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Recovery of chromium from plastic plating wastewater by cetyltrimethylammonium bromide MEUF and electrodialysis

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ABSTRACT

To study the recovery of chromium for micellar-enhanced ultrafiltration (MEUF) process, a two-stage process was designed to concentrate chromium by MEUF with cetyltrimethylammonium bromide (CTAB), then recovered by electrodialysis (ED). Wastewater from full-scale plant containing heavy metals, including chromium, copper, and nickel, with $\text{pH} < 2$ were tested to investigate the effects of pH, surfactant/metal (S/M) ratio, and operating pressures. The results showed the removal efficiencies of chromate were increased at lower pH and higher CTAB concentration. The micellar hydrodynamic diameters (D_{hs}) gradually increased with the concentration of CTAB as Cr(VI) of 1.0 mM, and ranged from 3.1 to 4.7 nm. The zeta potentials gradually increased with the concentration of CTAB, but reached plateau for concentration higher than critical micelle concentration. The experimental results indicated that the removal of Cr(VI) by MEUF exceeded 96% for operating parameters of pH 1.53, $\Delta P = 30$ psi, and $S/M = 2$. For ED process, the effects of current densities and current efficiencies were evaluated as the concentration ratio of 95%, and the chromium was able to pass through anion exchange membrane to recover at the anode side of the ED process up to 85.3% at current density of $20\ \text{mA/cm}^2$, and operating time of 360 min.

Keywords: Chromium; Cetyltrimethylammonium bromide; Micellar-enhanced ultrafiltration; Electrodialysis

1. Introduction

The electrodeposition of chromium on basis metals is widely used in the electroplating industry. A chromium plating bath consists of chromic acid and sulfate in predetermined concentrations, and iodide, bromide, or chloride ions are used as additives to operate at a high current efficiency (CE). Under oxidizing conditions, Cr(VI) is highly soluble and exists in different forms, such as H_2CrO_4, $HCrO_4^-$, CrO_4^{2-}, and $Cr_2O_7^{2-}$,

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and the proportions of these species vary with pH. Nowadays, several researches have been revealed about the carcinogen, mutagen, and high toxicity for chromium which greatly affect our environments [1–5]. The traditional procedure of chromium reduction is to reduce and precipitate the chromium species in the form of $Cr_2O_{3(s)}$ or $Cr(OH)_{3(s)}$ by pH adjustment, which yields significant amount of sludge and needs further disposal [6–10]. Therefore, this procedure is not cost effective and environmentally friendly [11]. To recover chromium, micellar-enhanced ultrafiltration (MEUF) with surfactant process is applied by forming micelle with surfactant over critical micelle concentration (CMC) in the solution, and then entrap the metal–micelle complexes by UF membranes.

This process has been widely reported for removal of heavy metals ions such as Cu^{2+}, Ni^{2+}, Pb^{2+}, Zn^{2+}, Cr^{3+}, and Cr(VI) [12–18]. For instance, Choi et al. [19] used cationic surfactants (cetylpyridinium chloride and hexadecyltrimethylammonium bromide) to investigate the adsorption of Cr(VI) on surfactant-modified activated carbons. Baek et al. [20] used modified cationic starch to remove chromate from an aqueous stream. Danis and Keskinler [21] applied CTAB under different transmembrane pressures and cross-flow velocities to evaluate surfactant and metal rejections, and transient and steady state fluxes. Ghosh and Bhattacharya [22] evaluated the influence of independent variables on the retention of the surfactant and chromate ions as well as on permeating flux using cetylpyridinium chloride to analyze the adsorption of chromate ions on the surfaces of the surfactant micelles. However, summarizes from the above research results, the leakage of Cr(VI) and surfactant monomer as well as membrane fouling may have occurred, which induces the secondary pollution and reduce the effectiveness of MEUF process. Therefore, study of optimal operation conditions is required for different chromate wastewater. In addition, the concentrate stream has to be recovered to avoid disposal problem.

