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Removal of hexavalent chromium by mixed micelles of cetyl trimethyl ammonium bromide using micellar enhanced ultrafiltration

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Abstract

Our study includes removal of Cr(VI) by using mixed surfactants (Non-ionic: Cationic). A common cationic surfactant Cetyl trimethyl ammonium bromide (CTAB) with two different nonionic surfactant Triton X-100 and Tween-80 were used to remove Cr(VI) by micellar enhanced ultrafiltration technique. The effect of parameters such as initial feed metal ion concentration, pH and filtration time on % Rejection of Cr(VI) were analysed. Low pH favoured the removal of Cr(VI) and exhibited maximum rejection percentage (>90%) at pH 2 for both systems. The optimum feed Cr(VI) ion concentration and filtration time can be suggested as 20 mgL⁻¹ and 60 minutes respectively. It was found that CTAB+Triton X-100 and CTAB+Tween-80 gave Cr(VI) percentage removal upto 97% and 93% respectively. The adsorption data were studied using Langmuir, Freundlich and Dubinin-Radushkevick (DRK) adsorption Isotherms. The data fitted well in Langmuir model (R²=0.99). From DRK isotherm, mean free energy E_{DR}, was much lower than 8 kJ.mol⁻¹ suggesting the Cr(VI) adsorption onto mixed micelles is physisorption. Thermodynamic parameters ΔG°, ΔH° and ΔS° were evaluated revealing the process to be spontaneous and exothermic accompanied by increased randomness at the interface.

Keywords: Cr (VI), Mixed surfactant system, Optimization, Membrane separation, Adsorption Isotherms

1. Introduction

Clean water accounts for only 1% of the total available water for drinking. Increasing urbanization, industrialization and several anthropogenic activities have caused serious damage to aquifers. Several inorganic and organic contaminants contribute to the water pollution. Heavy metals are major class of pollutants that are responsible for disturbing the ecological balance due to its bioaccumulation and biomagnifications properties.

Chromium among all heavy metals is discharged in environmental waters typically from industrial pollution sources, including tanning factories, steel works, wood preservation and artificial fertilizers [1]. Chromium exhibits a vast range of oxidation state in aqueous solution. However the most prevalent among these are Cr (III) and Cr (VI). Hexavalent chromium is known to have 100-fold more toxicity than trivalent chromium, for both acute and chronic exposures because of its high water solubility and mobility, as well as easy reduction [2]. Human toxicity of Cr (VI) causes lung cancer, liver, kidney and gastric damage, and epidermal irritation and sensitization [3]. It is also potentially carcinogenic and therefore, requires perpetual elimination in order to obtain safe drinking water. There are several techniques employed in Chromium removal such as precipitation [4], coagulation [5], adsorption [6] and membrane separation. But not all techniques are efficient enough at higher concentrations of Cr (VI). Surfactants have known to be dominating over the food industry in 20th century. They are extensively used in household products, detergents, oil recovery, paint technology, flotation, and water treatment. It has been observed that cationic surfactants such as cetyl trimethyl ammonium bromide (CTAB) have also known to remove Hexavalent Chromium [7]. However, in the recent past use of non-ionic surfactants as a template with ionic surfactants have known to expedite much attention in the removal of contaminants at lower cost and post treatment sludge production. Micellar enhanced ultrafiltration is a technique which removes heavy metal even at higher concentrations and also with an efficiency of almost ~97%. This technique involves trapping of heavy metal bound by micelle using a membrane having a pore size less than the size of formed micelles [8].

2. Materials and Methods

2.1 Chemicals and reagents

All reagents were of analytical reagent grade and were used as supplied. Stock solutions of Cr (VI) were prepared by direct dissolution of proper amounts of $K_2Cr_2O_7$ salt. All the solutions were further diluted as per the requirement. Cetyl trimethyl ammonium bromide (CTAB), Triton X-100, Tween 80, sodium hydroxide and hydrochloric acid were obtained from Merck. pH of the solutions were maintained by using 0.1M NaOH and 0.1M HCl.

2.2. Optimization of metal ion adsorption

The UF experiments were carried out in a batch stirred cell (Amicon 8400, Millipore) and the aqueous mixture was stirred at a constant speed of 160 rpm. pH 2 was maintained throughout the experiment. A peristaltic pump was used to feed the solution to ultrafiltration cell. The feed solution of 300 ml containing the mixture of aqueous metal solution of concentration 20 mg/L and mixed surfactant(non-ionic: ionic) in the molar ratio 0.1 at fixed 1.2mM concentration of CTAB, Triton X-100 and Tween-80 were placed in feed tank. The transmembrane pressure (TMP) was maintained at 100 kPa using high pressure nitrogen gas, and the temperature at 25 ± 1 °C. After each run, the membrane had to be thoroughly washed to recover its permeability. After each run, membrane was cleaned first, with tap water without pressure was used to rinse out the residual synthetic wastewater for 20 min. Then, it was washed with 0.1 M NaOH, and 0.1 M HCl at 30 kPa for 20 min, respectively [9].

