



Effects of co-existed anions on retention characteristics of chromate by MEUF

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ABSTRACT

Micellar enhanced ultrafiltration (MEUF) process was proposed to evaluate effects of anions (nitrate and sulfate) in single- and multi-component aqueous solutions for chromate removal with cationic surfactant (cetyltrimethylammonium bromide, CTAB) under constant transmembrane pressure drop (40 psi), temperature (25°C), and surfactant/chromium (S/M) molar ratio of 3 (S/M = 3 mM/1 mM). First of all, in the single-component anion (only one anion was used) solutions, the order of removal efficiency was: chromate 99.9% > sulfate 96.1% > nitrate 94.0%. These results indicate that chromate preferentially binds on the CTAB micelles but sulfate and nitrate bind less. On the other hand, in the multi-component anions (chromium together with co-existed anions) solution, the removal efficiency of chromate decreased (99.4–98.4%) with the increase of sulfate and nitrate. The retention of surfactant was enhanced by the addition of electrolytes into solution due to the counterion dissolved from electrolyte reduced the repulsive electrostatic force between the head groups of cationic surfactant molecule. In addition, the effect of temperature in multi-component on MEUF was evaluated simultaneously.

Keywords: Co-existed anions; Chromate; Micellar enhanced ultrafiltration; Cationic surfactant

1. Introduction

Chromate is a potential carcinogen with effluent discharge standard of 0.5 mg/L as Cr in Taiwan and is widely used in the electroplating industry. The traditional procedures of chromium reduction in the electroplating industry are to use a reducing agent such

as sodium bisulfite (HNaSO₃) to reduce Cr(VI) to Cr(III) [1,2]. Either Cr₂O_{3(s)} or Cr(OH)_{3(s)} is precipitated by adjusting pH to around 8. Coagulant, such as ferric salt, is also used to enhance the precipitation of Cr₂O_{3(s)} or Cr(OH)_{3(s)}. Since the effluent chromate concentration in the electroplating industry could be as high as several hundred mg/L as Cr⁶⁺, significant amounts of sodium bisulfite and coagulant are needed in these procedures [3–6]. Therefore, recovery of

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chromium is more feasible and should be considered. To recover chromium, micellar enhanced ultrafiltration (MEUF) with surfactant process is suggested by forming micelle with surfactant over critical micelle concentration (CMC) in the solution then and to entrap the metal–micelle complexes by UF membranes.

Surfactant monomer dissociates in aqueous solutions into hydrophilic head group and hydrophobic tail [7,8]. There are two competing forces in the formation of micelles, including the opening of hydrocarbon chains from water favors aggregation but electrostatic repulsions between the ionic head groups opposes aggregation. While at certainly higher concentration, i.e. CMC, the amphiphilic molecules self-assemble into aggregates or microstructures known as micelles. Counterions stabilize ionic surfactant micelles by binding to the micelles and reducing the electrostatic repulsions. The counterion accumulations in the vicinity of a micelle due to strong electrostatic coupling between the charged particle and counterions call counterion condensation [9–14].

The counterion condensation is the typical feature of ionic micelle, however, as CMC is exceeded, the free counterion declines with increasing the surfactant concentration [15]. The counterion condensation on the micelle decreases as the hydrated size of the counterion increases or the size of the micelle decreases [16]. Micelle formation leads to much stronger electrostatic attraction between the counterion and the highly charged sphere in comparison to the attraction of single surfactant ion with its counterion. Hsiao et al. [15] has investigated counterion condensation and release in micellar solutions, and found as addition of monovalent salts releasing counterion to the bulk, indicating that the specific ion effect plays an important role to the electrostatic attraction of micelles and counterions.

Since the salts of nitrate and sulfate are commonly used in electroplating industry together with chromium, and both have higher charges and hydrated sizes than that of Br^- , affecting the counterion condense phenomenon, the co-existence of these two ions were evaluated in a single- and multi-component aqueous solution for chromate removal by MEUF with cationic surfactant (cetyltrimethylammonium bromide [CTAB]) in this study. Therefore, the objectives of this study are as follows: (i) to evaluate the influence of three anions (chromate, nitrate, and sulfate) on MEUF process (ii) to determine Cr(VI) reduction on the presence of multi-components (sulfate, nitrate) for MEUF process and (iii) to evaluate the effect of temperature on MEUF.

