

Modelling of selective retention of Cd-Ni ions from aqueous solutions by polymer enhanced ultrafiltration

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Abstract: Polymer enhanced ultrafiltration (PEUF) technique economically enables separation and/or retention of various heavy metal ions from aqueous solutions for environmental and engineering problems. Polyethylenimine as the water-soluble complexation agent utilized to interact with heavy metal ions of Cd and Ni as cations in ultrafiltration experiments were modeled with protonation data to understand the mechanism. Test runs showed that an enrichment coefficient (the ratio of Cd over Ni in permeate) up to 10 was obtained at pH 4.2 in the temperature range of 20-22°C, whereas full retention of both cations is possible above pH 5.0 and those results are highly compatible with the model developed.

Keywords: cadmium, nickel, polymer enhanced ultrafiltration, polyethylenimine (PEI), selective heavy metal ion retention

1. Introduction

Wastewaters originating from metal industries such as mining, mineral processing, metal finishing, and battery production plants contain heavy metals that are toxic and hazardous to the environment (Hestekin, et al., 1998; Qasem et al., 2021). Any industrial water coming out of a facility or wasteland mixes into ecosystem where it threatens all living creatures' health and develops environmental risks. For this, the environmental restrictions about the heavy metal ion concentrations are becoming stringent than ever and the processes configured on the basis of earlier regulations are not in compliance with the new limits (Canizares et al., 2002).

The membrane techniques are often combined with other physical and chemical processes to improve their effectiveness or operation. These include hybrid processes: precipitation of sparingly soluble metal compounds and microfiltration or ultrafiltration, adsorption, and ultrafiltration, bioaccumulation, and microfiltration, complexation, and microfiltration or ultrafiltration (Scott and Hughes, 1996). The last process involves linking of metal ions with a high-molecular ligand to form a complex with a size larger than the pore of microfiltration or ultrafiltration membrane (Crini et. al., 2017). Because binding metal ions with polymer ligands is a reversible process, it is possible to regenerate the retentate in order to recover the metal and complexation agents. Industrially commercialized membrane system that fits the required separation size somehow correspond to Ultrafiltration (UF), where it requires low transmembrane operating pressure (TMP) compared to any other filtration system that could hold heavy metal ions (Abdullah et al., 2019). As UF membrane pores are much larger than those ions, additives are utilized for an effective method called polymer-enhanced ultrafiltration (PEUF), in which metal ions are selectively bond from aqueous solutions using water-soluble polymers. The pioneering work on this subject was reported by Geckeler et al. in the early 1980s; in order to achieve selective metal ion complexation with commercially available water-soluble polymers such as polyethyleneimine was recognized. Therefore, interest in designing more efficient and selective polymers that can attach more ions, in order to minimize the number of expensive polymers used for this purpose.

Briefly, the PEUF system is a combination of two phenomena namely binding of metal ions to a water-soluble polymer and ultrafiltration (Canizares et al., 2002). Binding a target metal ion to a polymer backbone advances the ion to the form of macromolecular complexes. The key advantages of the ultrafiltration method combined with complexation in water purification process are as follows: highly selective polyligands, high productivity by the use of selective polyligands, high productivity at insignificant power consumption (Rumeau et al., 1992), high flux and less space demand (Fu, F. and Wang, Q., 2011). Further, the process can be carried out under the continuous mode of operation that provides means to automate. In majority of the previous PEUF studies, the main purpose was selection and synthesis of various water-soluble polymeric complexation agents for targeted metals and study their binding characteristics using batch or semi-batch ultrafiltration systems.

Batch or semi-batch ultrafiltration systems were used to investigate the complexation of a single ion with a given polymer (Rumeau, et al., 1992). The use of polyacrylic acid as a complexation agent in a solution containing copper and nickel (Masse, et.al., 1987), and that of partially ethoxylated polyethyleneimine was demonstrated with Cu^{2+} , Ni^{2+} , Cd^{2+} , and Zn^{2+} solutions (Llannos et al., 2010). In both of the studies, the pH had a crucial role in the bulk and selective recovery of heavy metals. Also the study of Jarvis et. al. (1995) showed that the protonation of polymer and species distribution of heavy metal ions at particular pH plays a major role in the success. The bonding polymers have several types of functional groups (sulfonate, carboxylated, amine and phosphonic) and they function via chelating or ionic bonds (Rivas et al., 2009).