Electrodialysis (ED) is a membrane separation process based on the selective migration of aqueous ions transported through ion exchange membrane by either attraction or repulsion with outside electric field as driving force [23,24]. There are also several studies for chromium removal by ED process. Tor et al. [25] demonstrated the simultaneous recovery of trivalent and hexavalent chromium ions through charged ion exchange membranes by three detachable cells at different current densities, and found out that the transport of oxidation states of chromium ions through membrane was correlated with both current density and flux. Vallejo et al. [26] studied the transfer of Cr (VI) oxo-anions through an anion exchange membrane (AEM) and the results showed the electrotransport was a function of pH and $CrO_{3(s)}$ concentration on both sides of the membrane. This transport also depended on the polychromate ion formation within the membrane due to high chromic acid concentration [27,28]. Khan et al. [29] designed an electrochemical membrane reactor to effectively recover chromic acid and potassium hydroxide using the hydrogen and hydroxide ions from the water splitting process in the anode and cathode. Moreover, a two-stage ED process coupled with monovalent selective ED membrane has also been used to treat chromate from electroplating wastewater to concentrate and purify chromate [30]. As indicated above, the application of ED is not only able to recover ionized species, but also suitable for separating non-ionized from ionized components. Therefore, the purpose of this study is to recover chromate from a full-scale electroplating wastewater using an integrated process of MEUF followed by ED (MEUF/ED), and to establish a close loop on chromium recovery for electroplating industry, at the same time. Since no literature has reported the newly designed MEUF/ED system, this study discusses the operating parameters, including pH, molar ratio S/M, pressure, and current density, and purification effects of chromium recovery for the MEUF/ED system.

2. Methods and materials

Fig. 1 is the schematic diagram of experimental setup system. Stage 1 is the UF module that is comprised of batch tank and UF unit in which UF unit is fabricated with stainless outside crust and plastic membrane support. The UF membrane is thin-film composite membrane (type: GM) manufactured by GE-Desal which has the molecular weight cut-off of 8,000 Da with operating pH range from 2 to 11, maximum operating temperature 50˚C, and maximum operating pressure 200 psi. Stage 2 is the ED module in which the chambers are separated into two compartments by AEM, and two working electrodes are set at each side. AEM is manufactured by Sybron Chemicals Inc. (IONAC MA-3475); the permselectivity (0.5 N NaCl/1.0 N NaCl) 99%, ion exchange capacity (meq/g of dry membrane) 1.00, and thickness 16 mils. All chemicals used are analytical grade reagents, and K_2CrO_7 and CTAB are obtained from Merck and Sigma-Aldrich, respectively.

The first stage of MEUF experiments was carried out in a batch tank; the synthetic wastewater and full-scale plant wastewater with surfactant were well mixed according to experimental parameters,

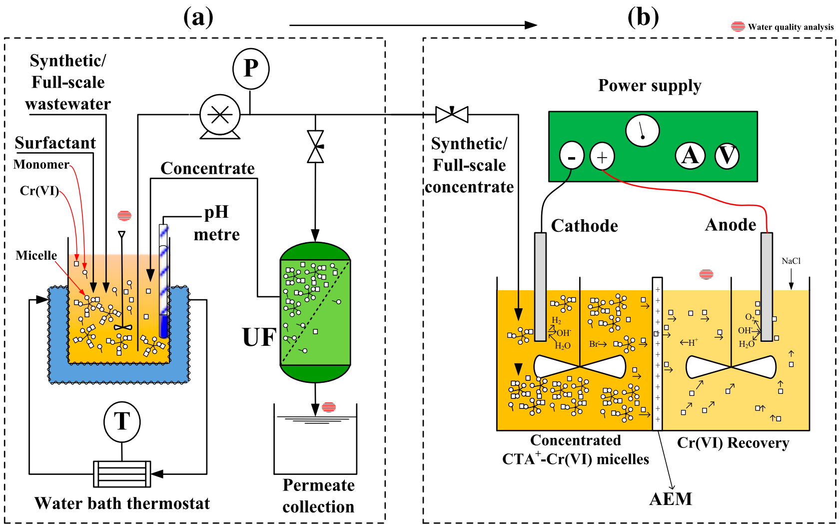

Fig. 1. Schematic diagram (a) stage 1: MEUF process, (b) stage 2: ED process.

respectively. For the preparation of synthetic samples, Cr(VI) and CTAB stock solutions were prepared by dissolving K_2CrO_7 and CTAB in deionized water, respectively. The full-scale plant wastewater used in this work was collected from the plastic electroplating plant located in Taipei, Taiwan, and water qualities for the plastic electroplating plant wastewater are given in Table 1. In these batches operation, concentrate stream was recirculated to the feed water tank for concentration ratio of 95%, but permeate was withdrawn for collection. The concentrated mixture solution of chromate and surfactant was then withdrawn into the second stage of ED process of the cathode side to evaluate the recovery of CTA^+ and Cr(VI), respectively.

