due to their toxicity, bioaccumulation, persistence in the environment, biomagnification in the food chain [1]. Industrial wastewater containing these metals 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]. Nowadays nickel (Ni) is widely used in numerous industries. The potential human health impacts of nickel are: carcinogen, developmental toxicant, immunotoxicant, kidney toxicant, neurotoxicant, reproductive toxicant, respiratory toxicant, skin or sense organ toxicant [3]. At present, popular techniques for treating nickel-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 nickel 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 RO (reverse osmosis) and the high flux of UF (surfactant based ultrafiltration) [5]. In the 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, nickel ions adsorbed onto the micelles are rejected and the permeation quality is adequate for reuse or direct discharge.

Addition of surfactant to the MEUF system results in release of some of the surfactant in the permeate. One of the major drawbacks of the MEUF process is thus 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 (PAC), granular activated

carbon (GAC) and activated carbon fiber (ACF). Among these, ACF has a uniform micro-pore structure, faster adsorption kinetics 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 nickel from wastewater using SDS, and to investigate the performance of the MEUF-ACF hybrid process for SDS removal, as well as for the removal of trace nickel from the MEUF effluent..

II. MATERIALS AND METHODS

In this study, nickel (II) sulfate hexa-hydrate ($\text{NiO}_4\text{S}_6\text{H}_2\text{O}$) of 99 % purity was procured from Sigma-Aldrich Co., USA (molecular weight of 262.85) and sodium dodecyl sulfate (SDS) of 99 % purity was procured from Acros Organic, Ltd., USA (molecular weight of 288.38). These were respectively used as the source of nickel 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.

Table - 1 Details of MEUF experimental operating conditions

Retentate pressure, MPa	0.2, 0.24, 0.28
pH	5, 7, 8.6, 10
Initial nickel concentration, mM	0.0761, 0.1902, 0.2853, 0.3804
Molar ratio of nickel to SDS	1:2, 1:5, 1:10, 1:15
Sampling time, min	5, 10, 20, 30, 40, 50, 60
Initial flux (distilled water), L/m ² h	21.8, 32.7, 43.6, 54.5

All solutions were prepared using distilled water. Solutions were prepared by mixing stoichiometric amounts of SDS surfactant and nickel in eight liters of distilled water for an hour with 100 rpm. Hollow fiber membrane (Chemicore Ltd., Korea), having two kinds of MWCO (molecular weight cut-off) 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.

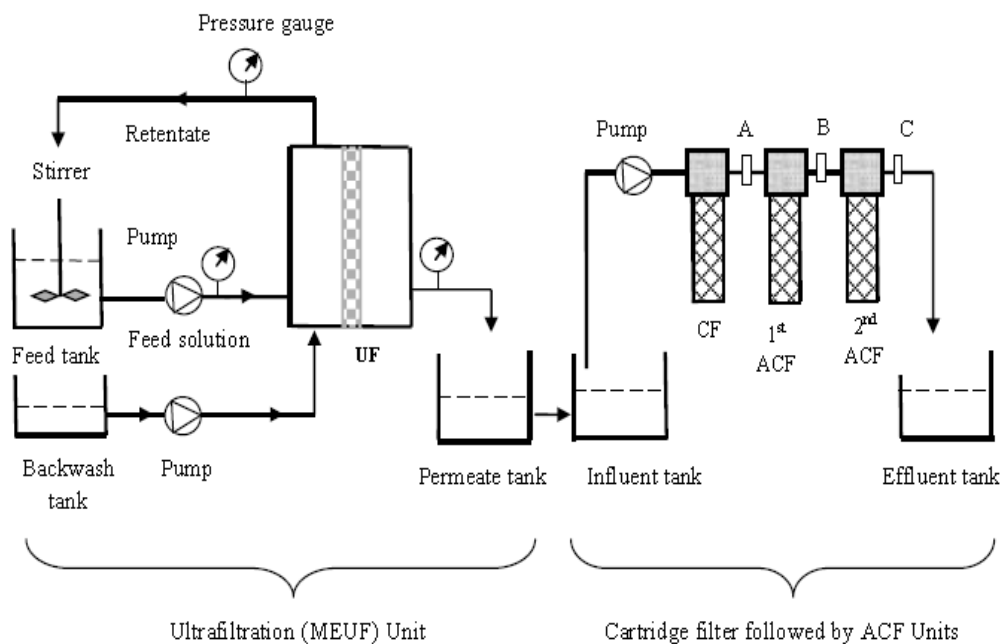


Figure 1. Experimental set-up for MEUF-ACF hybrid processes

The experimental module consisted of a feed tank, ultrafiltration membrane, wash out tank and permeate tank as shown in Figure 1. The ACF unit comprised a cartridge filter (CF) connected with a feed tank to prolong the life span of the ACF. 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 kg/m³ and 1,500 mg/g respectively. After each series of experiments, UF membrane was flushed and backwashed with the distilled water and cleaned

