Removal of Nickel(II) from Aqueous Solution by Complexation-Ultrafiltration with Polyvinyl Pyrrolidone

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Abstract: This study confers insight into the removal of Nickel(II) from aqueous solution by complexation-ultrafiltration (CP-UF) technique with water-soluble polymer polyvinyl pyrrolidone (PVP). The whole experiment process includes five parts: pre-treatment, CP-UF, concentration, decomplexation and regeneration of polymer. Numerous factors affecting the retention rate of Nickel(II) (R_{Ni}) and permeate flux *(J)*, such as pH, loading ratio, transmembrane pressure (TMP), complexation time and added salt have been investigated. In the CP-UF process, R_{Ni} reaches nearly 94% while pH of 7, loading ratio of 4, TMP of 1.0 bar, temperature of 25^0C, complexation time of 30 min are chosen to be the optimal parameters. In the process of concentration, J declines slowly and R_{Ni} is very high at loading ratio of 4 and pH of 7. Nickel ion concentration in the retentate solution increases linearly with volume concentration factor. In the process of decomplexation, the decomplexation percentage of nickel(II)-PVP complex reaches 42%. The binding capacity of the regenerated PVP is close to that of fresh PVP, and the recovery percentage of binding capacity is higher than 90%.

Keywords: Nickel(II), Polyvinyl pyrrolidone, Complexation, Ultrafiltration.

1. INTRODUCTION

Nowadays, water pollution has been drawing high concern, especially poisonous heavy metal ions present in industry discharge systems which causes more and more severe environmental issue and negative impact on human health. Therefore, the toxic heavy metals in the wastewater must be removed before discharged into nature water. Wastewaters containing heavy metal ions are produced by several sources like metal finishing, electroplating, electronics, hazardous waste disposal sites and galvanic industries, etc. Conventional treatment processes like chemical precipitation, ion exchange, adsorption, coagulation and flocculation, flotation and electrochemical removal have significant disadvantages like incomplete removal, high-energy requirements, and production of toxic sludge [1]. Recently, complexation-ultrafiltration (CP-UF) technology has been shown to be a promising method for heavy metal ion recovery from aqueous solutions [2-4]. CP-UF process uses the selectivity of a complexation agent (a water soluble macromolecular polymer) along with filtration ability of UF membrane to retain metal-polymer complexes of macromolecular size greater than its molecular weight cut-off (MWCO) [5]. Water-soluble polymeric ligands have shown to be efficient substances to remove heavy metals from aqueous solutions and industrial wastewater through ultrafitration processes [6].

Nickel is one of the most important transition metals, and its high-purity products are vital for developing advanced materials. Most Nickel is used for the production of stainless steel, non-ferrous alloys and Ni-based superalloys with high corrosion and temperature resistance properties [7]. But the extensive utilization of nickel-containing products sometimes leads to environmental pollution by nickel and its by-products [8]. Nickel(II) is a kind of non-biodegradable toxic heavy metal ion present in wastewater. In order to protect the environment and human health, the maximum level of Nickel(II) discharged is 1mg/L which is established by the Environmental Protection Agency of China. So the recycling of nickel is an attractive option as it provides a means to recover and reuse the metal values and, at the same time, avoid environmental risks.

Heavy metal removal depends on complexation of metal ions by polymer and the complexation depends on some parameters like pH of feed solution, loading ratio (L), TMP, complexation time, concentration of the added salt. This study will mainly be focused on investigating the applicability of the complexation-ultrafiltration process for removal of nickel ions from synthetic wastewater solutions.

2. EXPERIMENTAL

2.1. Materials

Anionic polyvinyl pyrrolidone (PVP) with average molecular mass 40000 provided by Shanhai Gobekie

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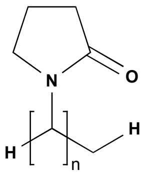
Table 1: Specification of Ceramic Ultrafiltration Membrane

Material	MWCO (kDa)	Effective filtration area (m^2)	Channel diameter (mm)	Channel numbers	Length (mm)
Aluminum oxide	10	0.03	2.27	7	600

Company (China) was used as a water-soluble polymeric ligand. Its structural formula is



Hexahydrate nickel sulfate, sodium hydroxide, hydrochloric acid, dimethylglyoxime, anhydrous ethanol and sodium chloride were all purchased from Aladdin Reagent Co., Ltd., as analytical reagent grade. Every solutions were prepared with deionized water of conductivity less than 1μS/cm.