Membrane

The UF membrane disc used are manufactured by Millipore (Biomax polyethersulfone, PBGC07610), which have a nominal molecular weight cut-off (MWCO) of 10 kDa and an effective filtration area of 41.8 cm² and filter diameter 76 mm was used. The membrane was hydrophobic in nature.

Analysis

The residual concentration of Cr (VI) in the solution was analysed by Perkin Elmer Aanalyst 400. The rejection percentage, %R_{Cr} analysis for Cr (VI) in the retentate was made by the equation (1) and (2) respectively.

$$\text{Percentage Rejection, \%R}_{Cr} = \frac{C_{iCr} - C_{fCr}}{C_{iCr}} \times 100 \quad (1)$$

where C_{iCr} and C_{fCr} are the initial and final values of corresponding concentration in feed and permeate respectively.

The permeate and feed flux were calculated using the following equation:

$$J_p = \frac{V_p}{S \cdot t} \quad (2)$$

$$J_f = \frac{V_f}{S \cdot t} \quad (3)$$

Where Q_p , V_p and Q_f , V_f are flux and volume of permeate and feed respectively, S is effective membrane area of membrane and t is time.

The amount of Cr (VI) adsorbed at equilibrium, r (mg/g), the adsorption capacity was evaluated by the following equation (4):

$$r = \frac{C_{iM} \times Q_f - C_{fM} \times Q_p}{C_{iSDBS} \times Q_f - C_{fSDBS} \times Q_p} \quad (4)$$

where Q_i and Q_p are feed and permeate flux (L/m².h). The adsorption experiment was carried out individually for Cr (VI) ion separately. The data for adsorption experiment were replicated three times and the average results were reported.

2.3. Isotherm study

Adsorption Isotherms were employed at different metal ion concentration 20 mg/L to 100 mg/L at agitation speed 160 rpm and temperature 25°C at pH 7.5. Two commonly used empirical adsorption models, Freundlich and Langmuir which correspond to heterogeneous and homogeneous adsorbent surfaces respectively, were employed in this study. To analyse the apparent energy of adsorption DKR Isotherm was used. We assume that all of the surfactant micelles are rejected by membrane. The linearised form of Langmuir, Freundlich and DKR adsorption isotherm are given in equation 5,6,7 and 8 respectively.

$$C_{eq}/r = 1/r_{max} \cdot b + C_{eq}/r_{max} \quad (5)$$

$$\log r_e = \log K_F + 1/n \log C_e \quad (6)$$

$$\ln r_e = \ln r_D - \beta e^2 \quad (7)$$

$$e = R \times \ln \frac{1+C_e}{C_e} \quad (8)$$

where r_e (mg g⁻¹) is the amount of adsorbates adsorbed per gram of adsorbents at equilibrium; C_e (mg L⁻¹) is the equilibrium concentration of adsorbates in the solution; K_F , is a Freundlich isotherm constant for the system and value of $n > 1$, indicative of the degree of nonlinearity between solution concentration and adsorption; r_{max} (mg g⁻¹) is the maximum monolayer adsorption capacity and K_L (L mg⁻¹) is the constant related to the free energy of adsorption and r_D is the calculated adsorption capacity obtained from DKR isotherm. The mean free energy From DKR isotherm is obtained from equation (9)

$$E_{DR} = 1/\sqrt{2\beta} \quad (9)$$

2.4. Thermodynamic study

Thermodynamics of the systems were studied at 25°C, 30°C, 35°C, 40°C, 45°C. For calculating thermodynamic parameters, following equations were followed:

$$K_c = \frac{C_{Ae}}{C_e} \quad (10)$$

$$\Delta G^0 = -RT \ln K_c \quad (11)$$

where C_e is the equilibrium concentration in solution in mg/L and C_{Ae} is the equilibrium concentration on the sorbent in mg/L and K_c is the equilibrium constant. Thermokinetic parameters such as preexponential factor A, and Activation energy E_{Act} were calculated using vant hoff plots derived from the following equation

$$\ln K_c = \ln A - \frac{E_{Act}}{RT} \quad (12)$$

where T is the temperature (K) and R is the gas constant having (=8.314 kJ/mol K). Energy of Activation E_{Act} , and pre-exponential factor obtained from the vant hoff plots of $\ln K$ vs $1/T$.