There is a detailed list of researchers utilized polymers for ultrafiltration purposes prepared by Crini et al. (2014) and accordingly polyethylenimine used for removal of various heavy metals with different combinations, Cd (Li et al. (2008), Cu and Ni (Molinari et al., 2006 and 2008), Ni (Shao et al., 2013), Co (Cojocararu et al., 2009), Hg (Uludag et al., 1997), Cu, Ni, Cd and Pb (Canizares et al., 2002). The main challenge of developing this technology is choosing appropriate water-soluble polymer macro-ligands and lowering the cost. Even there are more abundant and cheaper biopolymers are available, a conservative research is designed to explain the window of separation for mentioned metal ions. Due to proven efficiency towards different metal ions by the literature, well defined structure and protonation behaviour, PEI was utilized for the research.

In this study, firstly the effects of operational parameters were investigated by utilizing a statistical approach to determine the full retention of cadmium and nickel ions from solutions by polymer-enhanced ultrafiltration (PEUF). Further, the possibilities of selective separation of cadmium and nickel studied in order to find an optimum window for selective separation of these metal ions.

2. Materials and methods

The membrane used in this study was the Osmonics flat sheet (DS-GK) type with molecular weight cut off (MWCO) of 3000 Da and the polyethyleneimine used to bind heavy metal ions has an average molecular weight of 50000 Da (Sigma-Aldrich). Cadmium nitrate tetrahydrate ($\text{Cd}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$) and nickel chloride ($\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$) were used in the preparation of solutions at 100 ppm. Experiments were carried out at a constant flow rate of 250 L/h and pressure of 6 bar in order to study the effect of loading ratio, temperature, and solution pH on retention rates of Cd and Ni ions. Primarily, the loading ratio, pH and temperature values were adjusted to investigate the PEI binding behavior on both metals. In preliminary experiments the temperature values ranged between 12-38°C, pH 4.5-5.5-6.5, loading ratio 0.25-0.5-1 (Can et al., 2006). And those results with the help of models calculated, revealed the pH range of 4 to 6 for possible separation between metal ions.

Schematically polymer-enhanced ultrafiltration (PEUF) process is shown in Fig. 1. Solution samples taken from the tank at the beginning and end of each experiment and from the permeate flow at certain time intervals were analyzed by Atomic Absorption Spectrophotometer (AAS). Mathematica software is utilized for modeling the protonation and complexation of polymer ligand as described by Jarvis, et al., (1994). In details, Mathematica 10.0 software is capable of plotting 3D graphics with its "Plot3D" function for Fig. 2, 3, 4, and 8. And the mathematical equations below the graphics are calculated with the "NonlinearModelFit" function. For Fig. 5 and 6, stoichiometric equations have been set as function with total giving the species Ratio as 1 and with the help of "Solve" function of the software each species

distribution through the pH is calculated plotted with "Plot" function. For Fig. 7 experimental data from Jarvis et al., had been extracted from the article with "xyExtract" software and prepared a list of dots containing (x,y) and by utilizing "ListPlot" function of Mathematica 10.0 the graphics are formed. For modeled line the functions listed as 2, 3 and 4 solved with the data presented at Fig. 5 and 6 with the "Solve" function of the software, the sum of results are matched with experimental results with the same "ListPlot" function for presentation.