Ceramic ultrafiltration membrane with the molecular weight cut-off of 10000 Da and an effective filtration area of 0.03 m^2 was used. It were offered by Shanghai Laungy Technology Co., Ltd. (China). The specification of the ceramic ultrafiltration membrane is given in Table **1**.

2.2. Experimental Procedures

2.2.1. Pre-Treatment of Polymer

The PVP solutions were pretreated in a diafiltration process in the experimental apparatus in order to eliminate the shorter macromolecules that can go through the membrane. The deionized water was added to the reservoir in batches, and permeate streams were sent to a separate tank. The pre-treatment was ended until PVP concentrations in the permeate streams decreased to zero [9, 10]. The concentration of the resulting polymer solution was measured with UV-Vis spectrophotometer, and the pre-treated PVP solution was used in all complexation-ultrafiltration experiments.

2.2.2. CP-UF Process

Complexation-ultrafiltration (CP-UF) is a developing hybrid process for the removal of heavy metals which uses the selectivity of complexing agent and filtration ability of ultrafiltration membrane [5]. As shown in Figure **1**, the ultrafiltration system consists of a thermostatic water bath, a feed tank, a feed diaphragm pump for the flow, two pressure meters, a ceramic ultrafiltration membrane module, a temperature meter, a flow meter and three control valves which enabled optimal running conditions. A pH meter was used to detect pH value.

2.2.3. Concentration, Decomplexation and Reuse of Regenerated Polymer

The concentration experiment was performed at pH 7 and loading ratio 4. Permeate stream was sent to a separate tank, and this experiment was ended when the volume concentration factor (VCF, defined as the ratio between the volume of feed solution and the volume of retentate) reached to 10. Whereafter, it took full use of the concentrated retentate to complete the

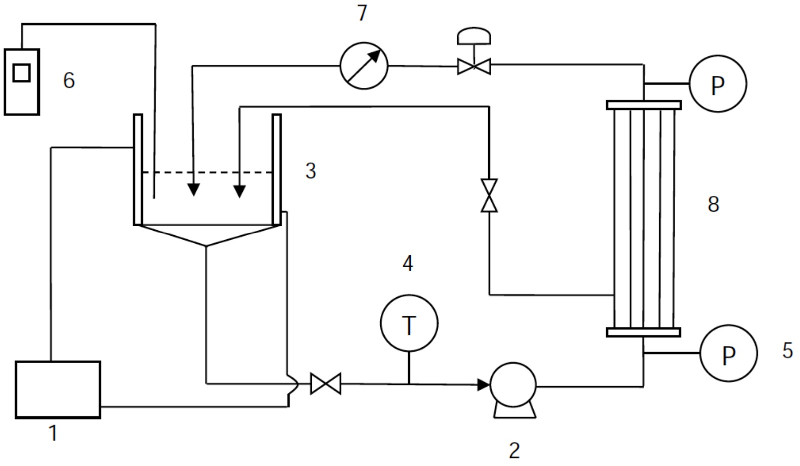


Figure 1: Schematic diagram of ultrafiltration system. (1) thermostatic water bath, (2) pump, (3) feed tank, (4) temperature meter, (5) pressure meter, (6) pH meter, (7) flow meter, (8) ceramic ultrafiltration membrane module.

decomplexation process. The decomplexation experiment was performed at pH 3 in total recirculation mode. Polymer reuse experiments were carried out using the regenerated PVP, and compared with the fresh PVP [10].

3. RESULTS AND DISCUSSION

The observed retention rate (R), which measures the separation capability of membrane, is given by Eq. (1):

$$R=(1-\frac{C_p}{C_0})\times 100\% \quad (1)$$

where C_p (g/L) is metal ion concentration in permeate solution and C_0 is initial metal ion concentration in feed solution.