In the first stage of MEUF process, the experimental works firstly used synthetic wastewater to evaluate the effects of various parameters such as pH, molar ratio S/M, and operating pressure. Since several literatures have been reported the CMC of CTAB range from 0.9 to 1.0 mM, the formation of micelles at concentrations above the CMC caused the micellization to adsorb counterion in MEUF process [31–34]. The effects of pHs were firstly determined as the molar ratio of surfactant to chromate ions (S/M) of $[CTAB]/[Cr^{6+}] = 1$ from pH 3 to 8, and S/M from 1 to 8 was evaluated for the rejections of Cr(VI) for pH 3.0, 6.5, and 8.0, respectively. The variations of micellar hydrodynamic diameters and electrokinetic potentials (zeta potential) were also evaluated for various concentrations of CTAB for Cr(VI) of 0 and 1.0 mM. Subsequently, the effects of operating pressure were evaluated as molar ratio $S/M = 3$, pH 3, and concentrated ratio of 95% for various pressures (30, 40, 60, and 80 psi). In the second stage, CE is an essential parameter for determining the optimal range of applicability of ED. CE is a measure of how effective ions are transported across the ion exchange membranes for a given applied current. According to Faraday's law, the CE calculated equation is derived as follows:

$$\mathrm{CE}(\%) = \frac{zFV\Delta C}{It} \times 100 \quad (1)$$

where z is the charge of ion, F is the Faraday constant (96,485 A s/mol), V is the volume of the electrolyte (dm^3), ΔC is the change in concentration (mol/dm^3), I is the current (A), and t is the operating time (s) [35,36].

Samples were taken every 15 min for analysis. Hexavalent chromium was measured colorimetrically according to the methods 3500-Cr listed in the 20th edition of the Standard Methods [37] using a UV–vis spectrophotometer (HACH Model DR-4000).

Table 1
The chemical analysis of the raw plastic electroplating wastewater

Wastewater properties	
pH	1.53
Cr(VI)	359
Cu^{2+}	72.9
Ni^{2+}	51.7

Note: Unit: mg/L except for pH which is unitless.

Concentration of CTAB was represented by total organic carbon (TOC) and determined by TOC analyzer (OI 1010, O.I. Corporation, USA). The micellar hydrodynamic diameter and electrokinetic potential (zeta potential) used Dymanic Light Scattering method and were determined by nanoparticle and zeta potential analyzer (SZ-100, HORIBA).

3. Results and discussion

3.1. Stage 1: effects of pH, molar ratio S/M, and operating pressure for MEUF

Fig. 2 showed a comparison of retention of chromium and CTAB as a function of pH for *S*/*M* of 1, since pH controls the adsorption capacity of micelle. The results showed that the retention of Cr(VI) was higher at low pH and increased from 54 to 89% due to the speciation of Cr(VI) since $HCrO_4^-$ was the predominant species below pH 6.8, and the CrO_4^{2-} was the favored species above pH 6.8 for Cr(VI) [2,18]. Furthermore, owing to CTAB dissociating into CTA^+ and Br^- in solution, only one mole CTA^+ was required to adsorb $HCrO_4^-$ as $pH < 6.8$; however, 2 mol CTA^+ was required to adsorb CrO_4^{2-} as $pH > 6.8$. Therefore, in limited surfactant concentration, the removal efficiencies of Cr(VI) and CTAB decreased as pH decreased. Besides, there was also a possibility of the complexion of CTA^+-OH^- together with the presence of $(CTA^+)_2$-CrO_4^{2-} to reduce the efficiency of CTA^+ on CrO_4^{2-} at higher pH. Thus, those CrO_4^{2-} were not adsorbed by CTA^+ and be able to pass through the membrane and induce a decrease of the rejection rate of CrO_4^{2-}.

Fig. 3 showed effects of pHs on retention of chromium for various molar ratios *S*/*M* for pressure of 30 psi. The retention of Cr(VI) increased with increasing the molar ratio *S*/*M*, and almost the same (98%) for different pHs as molar ratio *S*/*M* of 8. Since the retention of Cr(VI) was favored at lower pH range, the removal efficiency of chromium was not affected by pH, and the high removal efficiencies achieved at molar ratio *S*/*M* of 8 may be explained by forming larger micellar hydrodynamic diameter at higher molar ratio to offer enough CTA^+ formed micelles for CrO_4^{2-} adsorption. Pores clogging of membrane and the formation of gel layer may increase the rejection as well [21,38].