2. Methods and materials

The scheme of the experimental setup was shown in Fig. 1. The MEUF system contained a cross-flow ultrafiltration unit (tangential flow filtration unit). The UF module consisted of batch tank and UF unit, in which UF unit was fabricated with stainless outside crust and plastic membrane support. The UF membrane was thin film composite membrane (type: GM) manufactured by GE-Desal which with operating pH ranged 2–11, maximum temperature 50°C , maximum operating pressure 200 psi, and nominal MWCO 8,000 Da. All chemicals used were of analytical-grade reagents; potassium dichromate (K_2CrO_7), potassium nitrate (KNO_3), potassium sulfate (K_2SO_4), and CTAB ($\text{C}_{19}\text{H}_{42}\text{BrN}$) were obtained from Merck and Sigma Aldrich.

Firstly, the experiment evaluated the effects of anions (hexavalent chromium, nitrate and sulfate) in the single-component anion (only one anion was used) solutions on MEUF process. Secondly, removal of Cr(VI) was determined in the presence of multi-components (sulfate, nitrate) on MEUF process. Finally, the effect of different temperature against the relative flux and retention efficiency of chromate for MEUF process were evaluated. Before experiment, the basic normalized membrane flux had to be determined by deionized water. The relative flux ratio was defined as:

$$\text{Relative flux ratio } (J_r) = \frac{J_V}{J_{V0}} \quad (1)$$

where J_V is permeate flux and J_{V0} is permeate flux of deionized water. Subsequently, sample (anions/surfactant) was prepared and the concentrate was recirculated into the batch tank. In single-component anion, the operating parameters were CTAB of 3 mM,

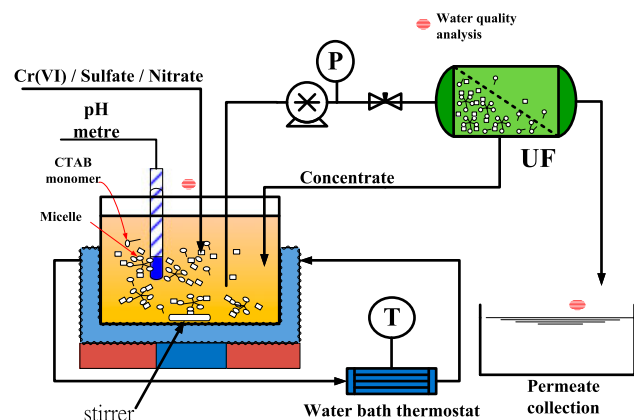


Fig. 1. Schematic diagram of MEUF process.

temperature 25°C, operating pressure 40 psi, and concentrated ratio (V_c) of 90% with anion of 1 mM. In order to evaluate the interaction of water matrix on Cr(VI) reduction for MEUF, the experiments were designed at different composites (multi-component) including sulfate and nitrate as [CTAB]:[Cr(VI)]=3:1 and 6:1, respectively.

Hexavalent chromium was measured colorimetrically according to the methods 3500-Cr listed in the 20th edition of the Standard Methods [17] using a UV-vis spectrophotometer (HACH Model DR-4000). Concentration of CTAB was represented by total organic carbon (TOC) and determined by TOC analyzer (OI 1010, O.I. Corporation, USA). The anions (SO_4^{2-} , NO_3^- and Br^-) were determined by ion chromatography (Dionex ICS-90).

3. Results and discussion

3.1. Comparison of single component on MEUF

Fig. 2 presented the relative flux vs. concentrated CTAB as initial CTAB = 3 mM, temperature 25°C, operating pressure 40 psi, and V_c of 90%. In Fig. 2, when the feed solution was without anions, the initial relative flux ratio was 0.81, then decreased to 0.71 with increasing V_c to 90% (concentration of 30 mM CTA^+). The result demonstrates the relative flux ratio is rapidly declined initially because of membrane fouling and concentration polarization due to gel layer formation from the deposition of surfactant micelles on the surface of membrane. Thereafter, the relative flux becomes steady since majority of the feed flow travels tangentially across the surface of the filter to partially