Loading ratio (L) is calculated as Eq. (2) to indicate the concentration ratio of polymer ($C_{polymer}$) to metal ion(C_{Metal}).

$$L=\frac{C_{polymer}}{C_{Metal}} \quad (2)$$

In Eq. (3), J is the permeate flux (L h^{-1} m^{-2}), V denotes the permeate volume collected within time (t) per hour (h) and A is the effective membrane surface area [11].

$$J=\frac{V}{A\,t} \quad (3)$$

The volume concentration factor (VCF) is calculated by using Eq. (4):

$$VCF=\frac{V_f}{V_f-V_p} \quad (4)$$

Where V_f and V_p are the volumes of the feed and the total permeate, respectively.

3.1. Ultrafiltration of PVP Solutions

Figure **2** shows the effect of the operating time on instantaneous permeate flux (J_i) under different transmembrane pressure (TMP). The studied pH was 7 and temperature was 25^{0}C. As it is clear, J_i decreases slightly as the time varies from 5 min to 50 min, while it tends to keep stable after 50 min under different transmembrane pressure. J_i increases with increasing TMP, which can be explained by the Forshcheimer equation.

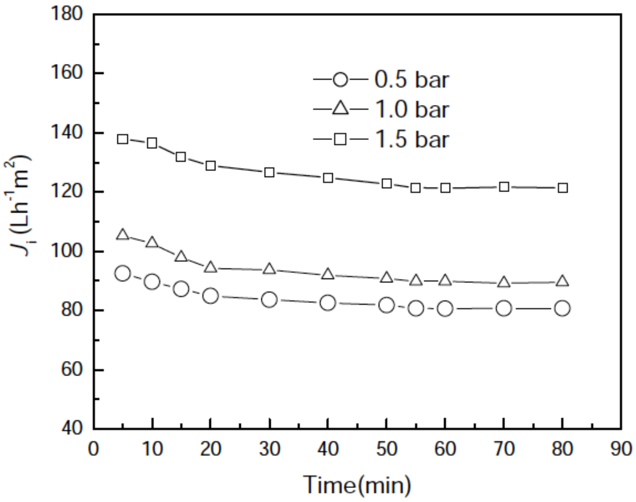


Figure 2: Effect of operating time on instantaneous permeate flux under different transmembrane pressures (pH, 7; temperature, 25˚C; initial PVP concentration, 106 mg/L).

The effect of transmembrane pressure on retention rate of PVP (R_p) has been shown in Figure **3**. When the TMP increases from 0.5 bar to 1.5 bar, R_p value changes insignificantly. It might be due to the mechanism that the TMP only changes the pressure on both sides of the membrane, just accelerates the permeate rate, and it hardly changes molecule structure of PVP or membrane's pore size, thus it does not affect retention rate.

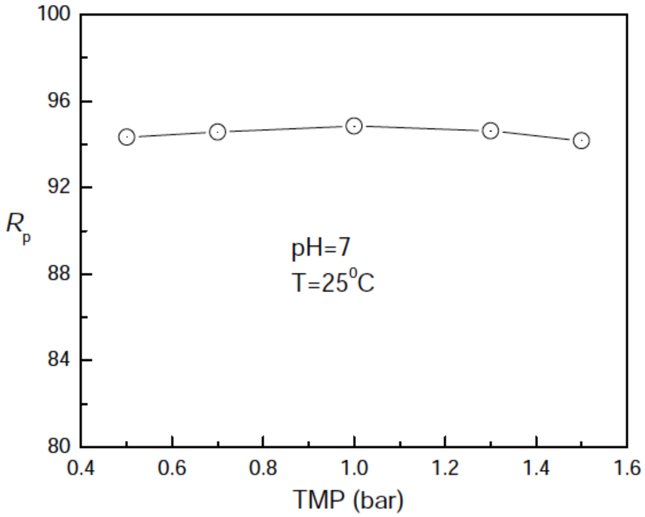


Figure 3: The effect of transmembrane pressur on retention rate of PVP (pH, 7; temperature, 25˚C; initial PVP concentration, 106 mg/L).

Figure **4** shows that steady permeate flux (J_s) are not influenced by pH. The phenomenon may be explained as: a change of the conformation of PVP molecule does not occur and membrane fouling does not change with pH.