with 0.1M NaOH and 0.5 % HCl. CF and ACF units were cleaned with distilled water before soaking in 0.1M of NaOH and 2 % of HCl for a day. Nickel concentration was measured using Inductively Coupled Plasma (ICP, Varian OES-720) with a wavelength of 231.604 nm. Samples of MEUF were pretreated according to standard methods for the examination of water and wastewater [11]. SDS was measured using chemical oxygen demand (COD) as per Standard Methods. Nickel and SDS removal efficiencies were calculated using Eq. 1.

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

Where, R = rejection (%); C_p = permeate concentration (mg/L); C_i = influent concentration (mg/L).

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 ²	0.055
Membrane diameter (inside/outside), mm	0.8/1.4
Molecular weight cut-off (MWCO)	100000, 300000
ACF BET surface area, m ² /g	1000
Weight of ACF, g/cartridge	30

III. RESULTS AND DISCUSSION

3.1. Effect of permeate flux on nickel removal –

A series of experiments were conducted using in various permeate fluxes. Figure 2 shows the removal characteristics of nickel at various permeate fluxes. Average nickel removal efficiency was 65.0 % with permeate flux of 21.8 L/m².h, while it was 62.1 %, 55.7 % and 50.0 % with the initial permeate fluxes of 32.7 L/m².h, 43.6 L/m².h and 54.5 L/m².h respectively. This implied that nickel removal efficiency decreased with an increase of permeate flux within the operational experimental range. In surfactant based ultrafiltration (UF) process, an increase in permeate flux led to an increase in concentration polarization (CP) on the membrane surface [12]. Flux decline is the main bottle-neck of this process. This is mainly caused by concentration polarization, fouling and adsorption [13]. Considering the higher removal efficiency of nickel (65.0 %) achieved at this value, 21.8 L/m².h was found to be the optimum initial permeate flux in this study.