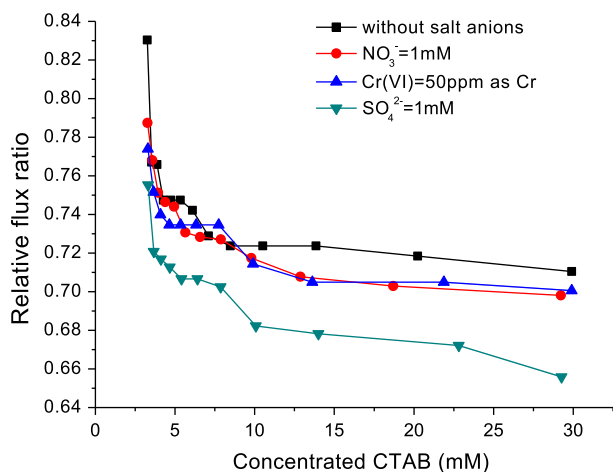


Fig. 2. The relative flux vs. concentrated CTAB as initial CTAB = 3 mM, temperature 25°C, operating pressure 40 psi and V_c of 90%.

carry away the gel layer to become steady state to achieve longer operating time [18,19]. In addition, the gel layer formation caused the flux decay but helped the surfactant and ions being trapped [20,21]. Furthermore, for individual anions, the results show the relative flux ratios are all lower than that of the single component of CTAB, results from the morphological characteristics of micellar solutions is strongly influenced by both the nature and concentration of added counterions [22], thus the counterions be attracted onto the surface of the micelle and reduction of the CMC also changes the shape and size of micelle [23]. The Cr(VI) was monovalent in MEUF system as [CTAB]:[Cr(VI)] = 3:1 since the predominant species was HCrO_4^- below pH 6.5 and CrO_4^{2-} over pH 6.5. Therefore, the formation of ionic surfactant micelle was affected by the added electrolyte and lower relative flux ratio was observed for high valence of anion.

Fig. 3 showed the retention of CTA^+ for individual component of hexavalent chromium, nitrate, sulfate, CTAB by MEUF system. Comparing these results, at initial operating phase (1–4 h), the retention efficiencies of CTA^+ fluctuated (92.5–95.5%). Without adding any electrolyte, the retention efficiency of CTA^+ was lowest due to less gel layer formation, but the addition of electrolyte can promote the aggregation of surfactant micellization to form gel layer formation and retention efficiency. Finally, when the concentrated ratio achieved of 90% (concentration of 30 mM CTA^+), the retention efficiencies of CTA^+ together with individual hexavalent chromium, nitrate, and sulfate all reached 99%. Therefore, the addition of hexavalent chromium, nitrate, and sulfate promoted the retention efficiency of CTA^+ for MEUF and the effect of high

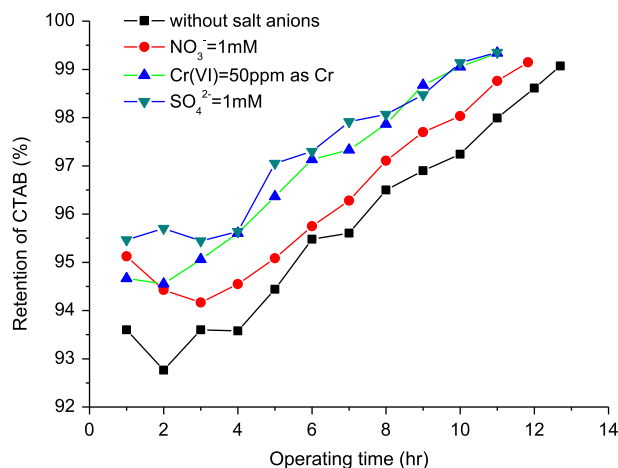


Fig. 3. The variation of retention of CTAB vs. operating time as initial CTAB = 3 mM, temperature 25°C, operating pressure 40 psi and V_c of 90%.

valent species (divalent sulfate) was larger than the monovalent (nitrate) on CTA^+ removal.

Fig. 4 showed the individual retention of nitrate, sulfate, and hexavalent chromium in CTAB surfactant solution by MEUF process. The retention efficiency of hexavalent chromium achieved was 99.9%, and the retention efficiencies of nitrate, sulfate were gradually increased to 94.0 and 96.0% at V_c of 90%, respectively. Although the sulfate was capable of promoting micellization [23], the more easily hydrated anions (e.g. SO_4^{2-}) were less likely to close interface of hydrophilic end for micellar solution [24] to induce the retention efficiency less than that of chromate.