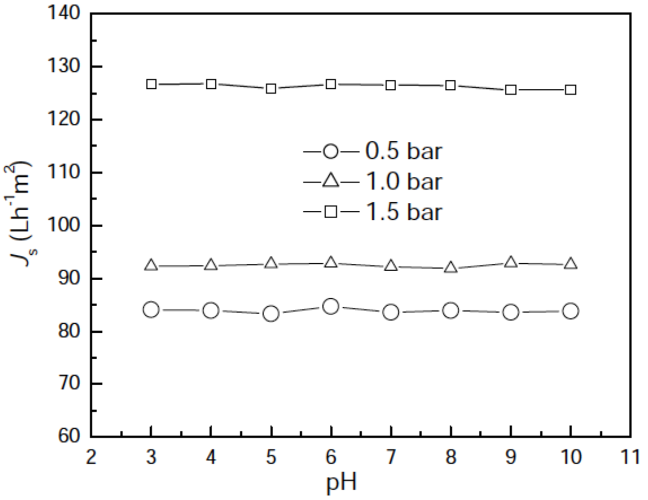


Figure 4: Effect of pH on steady permeate flux under different transmembrane pressures (temperature, 25˚C; initial PVP concentration, 106mg/L).

The effect of pH on R_p under different transmembrane pressure has been illustrated in Figure **5**. The retention rate obtained in the pH range 3-10 are almost equal. This means that R_p is not influenced by pH. This may be attributed to the stability of the PVP molecular structure which is pH independent.

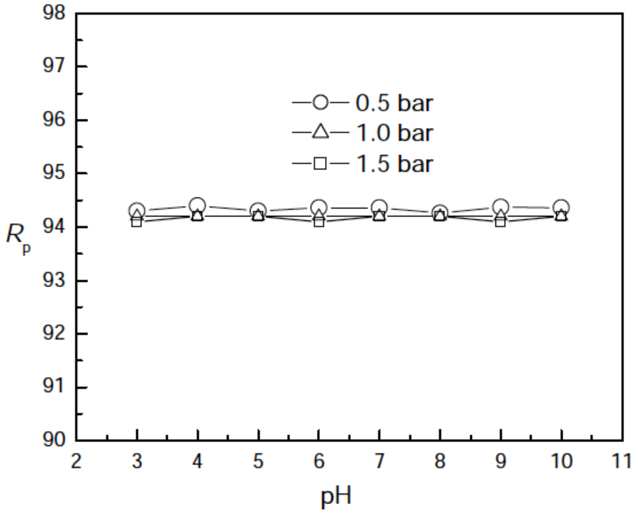


Figure 5: Effect of pH on retention rate under different transmembrane pressures (temperature, 25˚C; initial PVP concentration, 106mg/L).

3.2. Fourier Transform Infrared Spectoscopy Study

The FT-IR spectra of PVP(a) and PVP-Ni(b) are depicted in Figure **6**. As shown in lines a and b, the intensity of the broad band between 3600 and 3100 cm^{-1}, which is attributable to the -OH stretching vibrations of water. The stretching vibrations of methyl (-CH_3) and methylene (-CH_2-) groups are observed in the region 2950 to 2800 cm^{-1}. The stretching vibration of C=O bond is observed at 1674 cm^{-1} in line a, but it has weaken in lin b. The deformation vibration of C-N is found at 1098 cm^{-1} in line a, but it shifts to 1026 cm^{-1} in line b. Negatively charged nitrogen ions exist in the anionic polyvinyl pyrrolidone which may have coordination effect or electrostatic forces to bond with positive charge of nickel (II) ion. Comprehensive analysis indicates that nickel ions have been bound to the PVP.

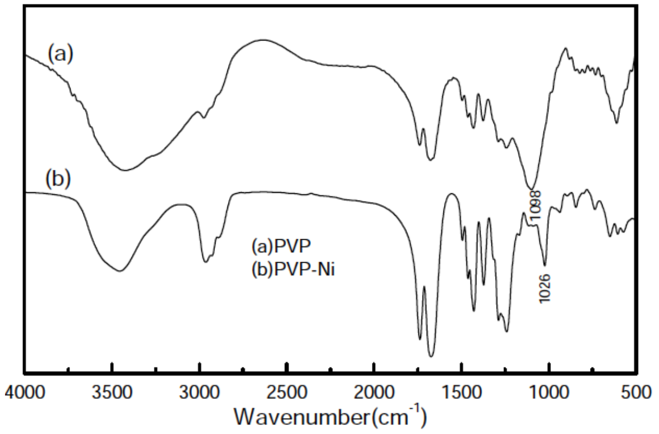


Figure 6: FTIR spectra of PVP(a) and PVP-Ni(b).