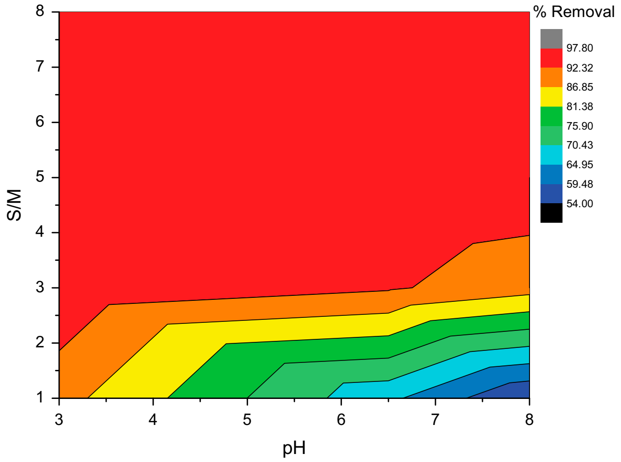

Fig. 3. Effects of pHs on retention of chromium for various molar ratios, *S*/*M* for a pressure of 30 psi.

The variation of zeta potential with various concentrations of CTAB with Cr(VI) of 0 and 1.0 mM is shown in Fig. 4. Since zeta potential was the degree of repulsion between adjacent charged particles/micelles in a dispersion solution, the zeta potential value can be related to the stability of colloidal dispersions,

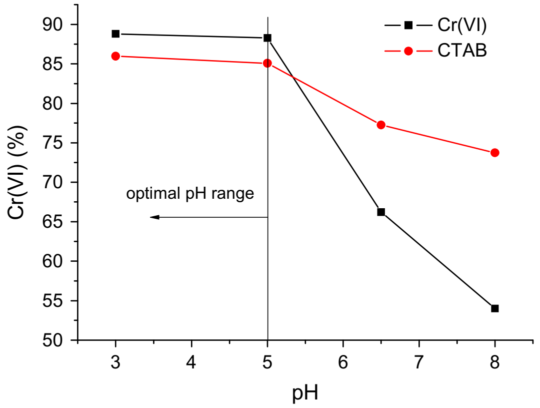

Fig. 2. Effects of pHs on retention of chromium and CTAB as molar ratio *S*/*M* = 1.

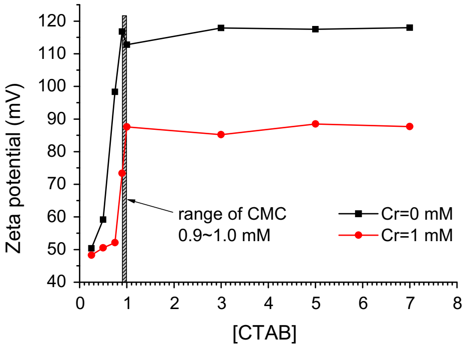

Fig. 4. A comparison of zeta potential as a function of concentration of CTAB, which ranges from 0.25 to 7.0 mM under two different concentrations of Cr(VI) 0 and 1.0 mM.

where a high zeta potential conferred stability for molecules (micelle) [39]. The results showed that, in the case of Cr(VI) of 1.0, all zeta potentials were lower than that without Cr(VI). That was due to the charge neutralization of CTA^+ with $CrO_4^{2-}/HCrO_4^-$ to reduce the electrical charge at the double layer. In addition, the concentrations of CTAB gradually increased for both experiments; however, the zeta potential presented plateau after CMC, indicating that micellization of CTAB and net charge reduction occurred when concentration exceeding CMC. Moreover, the constant values of zeta potential may infer that there were no Cr(VI) ions in the free state or full adsorption of the micelle. The micellar hydrodynamic diameter (D_h) was also measured to evaluate membrane performance, and D_{hs} gradually increased with the increase in the concentration of CTAB as Cr(VI) of 1.0 mM, as seen in Fig. 5. Moreover, the D_{hs} were ranged from 3.1 to 4.7 nm for CTAB exceeding 1.0 mM and was larger than the UF membrane (MWCO 8,000 Da) pore size of 2.9 nm, as calculated by steric hindrance pore model [40]. Therefore, the rejection of Cr(VI) in MEUF process gradually increased from 22.2 to 97.6% with increasing concentration of CTAB for pH 5, and a high rejection performance (>95%) was achieved as the concentration of CTAB exceeded 3.0 mM.