3.2. Comparison of multi-component on MEUF

Presence of multi-components on Cr(VI) reduction and the interaction of water matrix for MEUF are discussed in this section. There were four different influent synthetic wastewaters vs. operating time for CTAB with anions (chromate, sulfate, and nitrate) in MEUF process. Fig. 5 showed the relative flux ratios gradually decreased with operating time and the increasing concentrations of anions (sulfate and nitrate) and CTAB. Shorter operating time was observed with the V_c up to 90% as the concentrations of anions and CTAB were lower. Overall, even though the concentrations of anions and CTAB slightly influenced the relative flux ratio since the samples contained higher concentration of anions at V_c of 90% with micelles forming gel layer on the surface of UF membrane, the relative flux ratios ranged from 0.62 to 0.77 demonstrated excellent performance for MEUF system.

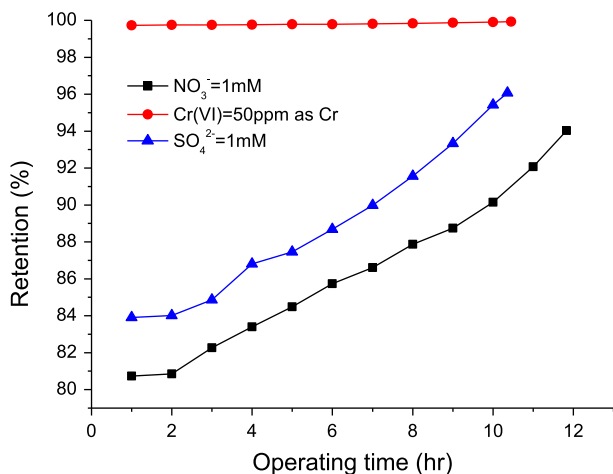


Fig. 4. The variation of retention of salt anions vs. operating time in single-component as initial CTAB = 3 mM, temperature 25 °C, operating pressure 40 psi and V_c of 90%.

Fig. 5(b) showed the retention efficiencies of bromide gradually increased with operating time and the highest retention efficiency was at CTAB of 6 mM, Cr(VI) of 1 mM, NO_3^- of 0.75 mM and SO_4^{2-} of 0.75 mM at V_c up to 90%. However, at CTAB of 3 mM, Cr(VI) of 1 mM, NO_3^- of 0.25 mM and SO_4^{2-} of 0.25 mM, shorter operating time was observed with retention efficiency of 76.1%. Fig. 5(c) showed the retention efficiencies of chromate gradually increased with operating time and the highest retention efficiency of 99.4% was observed at CTAB of 3 mM, Cr(VI) of 1 mM, NO_3^- of 0.25 mM and SO_4^{2-} of 0.25 mM for V_c of 90%, with shorter operating time. Fig. 5(d) showed the retention efficiencies of CTAB fluctuated and gradually increased with operating time and the highest retention efficiency of 74.3% occurred at CTAB of 3 mM, Cr(VI) of 1 mM, NO_3^- of 0.25 mM and SO_4^{2-} of 0.25 mM at V_c of 90% with shorter operating time. Fig. 5(e) showed the retention efficiencies of nitrate gradually increased with operating time and the highest retention efficiency was observed at CTAB of 6 mM, Cr(VI) of 1 mM, NO_3^- of 0.75 mM and SO_4^{2-} of 0.75 mM for V_c of 90%. However, for CTAB of 3 mM, Cr(VI) of 1 mM, NO_3^- of 0.25 mM and SO_4^{2-} of 0.25 mM, retention efficiency of only 78.9% was achieved with short operating time. Fig. 5(f) showed the retention efficiencies of sulfate gradually increased with operating time and the highest retention efficiency of 94.4% was observed as CTAB of 3 mM, Cr(VI) of 1 mM, NO_3^- of 0.25 mM and SO_4^{2-} of 0.25 mM at V_c of 90% with shorter operating time.

From Fig. 5(b)–(f), the retention efficiencies of hexavalent chromium of 99.4%, nitrate of 78.7%, sulfate of 94.4%, CTAB of 74.3%, and bromide of 72.6% was corresponded with the presence of CTAB of 3 mM, Cr(VI) of 1 mM, NO_3^- of 0.25 mM and SO_4^{2-} of 0.25 mM, and became hexavalent chromium of 98.4%, nitrate of 80.4%, sulfate of 90.1%, CTAB of 63.9%, and bromide of 75.8% as CTAB of 6 mM, Cr(VI) of 1 mM, NO_3^- of 1 mM and SO_4^{2-} of 1 mM. The retention efficiencies of hexavalent chromium, sulfate, and CTAB dropped by 1.0, 4.3, and 10.4%, but the nitrate and bromide were increased by 1.7 and 3.2%, respectively. Theoretically, the addition of ionic surfactant to electrolyte would decrease CMC due to the screening effect to promote formation of micelle and elevate adsorption capacity of anions. Therefore, the decrease of specific surface area of micelle with increasing solution volume concentrated ratio (V_c) up to 90% was observed to decrease adsorption capacity of anions.