3.3. Effect of Operating Parameters on Nickel Removal

3.3.1. Effect of Complexation Time on Nickel Removal

Figure **7** shows the variations of retention rate of nickel ions (R_{Ni}) and J_i with complexation time at TMP of 1.0 bar and pH of 7.0. With increasing complexation time, R_{Ni} increases rapidly in the first 30 min, and then

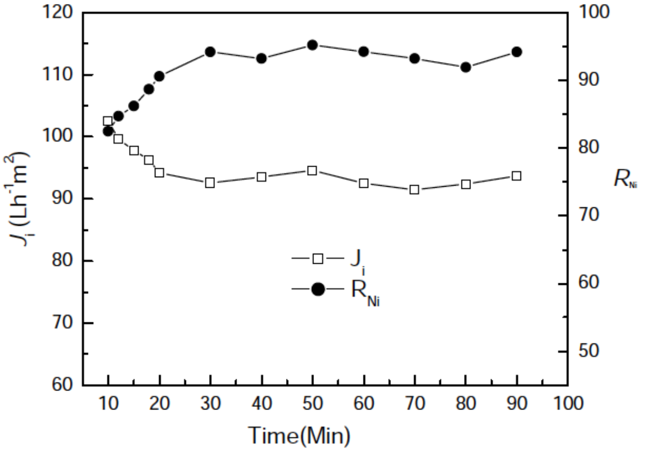


Figure 7: Effect of complexation time on permeate flux and retention rate (PVP concentration, 100mg/L; nickel concentration, 25mg/L; pH, 7; temperature, 25˚C; transmembrane pressure, 1.0 bar).

keeps steady. Since nickel concentration in the permeate is considered to be equal to free nickel concentration in the retentate, 30 min is regarded as the minimum time for reaching complexation equilibrium. In addition, J_i decreases slightly as the complexation time increases.

3.3.2. Effect of Loading Ratio on Nickel Removal

The loading ratio is one of the most important parameters for the CP-UF process [12-15]. The effect of loading ratio on R_{Ni} and J_s is shown Figure **8**. R_{Ni} value increases with loading ratio in the range 1-4, and then remains unchanged when loading ratio is beyond 4. This is because the available sites of PVP increase with loading ratio. Similar trends were observed in other studies [16, 17]. J_s decreases slightly with loading ratio, which may be due to the increased viscosity of feed solution. As a result, the optimum loading ratio of 4 was chosen for further complexation-ultrafiltration process.

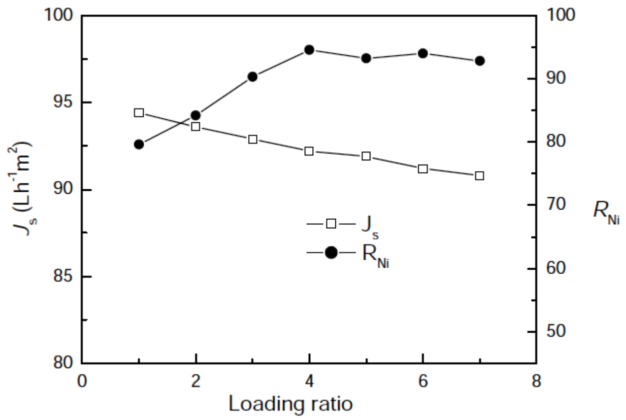


Figure 8: Effect of loading ratio on retention rate and steady permeate flux Js (nickel concentration, 25mg/L; pH, 7; temperature, 25˚C; transmembrane pressure, 1.0 bar).

