



Concentration and purification of chromate from electroplating wastewater by two-stage electro dialysis processes

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ARTICLE INFO

Article history:

Received 6 December 2007

Received in revised form 24 March 2008

Accepted 17 April 2008

Available online 3 May 2008

Keywords:

Chromate

Concentration

Purification

Two-stage electro dialysis

Electroplating wastewater

ABSTRACT

A designed two-stage electro dialysis system is proposed to concentrate and purify chromate from a low pH electroplating wastewater using monovalent selective electro dialysis membranes. With low pH of the raw water (pH 2.2) in the first stage, chromate was presented as HCrO_4^- and monovalent ions (HCrO_4^- , NH_2SO_3^- , Na^+ and Cl^-) were able to pass through the membrane thus chromate was concentrated up to 191%. Higher current density, flowrate and more membrane area all increased the chromium recovery. When pH was adjusted to 8.5 before entering the second stage, the chromate species was presented as divalent CrO_4^{2-} and retained in the concentrated stream, and the rest monovalent ions (NH_2SO_3^- , Na^+ and Cl^-) were separated by passing through the membrane. For example, 45% of the chlorides were separated in this study. The separation efficiencies in the second stage were also increased when the current density, flowrate and membrane area were increased. Electron Spectroscopy for Chemical Analysis was used to examine the surface chromate species for stage 1, and anion exchange membrane showed more chromate fouling comparing to cation exchange membrane due to more adsorption and concentration polarization effects for the anion exchange membrane.

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1. Introduction

Chromium is a carcinogenic, non biodegradable and highly toxic pollutant which is widely used in the electroplating industry and also greatly affects our environments. The traditional procedures of chromium reduction in the electroplating industry are to use a reducing agent such as sodium bisulfite (NaHSO_3) to reduce Cr(VI) to Cr(III). Either $\text{Cr}_2\text{O}_3(s)$ or $\text{Cr}(\text{OH})_3(s)$ is precipitated by adjusting pH to around 8. Since the effluent chromate concentration in the electroplating industry could be as high as several hundreds mg/L as Cr^{6+} , significant amounts of sludges are generated. Thus, it is significant to develop an effective and economic process to recover chromate in the manufacturing process for both economic and environmental purposes. Electro dialysis (ED) is a membrane separation process based on the selective migration of aqueous ions through ion-exchange membranes under electrical driving forces, and it has been commercially used for desalination, wastewater treatment and electrolyte separation for many different synthetic water [1–9] and wastewater [10–14]. Several studies have also reported for concentrating chromium species using ED [10,11,15]. However, the downside for using tra-

ditional ED process to recover chromate is that the traditional ED with anion exchange membrane (AEM)/cation exchange membrane (CEM) coupling can only concentrate the wastewater without any separation of other ion impurities. Therefore, in this work, an attempt has been made to recover chromate from an electroplating wastewater using a novel two-stage electro dialysis (TSED) process coupling with monovalent selective ED membranes to establish a close circuit for chromium reuse for electroplating industry and to remove the monovalent ion impurity at the same time. Since chromate is a divalent species with $\text{p}K_{a2}$ of 6.8, for pH lower than 6.8, chromium is shown as monovalent species HCrO_4^- , but for pH higher than 6.8, chromium is shown as divalent species CrO_4^{2-} . Monovalent selective ED membranes can achieve the purposes of separation and concentration of chromate based on these characteristics. Under low pH (<6.8), HCrO_4^- was able to pass through the membrane and concentrated together with other monovalent ions (i.e. HCrO_4^- , NH_2SO_3^- , Na^+ and Cl^-). Under high pH (>6.8), the chromate species was presented as divalent CrO_4^{2-} and retained in the concentrated stream, but the rest monovalent ions (i.e. NH_2SO_3^- , Na^+ and Cl^-) were separated by passing through the membrane. Therefore, the TSED system was designed to concentrate HCrO_4^- in the first stage, but to separate the monovalent ion from CrO_4^{2-} in the second stage based on their monovalent/divalent properties for different pHs.