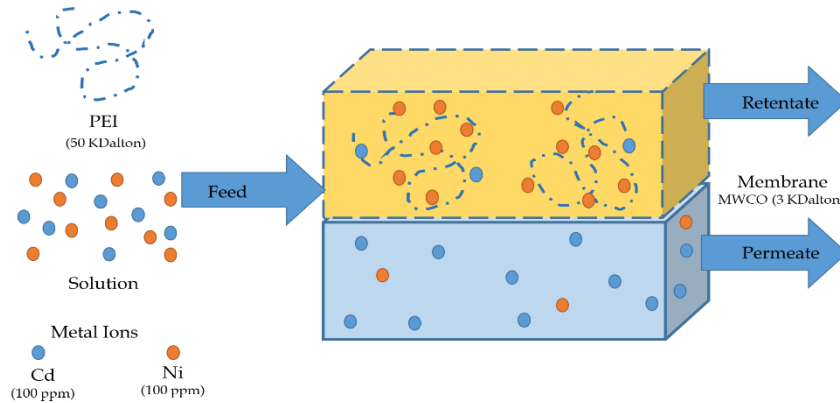
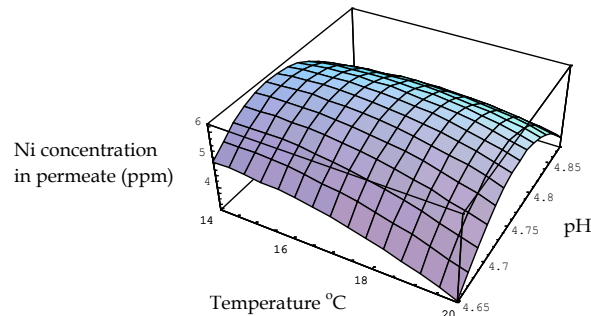


Fig. 1. Schematic representation of polymer-enhanced ultrafiltration (PEUF) (Sebate, 2016)

3. Results and discussion

The experimental results of Cd and Ni separation by polymer-enhanced ultrafiltration (PEUF) is presented in detail in our earlier article published in 2006 (Can et al., 2006), therefore only the experimental results used in the modelling will be given here. The effects of pH and temperature on flux rate and metal concentration were investigated. At higher pH ranges retention of both metal ions are possible, only at lower pH values between 4.5 to 6.0 preliminary experiments revealed a separation window occurs. Another point is about solution temperature; at higher temperatures over the flux rates increase but the rejection of metal ions decrease and PEUF loose efficiency. From those results, here the separation experiments run in the conditions of pH between 4.65 to 4.85 and temperature 14 to 22OC are chosen for the modelling study. Although there are similarities in the retention characteristics of both metal ions with pH and temperature, the concentration of cadmium ions in permeate is found much greater than nickel ions as presented in Fig. 2 and 3.

According to former studies with PEI, Müslehiddionğlu et al. (1998), utilized a membrane with 10 KDa MWCO. When compared with current results, 3 KDa cut off value membrane acquired higher retention rates with less PEI consumption. Uludağ et al. (1997) noted that with smaller membrane cut-off values, even a partial retention of heavy metals in the absence of polymer is possible. Therefore smaller pore diameter of membrane can improve the quality of permeate while decreasing quantity.



$$- 2842.34 - 4.61319 x - 0.0432914 x^2 + 1214.97 y + 1.25188 x y - 129.998 y^2$$

Fig. 2. Variation of nickel concentration in permeate with solution pH and temperature for nickel and cadmium mixture. pH represented by x and temperature represented by y in the model equation for retentate Ni concentration

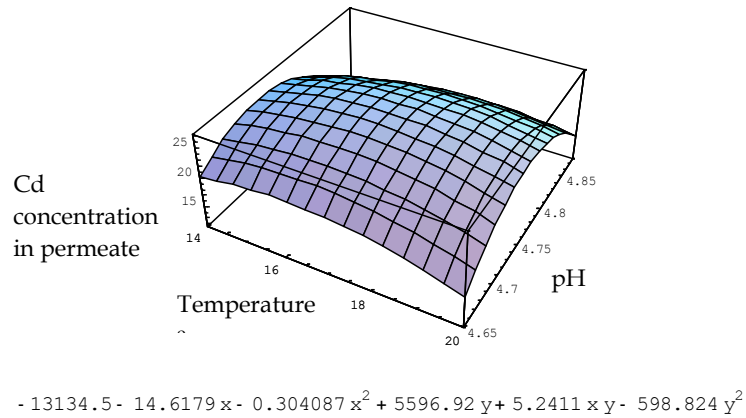


Fig. 3. Variation of cadmium concentration in permeate with solution pH and temperature for nickel and cadmium mixture. pH represented by x and temperature represented by y in the model equation for retentate Cd concentration