3.3.3. Effect of pH on Nickel Removal

In the process of CP-UF, pH value is another important parameter. Figure **9** shows the effect of pH on R_{Ni} and J_s at temperature of 25°C and TMP of 1.0bar. R_{Ni} value increases from 79.6% to 95.7% as pH value increases from 3.0 to 10.0. This phenomenon can be explained that, competition exists between Ni (II) and proton H^+ to bind PVP in complexation process. There is chemical equilibrium and reaction formula [9] between the ligand PVP (L), proton H^+ and Ni (II) in aqueous solution, as shown in reactions (a) and (b):

$$L + H^+ \rightleftharpoons LH^+ \qquad (a)$$

$$L + Ni(II) \rightleftharpoons LNi(II) \qquad (b)$$

At lower pH, much more proton H^+ accelerates easily the formation of LH^+ in reaction (a) while it inhibits the formation of LNi(II). So, many nickel ions exist still in the free state and pass through the membrane. At higher pH, owing to the lower concentration of H^+, the formation of LNi(II) is easy in reaction (b). This makes that many nickel ions are bound to the PVP and rejected by the membrane. At neutral or basic pH, high retention is attributed to partial hydrolysis of nickel ions, resulting in formation of $Ni(OH)_2$ with minimum protonation and maximum de-protonation phenomenon, leading to the enhancement of metal retention [18]. So pH of 7 is much more suitable to obtain better R_{Ni}. In addition, J_s decreases slightly as pH value increases from 3.0 to 10.0.

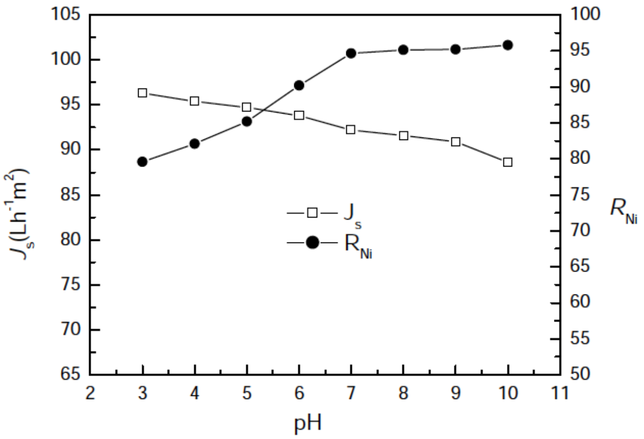


Figure 9: Effect of pH on retention rate and steady permeate flux (PVP concentration, 100 mg/L; nickel concentration, 25 mg/L; temperature, 25˚C; transmembrane pressure, 1.0 bar).

3.3.4. Effect of the Added Salt on Nickel Removal

Since chloride ions exist largely in actual industrial wastewater, sodium chloride is used as a representative salt in this work. Figure **10** shows the effect of the concentration of the added salt (sodium chloride) on R_{Ni} and J_s. Ultrafiltration experiments were conducted with the applied pressure of 1 bar, loading ratio of 4 and pH value of 7. One can see that R_{Ni} value decreases with increasing the concentration of sodium chloride, and then changes little. One possible mechanism to explain the effect of background electrolytes on nickel removal is that increasing the salt concentration of the solution probably leads to compression of the electric double layer, thus to reduction in the binding between ions and polymer [16]. As a result, the unbound nickel in the solution passes through the membrane, leading to lower retentions. It is similar to what Baticle *et al*. [19] observed for Ni(II) removal by using polyethyleneimine as a complexing agent. In addition, J_s slightly decreases from 92.2 to

87.6 L h^{-1} m^{-2} as the concentration of sodium chloride increases from 2 to 12 mg/L. It may be due to compression of the electric double layer which leads slight membrane fouling, thus there is a reduction of J_s.

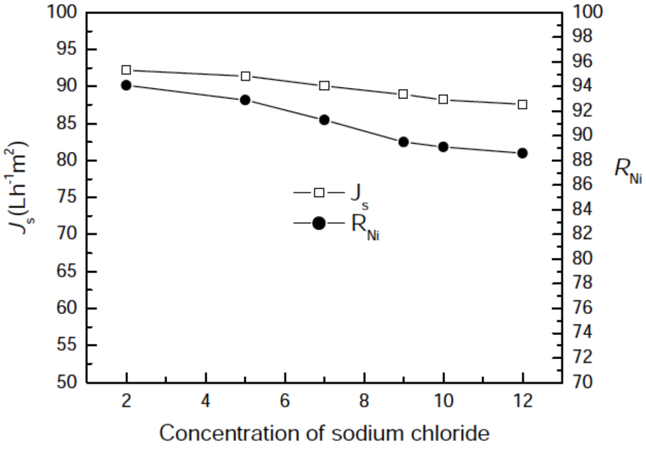


Figure 10: Effect of the concentration of sodium chloride on retention rate and membrane permeate flux (PVP concentration, 100 mg/L; nickel concentration, 25 mg/L; pH, 7.0; temperature, 25°C; transmembrane pressure, 1.0 bar).