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None of the researches has used the designed TSED processes, but there are several studies for chromium removal through ED membrane. Khan et al. [9] designed an electrochemical membrane reactor to effectively recover chromic acid and potassium hydroxide using the hydrogen ion and hydroxide ion from the water spitting process in the anode and cathode. Vallejo et al. [10] studied the transfer of Cr(VI) oxo-anions through an AEM and the results showed the electrotransport was a functions of pH and $\text{CrO}_3(\text{s})$ concentration on both sides of the membrane. This transport was also depended on the polychromate ion formation within the membrane due to high concentration [15]. Tor et al. [11] demonstrated the simultaneous recovery of trivalent and hexavalent chromium ions through charged ion-exchange membranes by three detachable cells at different current densities, and they found that the transport of both oxidation states of chromium ions through membranes was correlated with both current density and flux. Peng et al. [15] reported that chromate removal was achieved by chemical precipitation/electrodialysis, but sludge was generated in the chemical precipitation process. In addition, some literatures described the separation of chromium and other ions by ED processes. Lambert et al. [16] studied separation of sodium ions from trivalent chromium by electrodialysis using modified monovalent cation selective membranes and demonstrated that separation of trivalent chromium and sodium ion is possible by regulating the pH in the diluted chamber to avoid chromium hydroxide precipitation. Frenzel et al. [17,18] investigated various AEM for chromic acid recovery and found out that at low initial current densities ($10\text{--}20\text{ mA/cm}^2$) and feed flowrate higher than 7 cm/s could help overcoming the process limitations due to high AEM resistance.

Since no literature has reported the newly designed TSED system, this study discusses the operating parameters, including voltage, flowrate, current density and ED membrane surface area for concentration and purification effects of chromium recovery for the TSED system. Besides, possible fouling mechanisms for the ED membranes were also determined by membrane surface analysis using Electron Spectroscopy for Chemical Analysis (ESCA).

2. Methods and materials

The electroplating wastewater used in this work was collected from the Dashing electroplating plant located in Taipei, Taiwan. Water qualities for the wastewater are given in Table 1. The scheme

Table 1

The chemical analysis of the raw electroplating wastewater

Cr(VI)	Ca ²⁺	Mg ²⁺	SO ₄ ²⁻	Na ⁺	Cl ⁻	TOC	pH
418	7.4	2.3	19.8	164	90	4.2	2.2

Unit: mg/L except for pH is unitless.

of the experimental set up is shown in Fig. 1. The initial pH for raw water (spent plating solution) was around 2.2 where chromium was presented as monovalent HCrO_4^- . Since chromate is a divalent species with $\text{p}K_{a2}$ of 6.8, for pH lower than 6.8, chromium is shown as monovalent species HCrO_4^- , but for pH higher than 6.8, chromium is shown as divalent species CrO_4^{2-} . Therefore, in the first stage, HCrO_4^- was able to pass through the membrane and concentrated together with other monovalent ions (i.e. HCrO_4^- , NH_2SO_3^- , Na^+ and Cl^-). Between first and second stage, pH was adjusted to 8.5 to change the chromium speciation to divalent CrO_4^{2-} . Under high pH (>6.8), the chromate species was presented as divalent CrO_4^{2-} and retained in the concentrated stream, but the rest monovalent ions (i.e. NH_2SO_3^- , Na^+ and Cl^-) were separated by passing through the membrane. Therefore, the TSED system was designed to concentrate HCrO_4^- in the first stage, but to separate the monovalent ion from CrO_4^{2-} in the second stage based on their monovalent/divalent properties for different pHs.

The CEM/AEM membrane used in this study is Asahi Selemion AST/CMV membrane (Asahi Glass Co., Tokyo, Japan). Asahi Selemion AST membrane is a monovalent-anion-permselective membrane is of the styrene-divinylbenzene with high transportation for chloride with thickness of $0.23\text{--}0.27\text{ mm}$. Selemion CMV is a non-selective sulfonic acid groups as fixed charges with thickness of 0.15 mm . Since the wastewater in electroplating industry has the properties of small quantity but high concentration, a recirculation type operation was conducted in which three independent batch of raw water (two liter of concentrated, diluted, electrode solutions) were utilized initially and then recalculated to achieve higher efficiency. The system was operated at constant current; therefore, the system resistance was obtained from the voltage divided by the current. Besides, the electrode was reversed for 5 min/h to prevent possibly blockage in the TSED system due to high chromate concentration. Three different parameters with two levels each are evaluated in this study: current density (10 mA/cm^2 and 30 mA/cm^2), flowrate (5 ml/s and 10 ml/s) and membrane sur-