It is possible to remove the metal ions completely or to separate them from each other by adjusting the set of parameters. For separation, a model enrichment coefficient was required for the system. In order to evaluate the effectiveness of separation, an enrichment coefficient representing the proportion of cadmium and nickel ions was used. Enrichment coefficient is defined as;

$$X_E = \frac{C_{Cd}}{C_{Ni}} \quad (1)$$

where X_E = enrichment coefficient, C_{Cd} = cadmium concentration in permeate, C_{Ni} = nickel concentration in permeate.

The ion concentrations in permeate were constructed in the form of a 3D graphic to illustrate the variation of X_E with temperature and pH (Fig. 4). Evidently, at pH value of 4.2 and temperature values at 20-22°C, the enrichment factor (X_E) rises up to 10, indicating that cadmium ion concentration in the permeate is ten times greater than nickel ions.

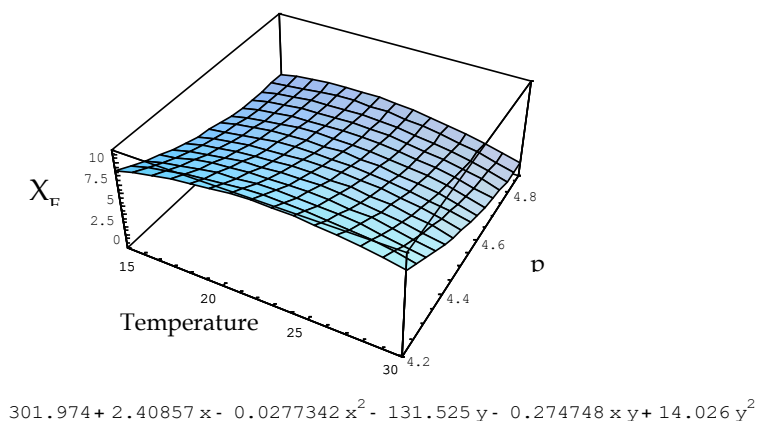


Fig. 4. Variation of enrichment coefficient (X_E) of the permeate with solution pH and temperature for the separation of Ni+Cd mixture. Temperature represented by x and pH represented by y in the model equation for enrichment coefficient (X_E)

As a result of this experimental study on retention of cadmium and nickel ions from solutions by polymer-enhanced ultrafiltration (PEUF), test runs showed that an enrichment coefficient (the ratio of Cd over Ni in permeate) up to 10 was obtained at pH 4.2 in the temperature range of 20-22°C, whereas full retention of both cations is possible above pH 5.0. Mathematica software was used for modeling the protonation and complexation of polymer ligand.

From the study of binding mechanisms of PEI to heavy metals made by N.V. Jarvis and J. M. Wagner (1994), apparent protonation and formation constants were taken in order to develop equilibrium models in Mathematica 10.0. Firstly, the equilibrium formation and constants of PEI were taken into

account as listed in Table 1 and developed a model for species distribution curves for protonation of PEI as shown in Fig 5. In the second step for nickel and cadmium, the same procedure was followed.

Table 1. Apparent protonation and formation constants for PEI (H=proton, L=Monomeric unit) (Jarvis & Wagner, 1994)

Equilibrium	Log K
$H+L=HL$	9.71
$HL+H=H_2L$	7.70
$H_2L+H=H_3L$	2.64
$2L+3H=H_3L_2$	30.87
$2L+5H=H_5L_2$	41.58

According to Fig. 5, Jarvis et al. (1995) explained the graphic as it is related to nearest neighbour interaction mechanism where intramolecular hydrogen bonding occurs, the species with two repeating units acquired. As you may follow the protonation from low pH to high pH, the ligand is stripped of H ions. When going from H_3L to H_2L or from H_2L to HL , the second repeating unit becomes deprotonated and form intermediating species of H_5L_2 and H_3L_2 .