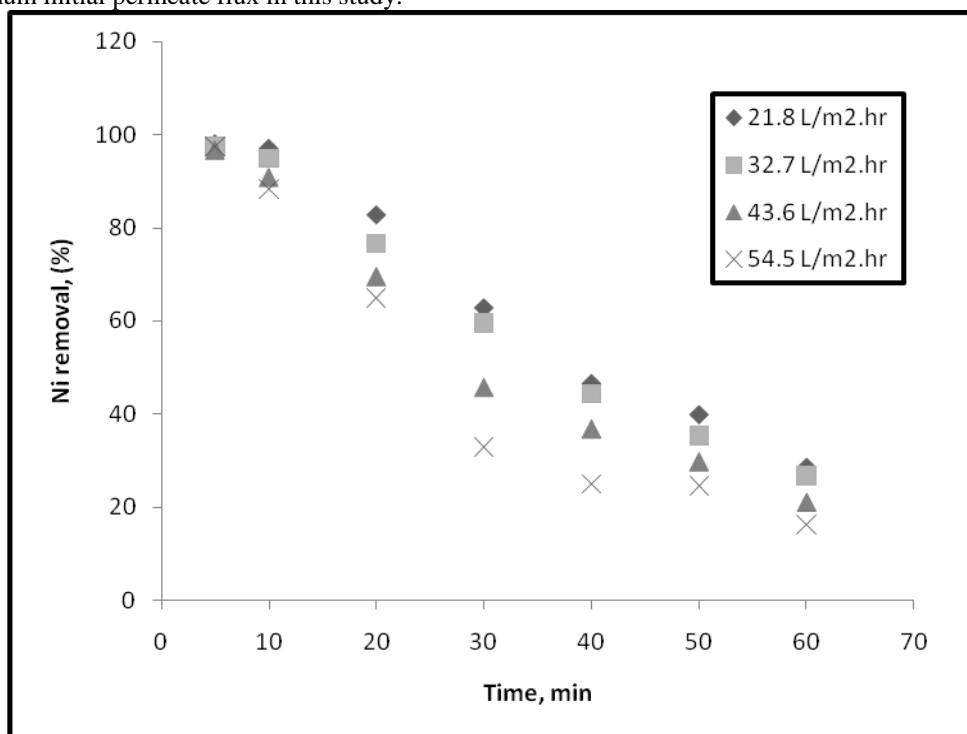


Figure 2. Effect of permeate flux on nickel removal efficiency (Retentate pressure = 0.2 MPa, initial concentration of nickel salt = 0.0761 mM, molar ratio of nickel to SDS = 1:5, MWCO of membrane = 100,000 Dalton)

3.2. Effect of retentate pressure on nickel removal –

The effect of initial retentate pressure on nickel removal was investigated under various initial retentate pressures. As shown in Figure 3, average nickel removal was 65.0 % with initial retentate pressure of 0.2 MPa, whereas it was 68.6 % and 68.8 % at the initial pressures of 0.24 MPa and 0.28 MPa respectively. Nickel removal increased with an increase in initial retentate pressure similar to that obtained in previous studies [12]. With an increase in retentate pressure, transmembrane pressure (TMP) also increased. The operation of membrane process at low transmembrane pressure is an important issue in terms of minimizing operating costs. An increase in pressure actually increases the gel layer thickness, in turn increasing rejection of the metal-micelle complex. For these reasons, the optimum retentate pressure was found to be 0.2 MPa.

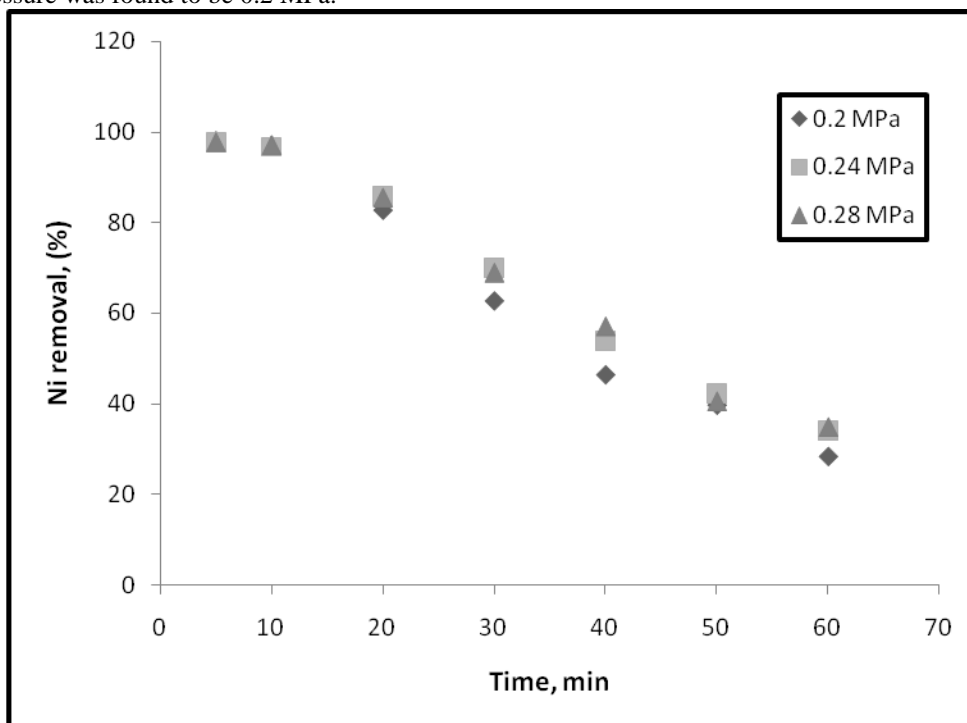


Figure 3. Effect of retentate pressure on nickel removal efficiency (Permeate flux = 21.8 L/m².hr, initial concentration of nickel salt = 0.0761 mM, molar ratio of nickel to SDS = 1:5, MWCO of membrane = 100,000 Dalton)