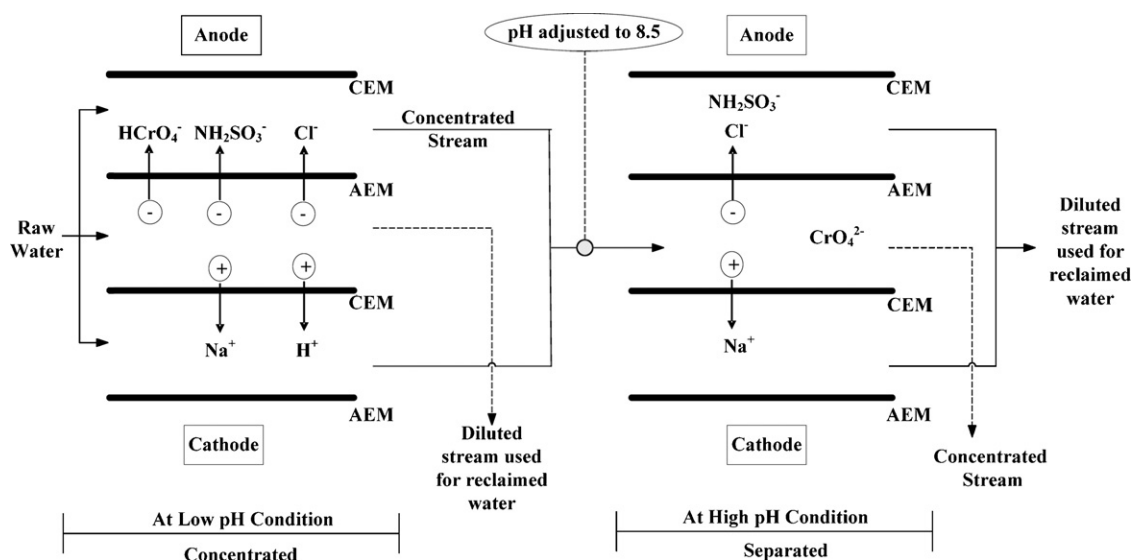


Fig. 1. Schematic flow diagram for the TSED process to demonstrate the transportation of the major ions.

face area (400 cm² and 800 cm²). The values of current density and flowrate are typically used values based on the results from Frenzel et al. [17,18].

The AEM and CEM were laminated in the TSED system, and the distance between the AEM and CEM is 14 mm including the spacer of 4 mm for water to pass through. A power supply (GPR16H50D, Good Will Instrument Co., Ltd., Taiwan) was used to provide a constant DC current. The electrodes used in this study are SUS-316 stainless steel and the membrane areas for each AEM or CEM are 10 cm × 10 cm or 100 cm².

Sodium, chloride, calcium, magnesium and sulfate were measured by an ion chromatography (Dionex ICS-90). Hexavalent chromium was measured colorimetrically according to the methods 3500-Cr listed in the 20th edition of the Standard Methods [19] by reaction with diphenylcarbazide in acidic solution to generate a red-violet color and measure the absorptivity at 540 nm using a UV-vis spectrophotometer (HACH Model DR-4000).

Membrane surface analysis was conducted by ESCA which is purchased from Omicron Nanotechnology GmbH, Germany. The used membranes were rinsed with de-ionized water and air-dried before ESCA analysis to avoid contamination from the concentrated solution.

3. Results and discussion

3.1. Speciation of chromium at different pH

When anhydride chromic acid CrO₃, a raw material for chromium electroplating process, dissolved into water, the chromium species could be H₂CrO₄, HCrO₄⁻, CrO₄²⁻, Cr₂O₇²⁻ or HCr₂O₇⁻ depended on the pH and influent chromium concentration according to Fig. 2 which is plotted using Eqs. (2)–(5) [20]. From Fig. 2, for pH lower than 6.8, chromium was shown as monovalent species HCrO₄⁻ and for pH higher than 6.8, chromium was shown as divalent species CrO₄²⁻. In order to separate the chromium species, the pH needs to be increased to at least 8.5 to become divalent CrO₄²⁻ in stead of monovalent species HCrO₄⁻. Besides, for raw water 418 mg/L as Cr or 10^{-2.1} M, there is only small portion of dichromate in the raw wastewater.