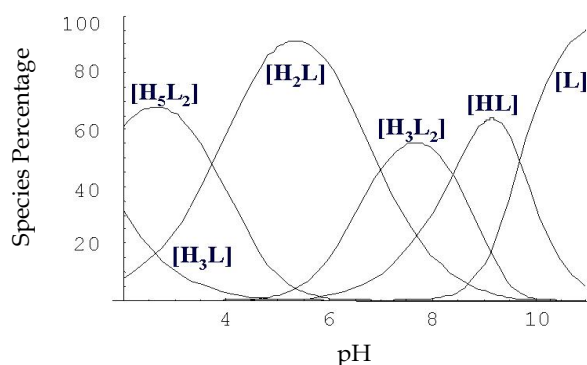


Fig. 5. Species distribution curves for protonation of the monomeric unit

Single metal protonation curves of Nickel and Cadmium presented in Fig 6 are prepared according to equilibrium values defined in Table 2. For the polymer, protonation function is the average number of protons released on complexation per metal ion, therefore the nickel and cadmium complexation with PEI performed experimentally and represented with the model shown in Fig. 7. After all calculations it is clear that any separations between metal ions are possible.

Table 2. Apparent protonation and formation constants for Cadmium and Nickel (OH=Hydroxide ion, M=Metal ion, L=Monomeric unit, H=proton) (Jarvis & Wagner, 1994)

CADMIUM		NICKEL	
Equilibrium	Log K	Equilibrium	Log K
$M+OH=MOH$	3.4	$M+OH=MOH$	3.7
$M(OH)+OH=M(OH)_2$	3.6	$M(OH)+OH=M(OH)_2$	5.0
$M(OH)_2+OH=M(OH)_3$	2.6	$M(OH)_2+OH=M(OH)_3$	3.0
$M(OH)_3+OH=M(OH)_4$	1.7	$2M+2OH=M_2(OH)_2$	4.7
$M(OH)+M=M_2(OH)$	1	$4M+4OH=M_4(OH)_4$	27.9
$4M+4OH=M_4(OH)_4$	24.2	$M+2L=ML_2$	19
$M+L=ML$	8.6	$ML_2+H=ML_2H$	6
$ML+OH=ML(OH)$	4.9		
$ML(OH)+OH=ML(OH)_2$	4.4		
$ML+L=ML_2$	7.8		
$ML_2+H=ML_2H$	6.6		
$ML_2+OH=ML_2OH$	4.2		