3.3. Effect of molar ratio of nickel to SDS –

To find the effect of molar ratio of nickel to SDS, another series of experiments were conducted at different molar ratios of nickel to SDS. Figure 4 shows that average nickel removal was 71.8 % for a molar ratio of 1:10. Removal efficiency decreased to 68.4 % and 64.8 %, at molar ratios of 1:5 and 1:2 respectively. Nickel removal increased with increase in molar ratio. Nickel removal efficiency was higher with higher initial SDS concentration that produced more micelles, making more micelle surface area available for electrostatic attraction of nickel ions. Nickel ions were retained on the membrane surface along with the micelles [2]. After surfactant concentration reaches critical micelle concentration (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 these reach CMC, and monomers pass through the membrane together with pollutant [14, 21, 22]. This results in a large micelle surface area being available for electrostatic attraction of nickel ions. As a result, a higher quantity of nickel is removed together with the micelles.

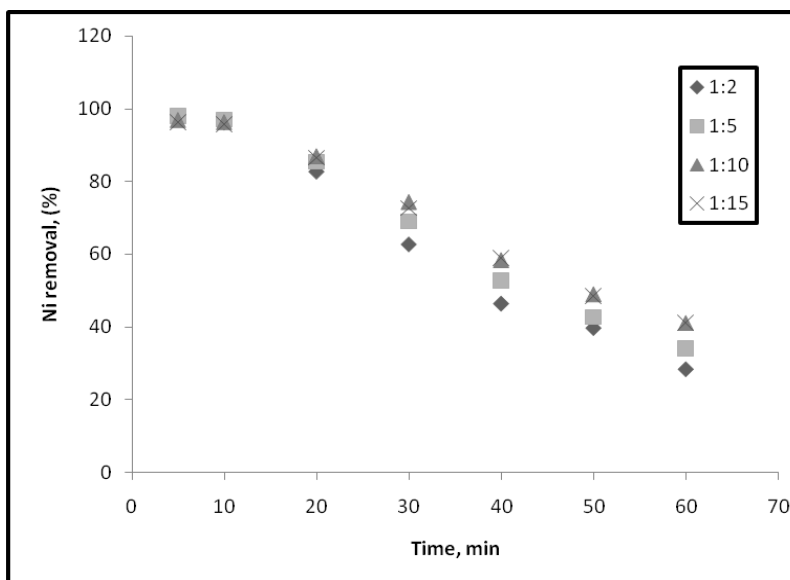


Figure 4. Effect of molar ratio of nickel to SDS on nickel removal efficiency (Permeate flux = 21.8 L/m².hr, retentate pressure = 0.2 MPa, initial concentration of nickel salt = 0.0761 mM, MWCO of membrane = 100,000 Dalton)

3.4. Effect of pH –

A series of experiments were performed to investigate the effect of pH on nickel removal. As shown in Figure 5, average nickel removal efficiency was 60.1 % and 76.2 % for feed solution pH values of 5 and 7 respectively, while it was 83.0 % and 97.1 % for pH values of 8.6 and 10 respectively. Nickel removal increased with an increase in the pH of the feed solution. At lower pH, nickel ion needs to compete with H⁺ ions for the micelle surface. Under acidic conditions, due to competition with H⁺ ions, less nickel was adsorbed onto the micelle surface leading to a reduction in nickel removal. On the contrary, at higher pH, H⁺ bound with functional groups can be dissociated easily, and the deprotonated functional groups can bind with nickel ions [15]. Previous research has also shown that copper removal increased with the increased acidity of the feed solution [16, 21, 22]. The effect of pH depends on the type of metals used in the solution, and specifically on whether H⁺ ions compete with the metal during electro-static adsorption on micelles.