Fig. 6 presented the effect of operating pressures on retention of chromium and permeate flux as pH 3, $S/M = 3$, and $Cr(VI) = 1$ mM, and the results showed the removal efficiencies of Cr(VI) decreased with increasing operating pressure but the flux increased with increasing operating pressure. The optimal operating pressure ranged from 30 to 60 psi, considering the removal efficiencies of Cr(VI) and permeate flux. Moreover, visual observations were taken for membrane surfaces by scanning electron microscope (SEM). Fig. 7 showed the SEM of membrane surfaces of clean membrane and operating pressures of 30, 40, and 80 psi. High decomposition at higher operating pressure was observed on the membrane surface. Furthermore, the possibility of gel layer formation induced a decreasing of the membrane pore size and increased the performance of rejection by the sieving effect for UF membrane [41].

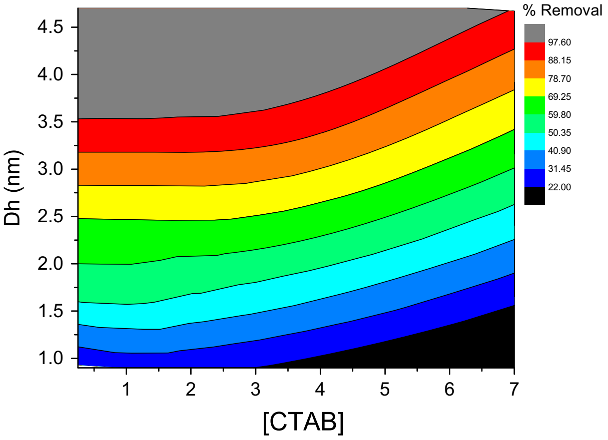

Fig. 5. The variation of rejection of Cr(VI) with micellar hydrodynamic diameter (D_h) as a function of concentration of CTAB, which ranges from 0.25 to 7.0 mM under the concentration of Cr(VI) 1.0 mM.

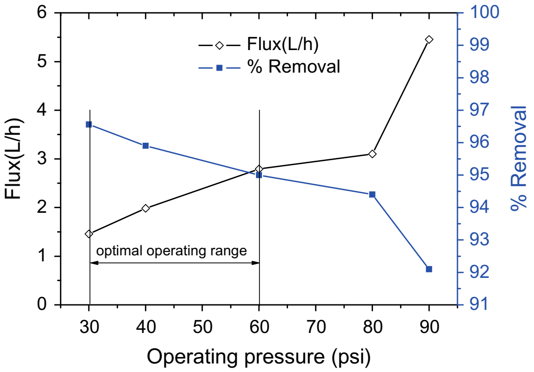

Fig. 6. Effect of operating pressures on retention of chromium and permeate flux as pH 3, $S/M = 3$, and $Cr(VI) = 1$ mM.

3.2. Stage 2: effects of current density on separation for ED

The solution of surfactant and chromate concentrated in the MEUF process was sent to ED process. In the second stage, ED module was used to separate the

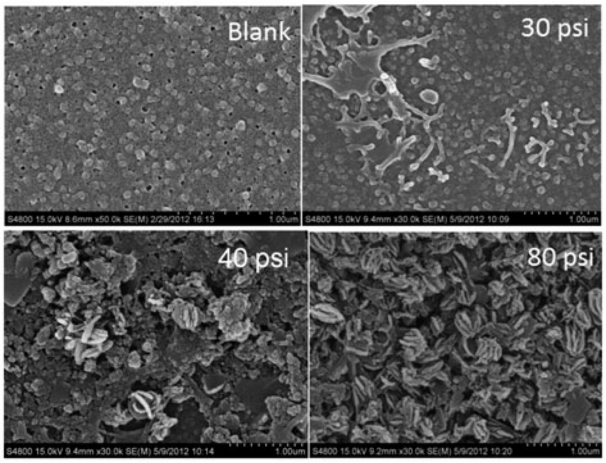

Fig. 7. SEM of membrane surfaces of clean membrane and operating pressures of 30, 40, and 80 psi.

chromium and CTA^+, and then to recover CTA^+ and chromate in the compartment of cathode and anode, respectively. In order to determine the optimal current density, four different current densities of 27.5, 42.4, 67.8, and 78.7% were designed, corresponding to 5, 10, 20, and 30 mA/cm^2, respectively, and the recovery efficiencies of Cr(VI) gradually increased with increasing current densities. In addition, an experiment was designed to assess the recovery efficiencies of Cr(VI) at the current density of 30 mA/cm^2 for different operating durations. The result showed that the recovery of Cr(VI) slightly increased with time, but reached a plateau after 480 min, resulting from the decreasing conductivity in the cathode compartment solution and slowed down the passage of the Cr(VI) in the dilute solution through the AEM membrane. Therefore, the recovery efficiency of Cr(VI) was achieved at 87.4% for 600 min, and current density over 20 mA/cm^2. The correlated tendency of operating time and current density vs. recovery of chromate is shown in Fig. 8.