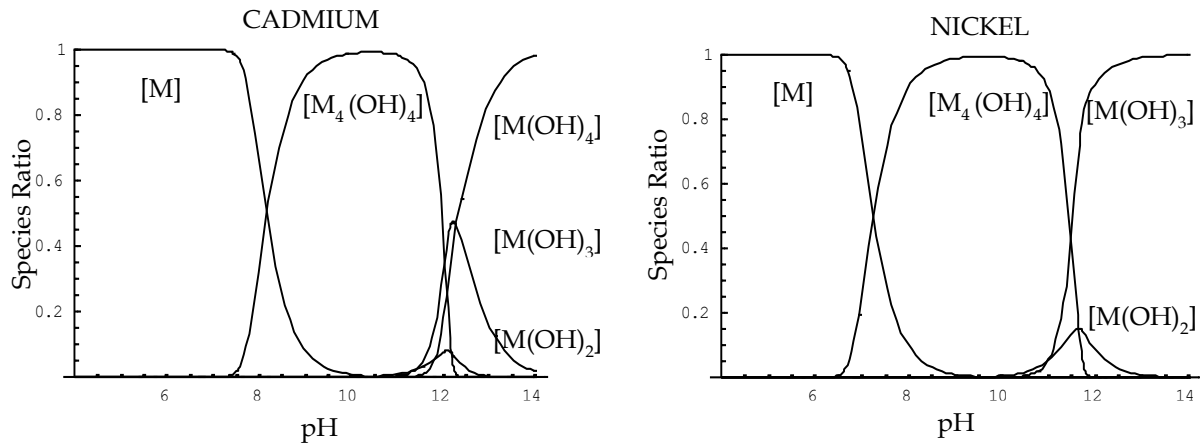


Fig. 6. Species distribution curves for protonation of Cadmium and Nickel

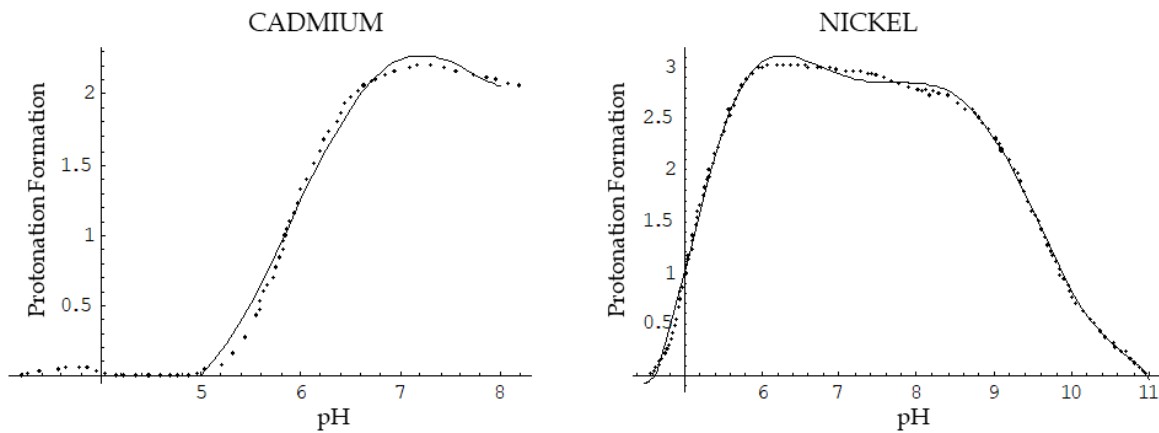


Fig. 7. Experimental (dots) and modeled (line) deprotonation curve for Cadmium and Nickel complexation with PEI

Due to the protonation graphic for nickel, the first peak of Fig. 7 indicates that three protons released per metal ion. When the protonation curve of the PEI examined, the protonation capacity of one polymer ligand is about 2 protons, so at that pH there is a complex formed by one metal ion two ligands and a H ion. That reveals ML_2H formation. The second peak of nickel in Fig. 7 is about pH 8.5, the protonation of nickel complex is nearly twice of the ligand protonation so the second type complex should be ML_2 . Equilibrium constants are calculated as:



From the species distribution curves of complexation:

$$[M] + [ML_2] + [ML_2H] = 100\% \quad (4)$$

During the membrane experiments the pH was between 4 and 7. The species distribution curve developed for the protonation of nickel ion indicates that Ni was not hydrolyzed, stayed in ionic form. So, uniform behavior of the metal ion is expected to calculate the stoichiometric retention of metal ion.

$$R = 1 - \left(\frac{[M]_p}{[M]_f} \right) \quad (5)$$

For the determination of retention rate R, $[M]_p$ and $[M]_f$ the concentration of metal ions in permeate and feed respectively are rated. The metal concentration in feed tank for Eq 4. is known, therefore retention may be formulated as;

$$R = 1 - \left(\frac{[M]_p}{[M] + [ML_2] + [ML_2H]} \right) \quad (6)$$

From the metal ion concentration in permeate $[M]_p$ it can be both obtained by calculation and experimentally. And if there is a difference between these two quantities that could be because of free metal ion retention of the membrane ($[M]_r$). So we can equalize these quantities as;

$$[M]_p = [M]_f - [M]_r \quad (7)$$

As the experiments performed batch-wise, it is recommended to make calculations step by step. Nguyen et al. (1980) used similar formula as our Eq.7 and found membrane does reject metal ions without any polymer. But this time is explained as the insoluble form of the metal ion. And there was similar approach to Aulas et al. (1980), where 1:1 complexes and complete rejection by membrane considered. Both articles had the disadvantages of hydrolysis effects in highly hydrolyzed metals.