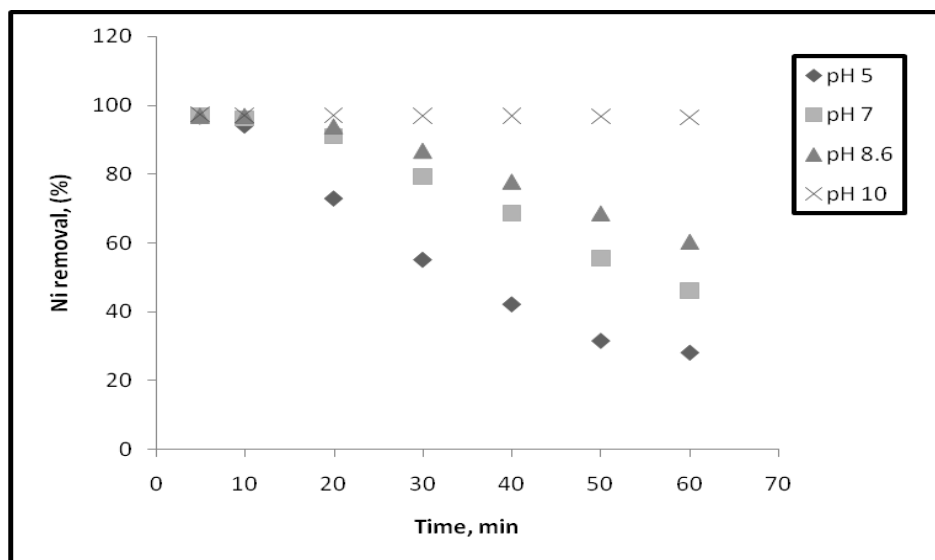


Figure 5. Effect of pH on nickel removal efficiency (Permeate flux = 21.8 L/m².hr, retentate pressure = 0.2 MPa, initial concentration of nickel salt = 0.0761 mM, molar ratio of nickel to SDS = 1:5, MWCO of membrane = 100,000 Dalton)

3.5. Effect of initial concentration of nickel –

Another series of experiments were conducted with SDS concentration of 0.38 mM at various initial nickel concentrations in feed solution as shown in Figure 6. Average nickel removal efficiency was 65.0 % for 0.0761 mM concentration of nickel. For initial nickel concentration of 0.1902 mM, 0.2853mM and 0.3804 mM, average nickel removals were 52.3 %, 45.6 % and 39.2 % respectively. nickel 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 [14, 21, 22]. Average permeate flux remained almost the same for the given surfactant concentration, and the charge surface available for nickel on the micelle surface remained constant for constant initial surfactant concentration. This resulted in lower removal of nickel at its higher concentration in feed solution [17].

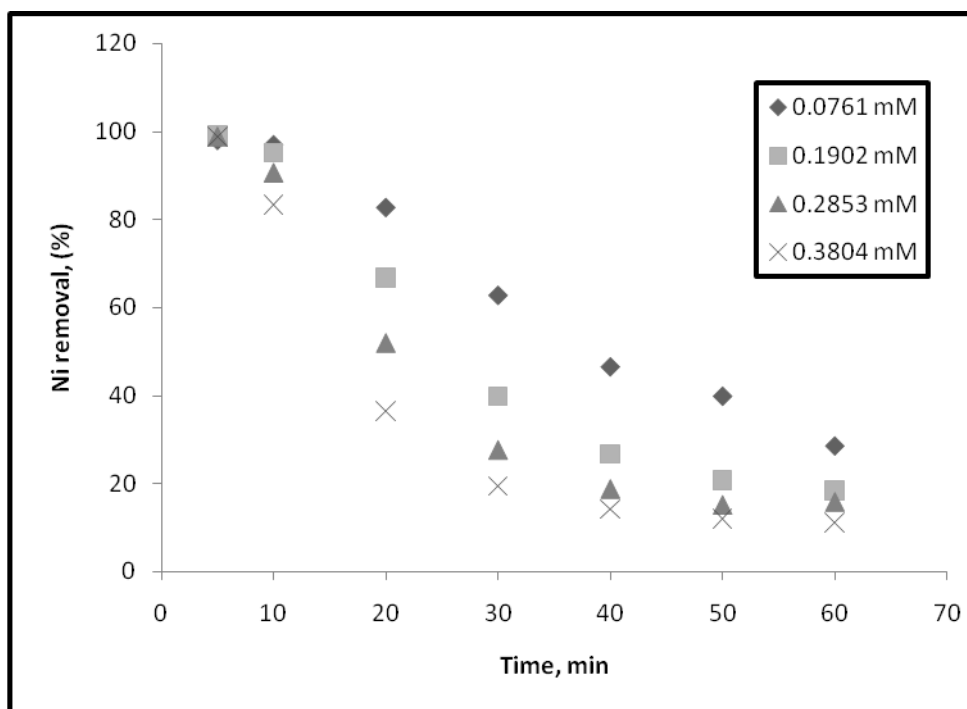


Figure 6. Effect of initial concentration of nickel on nickel removal efficiency (Permeate flux = 21.8 L/m².hr, retentate pressure = 0.2 MPa, SDS concentration = 0.38 mM, MWCO of membrane = 100,000 Dalton)