3.3.5. Effect of Transmembrane Pressure on Nickel Removal

Figure **11** shows the effect of TMP on R_{Ni} and J_s. R_{Ni} almost does not vary with increasing the TMP. This is because the formation of PVP-Ni(II) is not influenced by the TMP. J_s rapidly increases from 83.6 to 126.6 L h^{-1} m^{-2} as the TMP increases from 0.5 to 1.5 bar. This fact indicates that the effect of concentration polarization is slight. Aliane *et al.* [20] obtained similar results in the studies of removing Cr(III) from wastewaters by ultrafiltration with the help of polyethyleneimine.

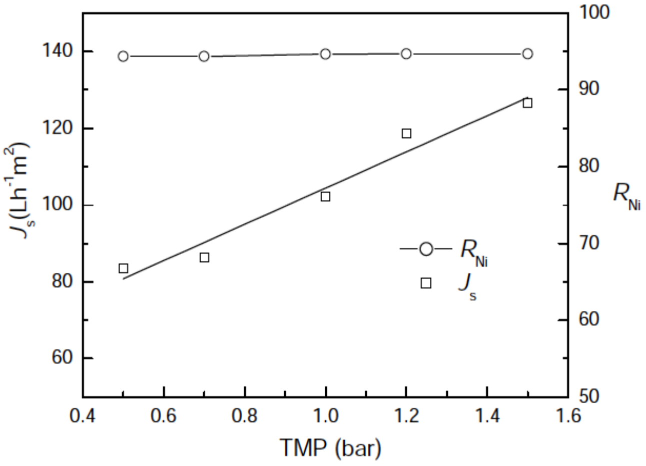


Figure 11: Effect of transmembrane pressure on retention rate and membrane permeate flux (PVP concentration, 100 mg/L; nickel concentration, 25 mg/L; pH, 7.0; temperature, 25°C).

3.4. Concentration Process

According to the results of the previous experiments, pH of 7, TMP of 1.0 bar and loading ratio of 4 were selected for the concentration process. Figure **12** shows the effect of volume concentration factor (VCF) on nickel retention rate, membrane permeate flux, nickel concentrations in the retentate (C_r) and the permeate (C_p). R_{Ni} obtained at different VCF are very high (approximated to 93%). Membrane permeate flux decreases from 97.3 to 88.6 L h^{-1} m^{-2} as the VCF increases from 1 to 10, which can be attributed mainly to the insignificant concentration polarization. With increasing VCF, there is a rapid linear increase of C_r. C_r is 192.3 mg/L when VCF reaches 10. However, C_p almost keeps a steady value and is approximately 1.5 mg/L in the whole concentration process. This implies that nickel is concentrated efficiently by the membrane under the conditions. A similar result has also been observed in our previous study [10].

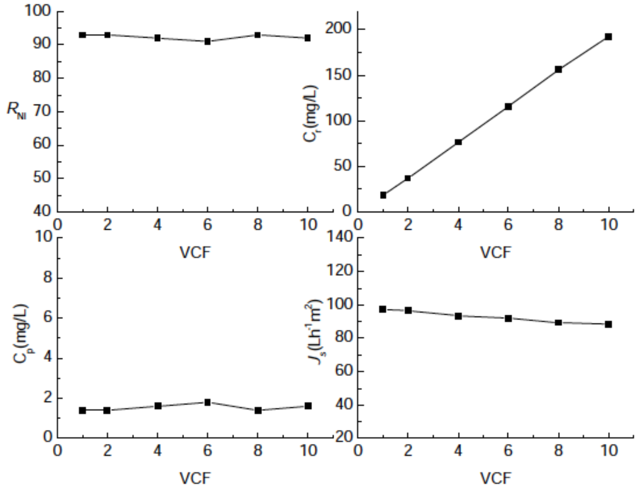


Figure 12: The effects of volume concentration factor on nickel retention rate, membrane permeate flux, nickel concentrations in the retentate and the permeate (feed volume, 10 L; initial PVP concentration, 80 mg/L; initial nickel concentration, 20 mg/L; pH, 7.0; temperature, 25°C; transmembrane pressure, 1.0 bar).