3.3. Effect of temperature on MEUF

Effect of temperature on MEUF was studied since high temperature could occur in the wastewater

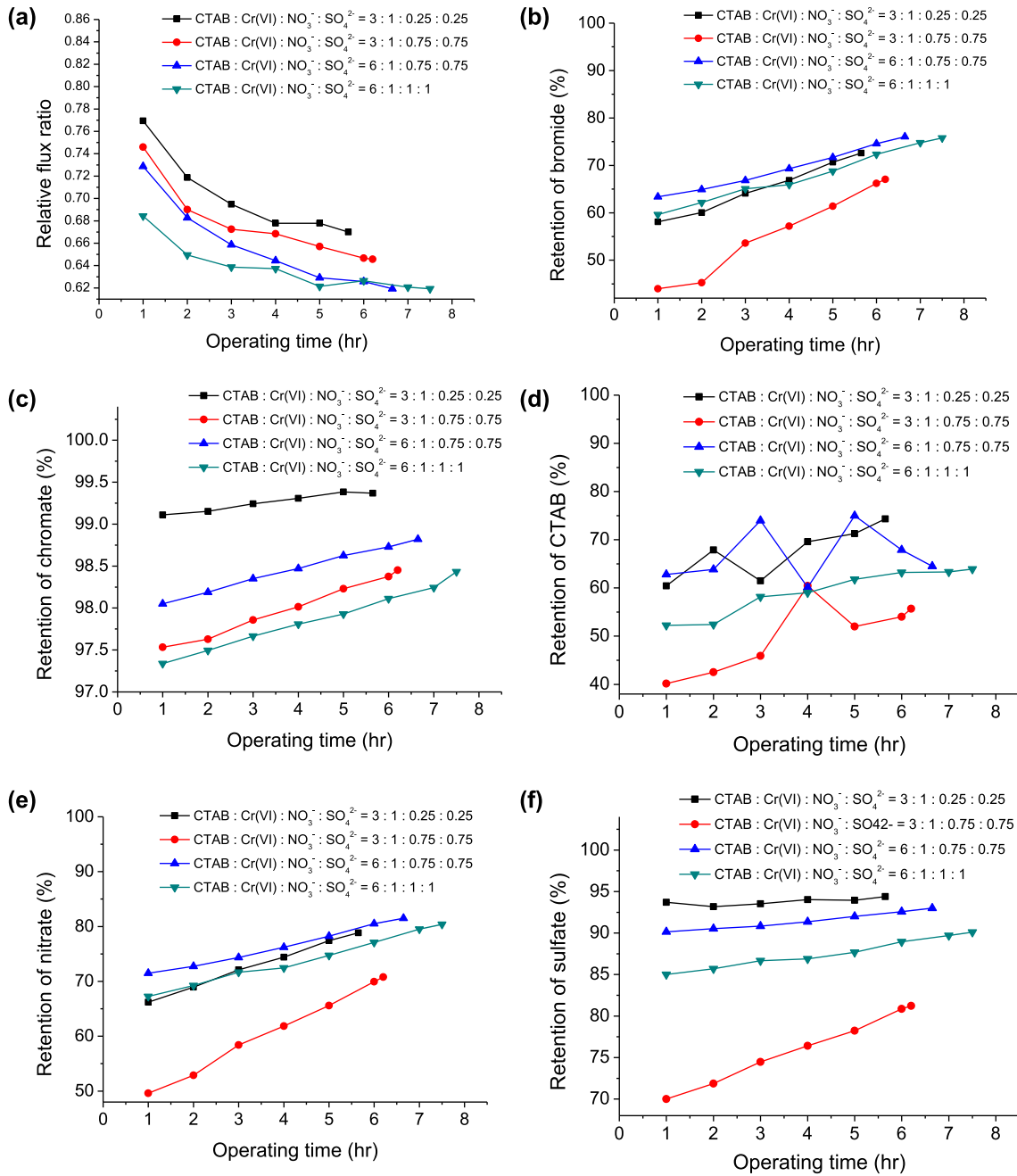


Fig. 5. The variation of relative flux vs. operating time in multi-component as initial CTAB=3, 6 mM, temperature 25°C, operating pressure 40 psi and V_c of 90%.