3.2. Stage 1: effect of current density, flowrate and membrane surface area for condensation of chromium

Table 2 presents the pH, voltage and resistance for the beginning and end conditions for two different current densities in the stage 1. Firstly, pH was decreased in the concentrated stream, since monovalent HCrO₄⁻ and hydrogen ion were transferred from diluted stream to the concentrated stream, resulting in high HCrO₄⁻ and low pH in the concentrated stream. The pH of electrode solution also increases since the solution was directly attached to the electrode. The end voltage was gradually increased to more than twice as the initial voltage since the system was operated at constant current but the resistance was gradually increased due to less con-

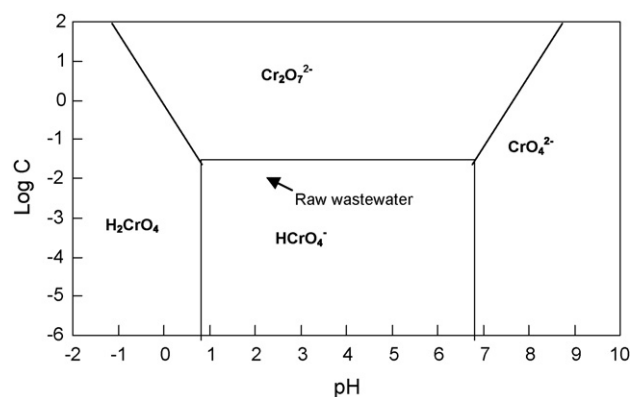


Fig. 2. Predominance diagram of five major chromate species (H₂CrO₄, HCrO₄⁻, CrO₄²⁻, HCr₂O₇⁻ and Cr₂O₇²⁻) based on thermodynamic database listed in Stumm and Morgan [20].

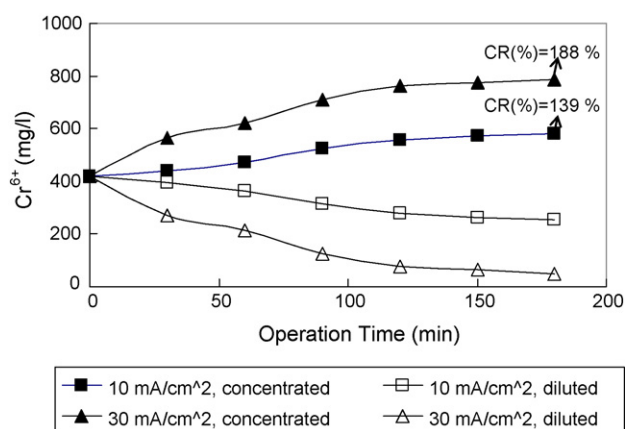


Fig. 3. The chromium recovery with different current density in 180 min. Operating condition: flowrate is 5 ml/s with two pairs of ED membranes.

ductivity (TDS decreased from 700 mg/L to 105 mg/L) in the diluted solution and the possible fouling on the ED membrane surface due to polychromate formation [15]. ESCA was used for analysis of fouling materials and presented in the later section. Comparing these two different current densities, pH, voltage and resistance were all higher for the high current density since more transferred HCrO₄⁻ from diluted stream to concentrated stream, causing more pH variation and more voltage drop. Fig. 3 presents the chromium recovery (CR) for different current densities, where CR is defined as the chromate concentration at time *t* of the concentrated stream (*C_t*) divided by the initial chromate concentration in the feed stream (*C₀*).