3.5. Decomplexation Process

1 L concentrated solution was obtained which contains nickel and PVP concentrations of 192.3 mg/L and 742.5 mg/L, respectively. The possibility of achieving the decomplexation of PVP-Ni(II) complexes was tested at pH 3 in total recirculation mode. Once the decomplexation began, C_r and C_p were measured at intervals. Figure **13** shows the effect of decomplexation time on permeate flux, C_r and C_p. It can be seen that

permeate flux and C_r hardly change with decomplexation time. C_p increases with decomplexation time, and it changes little after about 15 minutes. Since nickel concentration in the permeate is considered to be equal to free nickel concentration in the retentate, 15 min is regarded as the minimum time for reaching decomplexation equilibrium. The equilibrium nickel concentrations in the retentate and the permeate are 192.3 mg/L and 80.7 mg/L respectively, which means that about 42% of PVP-Ni(II) complexes are dissociated. It demonstrates that some of nickel ions remain bound by the polymer.

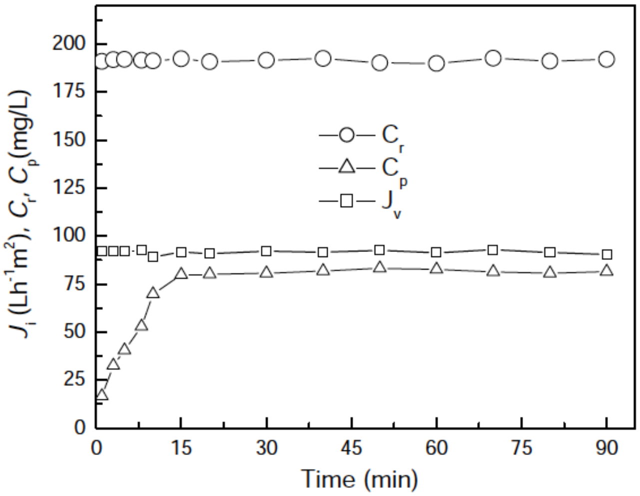


Figure 13: Effect of decomplexation time on permeate flux and nickel concentrations in the retentate and the permeate solution (feed volume, 1 L; pH, 3; temperature, 25°C; transmembrane pressure, 1.0 bar; initial nickel concentration, 192.3 mg/L; PVP concentration, 742.5 mg/L).

Table 2: The Binding Capacities of the Regenerated and Fresh PVPs

loading ratio *R* value	**1**	**2**	**3**	**4**	**5**	**6**	**7**
R of fresh PVP	79.6	84.2	90.3	94.56	94.53	94.61	94.52
R of regenerated PVP	72.6	77.4	83.5	86.1	86.7	87.0	87.1

3.6. Regeneration of Polymer

After the decomplexation experiments, the retentate was treated by adding hydrochloric acid solution of pH=3 and the regenerated PVP as pure as possible were tested by using the diafiltration method. Nickel should be progressively washed by passing the permeate, while PVP was retained in the retentate. After the previous diafiltration experiment, the regenerated PVP was used to bind nickel ions. As shown in Table **2**, the binding capacity of the regenerated PVP is close to that of fresh PVP, and the recovery percentage of binding capacity is higher than 90%.

4. CONCLUSION

The CP-UF process was successfully applied for removal of nickel from aqueous solutions. Ceramic membrane was used as the ultrafiltration membrane, while polyvinyl pyrrolidone was used as a water-soluble polymeric ligand . Summing up the results obtained, special attention should be paid to the strong influence of pH on complexation-ultrafiltration. By changing pH values, it is possible to carry out the separation of nickel(II) or to decompose nickel(II)-PVP complex. Nickel(II)-PVP complex can be concentrated effectively when the appropriate pH and loading rate (such as pH=7.0 and L=4.0) are selected. Nickel(II) is removed effectively from the retentate using the decomplexation, and the purification of regenerated PVP is acceptably satisfactory. The binding capacity of regenerated PVP is close to that of fresh PVP. Therefore, it is possible to carry out the recovery of nickel(II) and the reuse of PVP by the integration of experiments including complexation, concentration, decomplexation and regeneration.

ACKNOWLEDGEMENTS

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