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 (nickel to SDS) using ultrafiltration membranes of MWCO 100,000 and 300,000 Dalton. Nickel removal efficiencies using 100,000 Dalton MWCO membrane were found to be 60.1 %, 76.2 %, 83.0 % and 97.1 %, with pH values of 5, 7, 8.6 and 10 respectively. Similarly, nickel removal efficiencies using 300,000 Dalton MWCO membrane were 58.9 %, 68.2 %, 72.3 % and 97.0 % for the same pH values respectively (Figure 7a). Previous research has also shown similar trend [17]. On the other hand with different molar ratios (Nickel to SDS) of 1:2, 1:5, 1:10 and 1:15, average nickel removal efficiencies using 100,000 Dalton MWCO membrane were found to be 64.8 %, 68.4 %, 71.8 % and 71.4 % respectively, while with the same molar ratios, removal efficiencies using the 300,000 Dalton MWCO membrane reduced to 39.2 %, 41.1 %, 41.3 % and 41.2 % respectively (Figure 7b). Similar results were obtained in previous studies on the removal of anionic pollutants through MWCO of 100,000 and 300,000 Daltons [18].

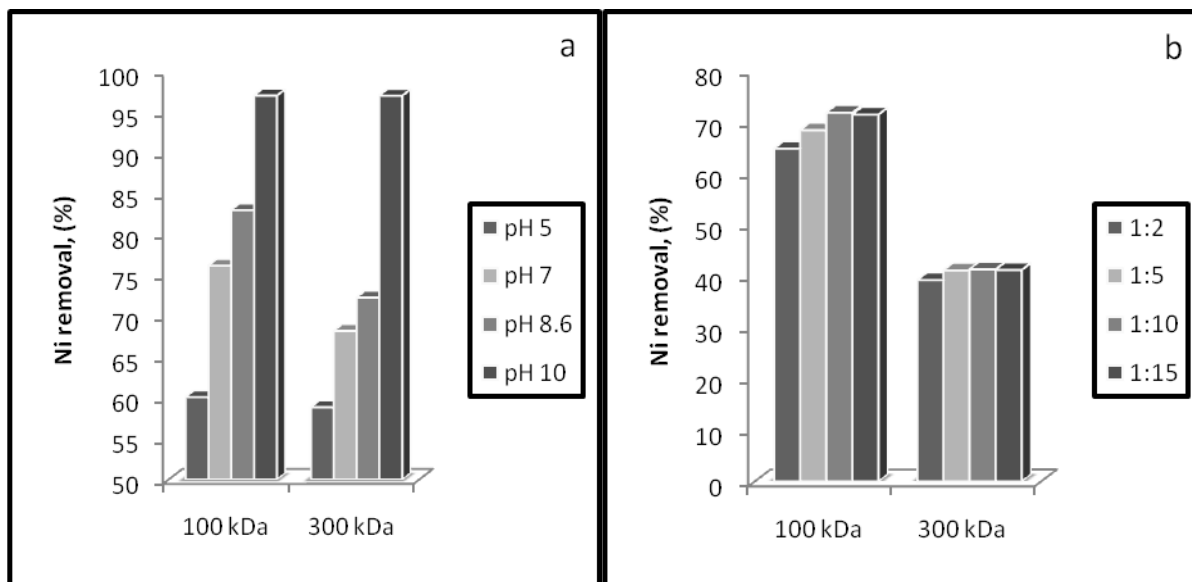


Figure 7. Effect of MWCO of membrane on nickel removal efficiency (a) in various pH (Permeate flux = 21.8 L/m².hr, retentate pressure = 0.2 MPa, MWCO of membrane = 100,000 & 300,000 Dalton) and (b) in various molar ratios of nickel to SDS (Permeate flux = 21.8 L/m².hr, retentate pressure = 0.2 MPa, MWCO of membrane = 100,000 & 300,000 Dalton)

3.7. Removal of nickel by MEUF without SDS –

An experiment was conducted without surfactant (SDS), in feed solution containing initial nickel concentration of 0.0761 mM (20 mg/L). As shown in Table - 3, nickel removal percentage was 30.5 % during the 60 minutes of operation. This strongly indicated inefficiency of UF alone in removing of nickel 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 [19].