In Fig. 3, with the current density stayed at 10 mA/cm², only 139% CR were achieved after 180 min of operation, but when the current density increased to 30 mA/cm², CR were as high as 188% after 180 min of operation. According to Nernst equation, high current would contribute to more ion transfer in ED process thus higher

Table 2
pH, voltage and resistance for the two different current densities in stage 1

Current density (mA/cm ²)		Initial pH	Final pH	ΔpH	Initial voltage (V)	Final voltage (V)	ΔV	Initial resistance (Ω)	Final resistance (Ω)	ΔΩ
10	C	2.20	1.98	-0.22						
	D	2.20	2.30	+0.10	19.5	43.2	23.7	19.5	43.2	23.7
	E	2.20	3.15	+0.95						
30	C	2.20	1.57	-0.63						
	D	2.20	2.40	+0.20	61.5	167.5	106.0	20.5	55.8	35.3
	E	2.20	3.22	+1.02						

C: concentrated stream, D: diluted stream, E: electrode solution. Flowrate=5 ml/s and membrane surface area = 400 cm².

Table 3
pH and voltage for the two different flowrates in stage 1

Flowrate (ml/s)		Initial pH	Final pH	Δ pH	Initial voltage (V)	Final voltage (V)	Δ V	Initial resistance (Ω)	Final resistance (Ω)	$\Delta\Omega$
5	C	2.20	1.98	-0.22	19.5	43.2	23.7	19.5	43.2	23.7
	D	2.20	2.30	+0.10						
	E	2.20	3.15	+0.95						
10	C	2.20	1.88	-0.30	21.7	73.1	51.4	21.7	73.1	51.4
	D	2.20	2.68	+0.30						
	E	2.20	3.20	+1.00						

C: concentrated stream, D: diluted stream, E: electrode solution. Membrane area = 400 cm² and with current density = 10 mA/cm².

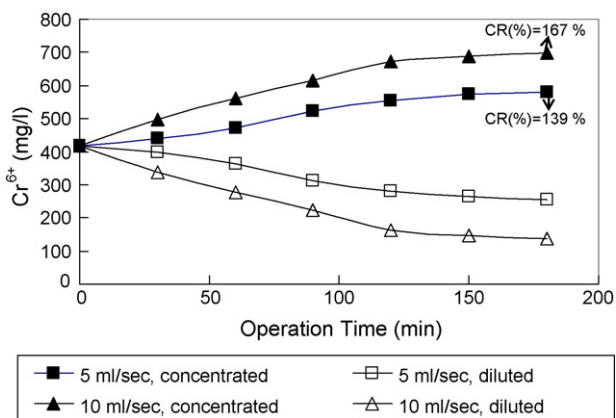


Fig. 4. The chromium recovery with the two flowrates. Operating condition: CD = 10 mA/cm² with two pairs of ED membranes.

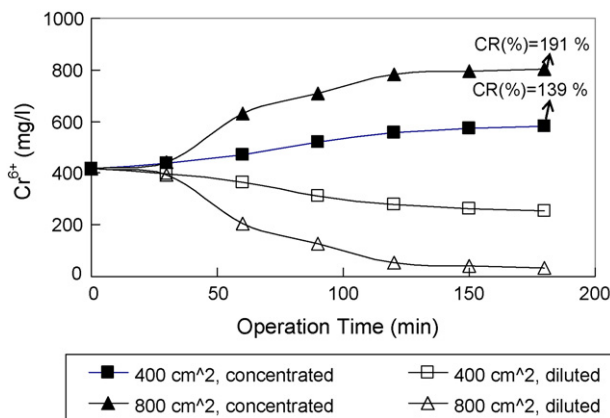


Fig. 5. The chromium recovery with varied membrane pairs. Operating condition: CD = 10 mA/cm² with flowrate of 5 ml/s.

current density was more effective accordingly. Moreover, theoretically, when the current densities increased three times (i.e. from 10 mA/cm² to 30 mA/cm²), the ion transfers (or the increases in CR) should be increased three times. However, according to Fig. 3, the increases in CR were about 2.3 times, indicating more concentration potential has to overcome and a lower CR was observed at the higher current density. Furthermore, with maximum CR of 200% for the system (i.e. all the monovalent chromate transferred to the concentrated stream), 188% was quite high and can be even higher if the system was designed in series.