Fig. 9 showed the correlation of chromate recovery on current density and CE at the operating time of 360 min. Apparently, higher current density achieved higher recovery efficiency of Cr(VI), but led to a decrease in CE, resulting from side reactions such as the evolution of O_2 and H_2 gases. The possibility of fouling on AEM also led to CE decline. Although, CE values were not high at higher current density, higher recovery of chromate was achieved at a longer operating time to recover the chromium and surfactant from the first stage.

3.3. Evaluation of the full-scale plastic plating wastewater by MEUF/ED

The experiment was applied to evaluate the full-scale plastic plating wastewater by the MEUF/ED process, and the results demonstrated that the removal efficiency of chromate by MEUF was higher exceeding 96% at operating parameters of pH 1.53, $\Delta P = 30$ psi, and $S/M = 2$ under concentration ratio of 95%. Thereafter, the concentrated mixture solution of chromate and surfactant was then withdrawn into the second stage of ED process, and the recovery of chromate was achieved at 85.3% at the current density of 20 mA/cm^2 and operating time of 360 min. This result demonstrated that the MEUF/ED process could be utilized for recovery of chromate in the wastewater.

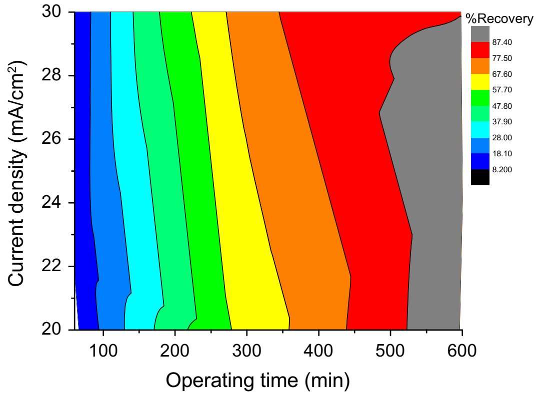

Fig. 8. The correlation of current density and CE vs. recovery of chromate.

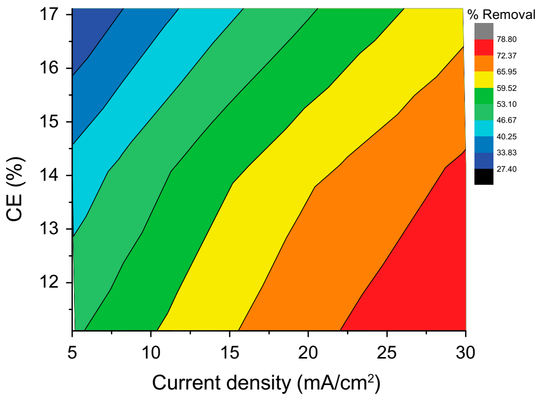

Fig. 9. The correlation of chromate recovery on current density and CE at the operating time of 360 min.

4. Conclusions

MEUF combined with ED process successfully recovered Cr(VI) at optimal operating parameters such as pH, molar ratio S/M, operating pressure, and current density. The results showed the removal efficiencies of chromate increased at lower pH and higher CTAB concentration. The micellar hydrodynamic diameters gradually increased with the concentration of CTAB as Cr(VI) of 1.0 mM, and ranged from 3.1 to 4.7 nm. The zeta potentials gradually increased with the concentration of CTAB, but reached plateau for concentration higher than CMC. The experimental results indicated that the removal of Cr(VI) by MEUF exceeded 96% for operating parameters of pH 1.53, $\Delta P = 30$ psi, and $S/M = 2$. For ED process, the effects of current density and CE were evaluated as the concentration ratio of 95%, and the chromium was able to pass through AEM to recover at the anode side of the ED process up to 85.3% at current density of 20 mA/cm^2 and operating time of 360 min. Overall, chromium from the plastic plating wastewater could be successfully recovered by the proposed MEUF/ED process.

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