treatment process due to the weather or the heat generated by the operation unit (e.g. a pressurized pump, etc.). Since the transition in CTAB solution around the Krafft temperature, T_K , could occur and alter many physical properties of the surfactant solution such as the solution surface tension, the osmotic pressure or the conductivity, and the reported Krafft temperature values of T_K for CTAB in water vary considerably

from 20 to 25°C [25–29], this study was carried out in four different temperatures to evaluate the variations of flux and chromate retention efficiencies on MEUF. Fig. 6 showed the relative flux ratio and retention efficiency of chromate vs. operating time for four different temperatures (25, 30, 35, and 40°C). From Fig. 6(a) when the temperature increased from 25 to 30°C, the initial relative flux ratios increased from 0.68 to 0.84,

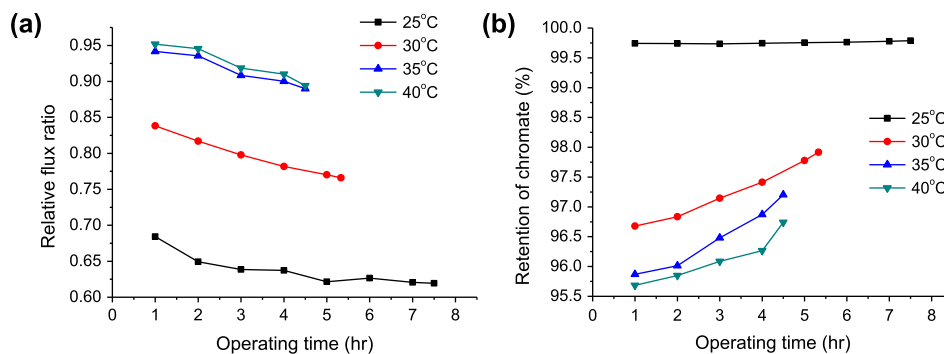


Fig. 6. The relative flux ratio and SO_4^{2-} retention performance vs. operating time for different temperatures as initial CTAB = 6 mM, Cr(VI) = 50 ppm, $\text{NO}_3^- = 1$ mM, $\text{SO}_4^{2-} = 1$ mM, operating pressure 40 psi and volume concentrated ratio of 50%.

and when the temperature reached 35 and 40°C, the initial relative flux ratios reached 0.94 and 0.95, respectively, indicating the relative flux ratios was influenced by the temperature on MEUF system, since resulted from the influent flux was inversely proportional to liquid viscosities with 0.975, 0.893, 0.784, and 0.725 mPaS for temperatures of 25, 30, 35, and 40°C, respectively. Fig. 6(b) showed retention efficiencies of chromate vs. operating time, and the initial removal efficiencies were: 99.7% (at 25°C), 96.7% (at 30°C), 95.9% (at 35°C) and 95.7% (at 40°C). Chromate removal decreased with increasing temperature, since high temperature makes micelle formation difficult and thermal disturbance also affected the adsorption of ions onto the surface of micelles. In addition, when the concentration ratio achieved 50%, the removal efficiencies of chromate were increased to 97.9% (at 30°C), 97.2% (at 35°C), 96.7% (at 40°C), except for higher removal at 25°C. From aforementioned results, the temperature above Krafft point of CTAB not only decreased the influent liquid viscosity with increasing temperature, but also increased the CMC of CTAB [30] and hindered the hydrophobic tail aggregates to form the micelle due to the reduction of hydrophobicity of tail group for surfactant decreased by increasing temperature.

4. Conclusion

MEUF process for removal of co-existence anions (nitrate and sulfate) and chromium were successfully evaluated in a single- and multi-component aqueous solution. First of all, in the single-component anion (only one anion was used) solutions, the order of removal efficiency was: chromate 99.9% > sulfate 96.1% > nitrate 94.0%. These results indicate that chromate preferentially binds on the CTAB micelles but sulfate and nitrate bind less. On the other hand, in the multi-

component anions (chromium together with co-existed anions) solution, the removal efficiency of chromate decreased (99.4–98.4%) with the increase of sulfate and nitrate. The retention of surfactant was enhanced by the addition of electrolytes into solution due to the counterion dissolved from electrolyte reduced the repulsive electrostatic force between the head groups of cationic surfactant molecule. In addition, the relative flux ratios were obviously increased with increase in temperature above the Krafft point temperature.

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