The basics of the polymer binding/ultrafiltration process mentioned can be applied in certain circumstances, such as absence of the hydrolysis, the formation of 1:1 complexes, complete metal-binding etc. But in the system described for nickel and hereafter will be described for cadmium. Where calculations with hydrolyzed metals can be performed with metal ion species distribution curves models (Fig. 6). According to protonation of polymer-metal ion complex, there is no 1:1 complex and the solutions for 1 metal ion for 2 ligands are presented. And due to species distribution curves for metal ion polymer complex, full complexation at each pH is not expected.

In case of developing a model for a multi-component solution containing two or more different ions to bind with the polymer used in the system; the reactions in the systems are; (a) metal-polymeric complexes, (b) hydrolysis of metals, and (c) passivation of functional groups of a polymer by protonation.

The success of separation is also in agreement with the work of Jarvis et al. (1995), Li et al. (2008), Molinari et al. (2006 and 2008), Shao et al. (2013), where protonation conditions for PEI are suitable for cadmium and/or nickel are presented. According to proposed deprotonation curves for Cd-polymer and Ni-polymer complexation, it can be foreseen that Ni ions bound polymer more than Cd ions within a certain pH range of 4-5.

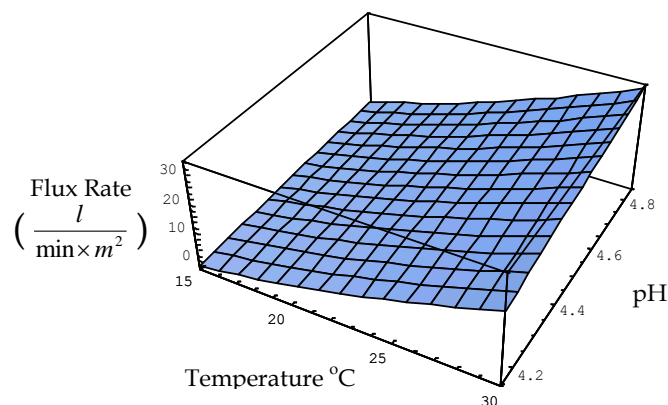


Fig. 8. Variation of flux rate of permeate with solution pH and temperature during separation experiments

The membrane system efficiencies are also evaluated by the quantity of permeate in liters produced by unit square meters of membrane area in unit time and thus the quantity of permeate is given in the form of flux rate. The increase in temperature causes the increase in flux rate, this is because of the decreasing viscosity of the solution (Fig. 8). If two graphs Fig. 2 and 3 are matched, the increase in temperature does not affect the enrichment coefficient as much as the pH differentiation does. Again this time with increasing pH in this range, the flux rate increases but the quality of retention decreases resulting as an apparent decrease in enrichment coefficient (X_E).

4. Conclusions

A polymer-enhanced ultrafiltration (PEUF) set up was designed to investigate heavy metal ions (Cd and Ni ions in this study) complexing with PEI. For a given ultrafiltration membrane, binding of heavy metal ions depends upon the loading ratio of polymer, pH, and temperature of the solution. The solution pH is particularly crucial as it controls the ability of polymer to complex with heavy metal ions

and consequently hinders their passage through the membrane. In a multi-ion system, as binding of each type of heavy metal ions depends upon the loading ratio of polymer, pH, and temperature of the solution, there is a separation window that allows selectively enrichment of each ion type whether in permeate or retentate. In this study, the increase in temperature causes the increase in flux rate and does not affect the enrichment coefficient as much as the pH differentiation does. With increasing pH in this range both of the metal ions are held by the PEI and avoid separation, and when the flux rate increases, the quality of retention decreases resulting as an apparent decrease in enrichment coefficient. The test runs showed that an enrichment coefficient (the ratio of Cd over Ni in permeate) up to 10 was obtained at pH 4.2 in temperature range of 20-22°C, whereas full retention of both cations is possible above pH 5.0 and those results are highly compatible with the model (modeling the protonation and complexation of polymer ligand) developed by using Mathematica software.

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