Effect of flowrate was discussed since it directly affects the ion transfer, as specified by Tor et al. [11] and Frenzel et al. [17,18]. Table 3 presents the pH, voltage and resistance for the beginning and end conditions for the two different flowrates. pH was decreased in the concentrated stream but increased in the diluted stream, and voltage was gradually increased to more than twice after 180 min. Comparing these two different flowrates, pH, voltage and resistance were all higher for the high flowrate since more transferred HCrO₄⁻ from diluted stream to the concentrated stream resulting in more pH variation and more voltage drop. Fig. 4 presents CR for the two different flowrate. In Fig. 4, with the flowrate stays at 5 ml/s (flux of 0.0625 cm/s), only 139% of CR were achieved after 180 min, but when the flowrate increased to 10 ml/s (flux of

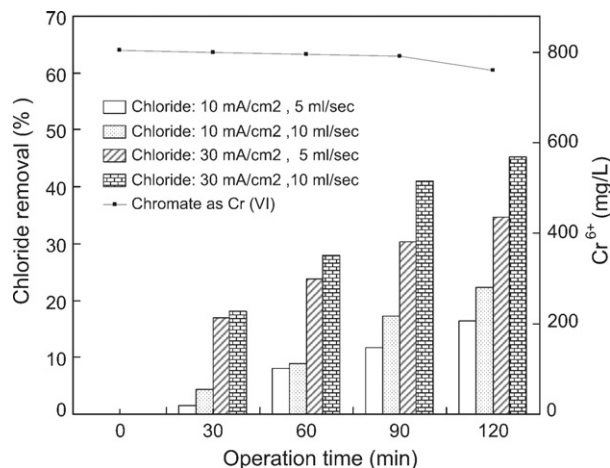


Fig. 6. Chloride separation efficiency from divalent chromate for the stage 2.

Table 4
pH, voltage and resistance for the two different membrane areas in stage 1

Membrane area		Initial pH	Final pH	Δ pH	Initial voltage (V)	Final voltage (V)	Δ V	Initial resistance (Ω)	Final resistance (Ω)	$\Delta\Omega$
400 cm ²	C	2.20	1.98	-0.22	19.5	43.2	23.7	19.5	43.2	23.7
	D	2.20	2.30	+0.10						
	E	2.20	3.15	+0.95						
800 cm ²	C	2.20	1.60	-0.60	43.5	167.7	124.2	43.5	167.7	124.2
	D	2.20	2.40	+0.20						
	E	2.20	2.26	+1.06						

C: concentrated stream, D: diluted stream, E: electrode solution. Flowrate = 5 ml/s and current = 1 A with current density = 10 mA/cm².

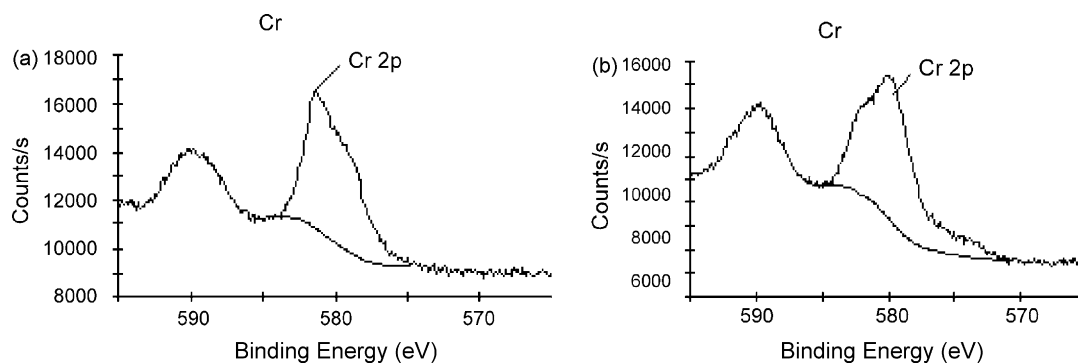


Fig. 7. ESCA Spectrum for (a) used AEM (b) used CEM.

0.125 cm/s), CR was as high as 167% after 180 min. The results indicated CR reached maximum after 180 min of operation.