Table - 3 Nickel removal without using SDS

Time (min)	Permeate conc. (ppm)	Removal efficiency (%)	Flux rate L/m ² .hr
30	0.98	72.2	21.8
40	1.54	56.5	21.8
50	2.08	41.2	21.8
60	2.46	30.5	21.8

(Initial nickel concentration = 0.0761 mM, initial retentate pressure = 0.2 MPa, initial permeate flux = 21.8 L/m².hr, MWCO of the membrane = 100,000 Da)

3.8. Performance of MEUF-ACF hybrid process –

A series of experiments were conducted to investigate the removal of excess nickel ions present in the MEUF permeate by coupling with activated carbon fiber (ACF). Further, comparative analysis was carried out regarding the average nickel removal percentage from various units of MEUF-ACF hybrid process such as MEUF, cartridge filter (CF), and two ACF units (ACF1 and ACF2), at constant initial SDS concentration and initial nickel concentration of 0.38 mM (100 mg/L) and 0.0761 mM (20 mg/L), respectively.

As shown in Table - 4, the 100,000 Da UF membrane has shown an average nickel removal percentage of 68.4 % for initial nickel concentration of 0.0761 mM during 1 hr operational time. Average nickel removal percentage reached 96.2 % when coupled with ACF units. On the other hand, the 300,000 Da UF membrane produced an average nickel removal percentage of 41.1 %, and the nickel removal percentage reached 95.1 % after being combined with the ACF units. Furthermore, average nickel removal percentages from the MEUF, CF and two ACF units were higher when using 100,000 Da UF membrane than when using 300,000 Da UF membrane.

Table - 4 Comparative nickel removal percentage from various units of MEUF-ACF hybrid process

100,000 Da				300,000 Da			
MEUF	CF	ACF1	ACF2	MEUF	CF	ACF1	ACF2
68.4	70.3	83.6	96.2	41.1	44.7	68.5	95.1

(Molar ratio of nickel to SDS = 1:5, initial retentate pressure = 0.2 MPa, initial permeate flux = 21.8 L/m².hr, pH = neutral, operation time = 5 hr, MWCO = 100,000 Da and 300,000 Da)

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, cartridge filter (CF), and two ACF units (ACF1 and ACF2), at constant initial SDS concentration, and initial nickel concentrations of 0.38 mM (100 mg/L) and 0.0761 mM (20 mg/L), respectively.

As shown in Table - 5, SDS removal percentages were 32.2 % and 23.8 % for 100,000 Da and 300,000 Da UF membranes respectively. After coupling with the ACF units, SDS removal percentages reached to 82.2 % and 80.1 % for 100,000 Da 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 Da and 300,000 Da membranes. As seen in Table 4, 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 concentration polarization (CP) and reduced the release of surfactant in the permeate [20].

Table - 5 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
32.2	40.7	68.5	82.2	23.8	38.3	62.6	80.1

(Molar ratio of nickel to SDS = 1:5, initial retentate pressure = 0.2 MPa, initial permeate flux = 21.8 L/m².hr, pH = neutral, operation time = 5 hr, MWCO = 100,000 Da and 300,000 Da)

3.9. SDS removal by ACF filter –

MEUF process can remove SDS only to a lower concentration, while the leakage of SDS in the permeate creates 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 mg/L, 200 mg/L, 600 mg/L 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	
Ce (mg/L)	Removal (%)	Ce (mg/L)	Removal (%)	Ce (mg/L)	Removal (%)	Ce (mg/L)	Removal (%)
194.9	83.8	93.8	84.4	26.7	86.7	10.4	89.6

IV. CONCLUSION

In the MEUF process, the average nickel removal efficiency under optimum operating conditions was 71.8 % at neutral pH. Furthermore, the 100,000 Dalton MWCO membrane was better choice for higher nickel removal efficiency over 300,000 Dalton MWCO membrane with a comparative studies of various molar ratios of nickel to SDS and various pHs. During 60 minutes of ultrafiltration without SDS, the nickel removal percentage was 30.5 %. In the MEUF-ACF hybrid process, removal efficiencies of nickel and SDS at the end effluent were 96.2 % and 82.2 % with 100,000 Dalton membrane whereas 95.1 % and 80.1 % with 300,000 Dalton membrane, respectively. SDS removal efficiency of the ACF unit was 89.6%.

V. ACKNOWLEDGEMENTS

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