Effects of membrane surface area were also discussed, since high membrane surface can achieve more ion transfers. Two different membrane surface areas (two CEM and AEM each, totally 400 cm², and four CEM and AEM each, totally 800 cm²) were experimented. Table 4 shows pH, voltage and resistance for two different membrane areas. The pH decreases in the concentrated stream, increases in the diluted stream and voltage increases were explained previously. Higher pH variations for higher membrane areas were seen for concentrated, diluted and electrode stream due to more membrane areas allowing more HCrO₄⁻ to pass. Initial voltage was also higher since more membrane areas attributed to more resistance. Fig. 5 presents CR for the two different membranes areas. In Fig. 5, with membrane area of 400 cm², about 139% CR were achieved after 180 min of operation, but when the membrane area increased to 800 cm², the increase in ion transfer was increased about twice, corresponding to the twice of the membrane surface area. Therefore, in order to improve the CR, more membranes are also necessary to allow more HCrO₄⁻ to pass through. The results also showed CR reached steady state after 180 min of operation, representing the effect of concentration for chromate reached maximum after 180 min for these ED membranes.

3.3. Stage 2: effect of current density, flux and membrane surface area for separation of chromium

Effect of chromium separation from monovalent ion for the stage 2 of the TSED system is shown in Fig. 6, where bar chart is the monovalent ion removal (%) and chromium concentration is the dashed line. The monovalent removal is represented by chloride since chloride is the most prevalent monovalent anion in this system. Influent water from the stage 1 was operated with membrane area of 800 cm², flowrate of 10 ml/s, current of 1 A and current density of 10 mA/cm². Since pH was adjusted to 8.5 before the second stage, chromate speciation was converted to divalent CrO₄²⁻, thus the CrO₄²⁻ was retained in the concentrated stream and the other monovalent ion impurities passed through the monovalent selective ED membrane. Consequently, divalent chromate and monovalent ion were separated in the second stage. In Fig. 6, chromate concentration remained around 800 mg/L (as Cr⁶⁺) throughout the operational time 120 min, but the chloride was decreased as the time was increased. With 30 mA/cm² of current density and flowrate of 10 ml/s, the chloride was reduced from 90 mg/L to 50 mg/L. Therefore, in the second stage, chloride were able to separate about 45% from CrO₄²⁻ for the second stage of the TSED system and would be higher if current density, flowrate and membrane area were increased.

3.4. Surface analysis for the TSED membranes by ESCA

ESCA was used to exam the oxidation state of the surface species for the first stage TSED membrane, since monovalent HCrO₄⁻ actually passed through the membrane. The theory for ESCA is the measurement of the chemical binding state of the surface molecules determined by the chemical shift of the electronic states of the atoms involved in the reaction based their binding energies on the surface material. Fig. 7(a) and (b) shows ESCA spectrum for AEM and CEM in the stage 1 for the surface analysis of the binding energy for different orbital. From Fig. 7(a) and (b), the binding energies are clearly important around 580 eV (the peaks above the baselines) and hexavalent chromium were identified according the Handbook of X-ray Photoelectron Spectroscopy [21]. Although both AEM and CEM are shown with the presence of the hexavalent chromium, higher response for AEM was obtained comparing to CEM. It can be explained as, since monovalent HCrO₄⁻ passed through the AEM rather than CEM, both adsorption and concentration polarization result in the higher surface concentration on the AEM. The lower chromate signal in CEM can be attributed to the adsorption of cation on the CEM but the cation was attached with chromate to maintain electroneutrality. Therefore, the response for AEM was higher for the AEM (17,000 count/s) than that of the CEM (15,000 count/s).

4. Conclusions

With low initial pH for the raw water (pH 2.2) for the TSED system, chromate species was presented as monovalent HCrO₄⁻ and was able to pass through the first stage ED membrane with other monovalent ions, thus chromate was concentrated to 191% in this study. Higher current density, flowrate and more membrane areas can increase the chromium recovery. With pH was adjusted to 8.5, the chromate species was presented as divalent CrO₄²⁻ and retained in the concentrated stream, but the monovalent ions were able to separate by passing through the second stage ED membrane. With 30 mA/cm² of current density and flowrate of 10 ml/s with membrane area of 400 cm², chloride were able to separate about 45% from CrO₄²⁻ for the second stage of the TSED system. ESCA was used to examine the oxidation state of the surface chromate species in the stage 1 for used AEM and CEM. AEM showed more chromate fouling comparing to CEM probably due to more adsorption and concentration polarization effects for the AEM.

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