3. Result and Discussion

3.1. Influence of feed Fe²⁺ concentration

This experiment was performed to study the effect of initial feed concentrations of Cr (VI) on percentage rejection of Cr (VI). Temperature, pH and agitation speed were kept constant and initial feed Cr (VI) concentration was varied from 20 mg/L to 100 mg/L. It was found that at 20 mg/L of feed metal ion concentration, maximum rejection percentage of Cr(VI) was 97.57% and 93.05% in the presence of CTAB +Triton X-100 and CTAB +Tween-80 respectively. It was observed that percentage rejection decreased from 97.57% to 82.88% and 93.05% to 80.96% when treated with CTAB +Triton X-100 and CTAB +Tween-80 respectively on increasing the feed metal ion concentration from 20 to 100 mg /L (Fig. 1). The ratio of number of moles of metal ions to the binding sites of

adsorbent is large at optimum concentration (20 mg/L), so adsorption takes place without any interruption. When Cr (VI) feed concentration increase, the zeta potential of the SDBS micelles increases as a result of decrease in surface charge density. With the continual rise of Cr (VI) in the feed

solution, the concentration of unbound Cr (VI) also increases. Therefore, the reduction in removal percentage at higher feed Cr (VI) concentration might be due to the lack of available binding sites, which in turn can be explained by the increase in zeta potential^[10].

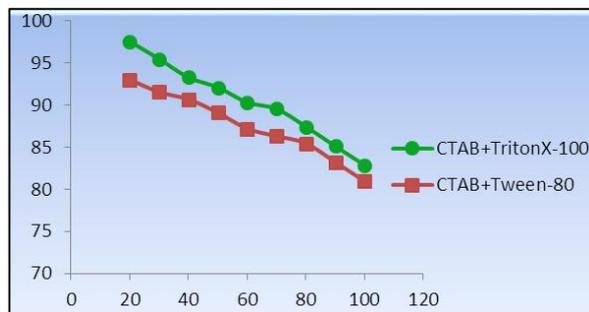


Fig 1: Influence of feed metal ion concentration on the percentage rejection of Cr (VI) with CTAB+Triton X-100 and CTAB+Tween-80 at constant temperature =25°C, pH=2, shaking rate=160 rpm and filtration time = 60 minutes

3.2. Influence of pH on the removal of Cr (VI)

pH of the solution posed significant effect on the percentage rejection of Cr(VI) because of metal speciation characteristics of Cr(VI) in aqueous solution. The optimum pH can be suggested as 4 for Cr(VI) adsorption on the mixed micelles of CTAB+Triton X-100 and CTAB+Tween-80. The percentage rejection of Cr(VI) decreased from 97.17% to 79.48% and 93.51% to 76.53% by CTAB +Triton X-100 and CTAB +Tween-80 micelles respectively on increasing the pH from 2 to 10 illustrated in Figure 2. Cr (VI) exists in various oxoanionic forms in aqueous solution depending upon the solution pH such as HCrO_4^- , $\text{Cr}_2\text{O}_7^{2-}$, CrO_4^{2-} . The stability of these forms is sternly reliant on the pH of the system^[11]. It has been suggested that at low pH the molecular form is predominantly adsorbed species, whereas at higher pH values the ionized form is preferentially adsorbed. The following reaction mechanism for adsorption of Cr (VI) at different pH is proposed by Babu & Gupta^[12].

- (1) $\text{H}_2\text{CrO}_4 \leftrightarrow \text{H}^+ + \text{HCrO}_4^-$
- (2) $\text{HCrO}_4^- \leftrightarrow \text{H}^+ + \text{CrO}_4^{2-}$
- (3) $2\text{HCrO}_4^- \leftrightarrow \text{Cr}_2\text{O}_7^{2-} + \text{H}_2\text{O}$

According to Muhammad *et al.* HCrO_4^- is the dominant and ionic form of Hexavalent Chromium between pH 2 and 4^[13]. A considerably high rate of adsorption up to pH 4 may be accounted on the basis of the fact that at low pH the species (HCrO_4^-) gets adsorbed as the CTAB surface which is positively charged. This could be because at low pH Cr (VI) exist as Cr^{3+} and can therefore be better adsorbed by the $(\text{CH}_3)_3\text{N}^+$ functional group present on the CTAB. Since functional moiety $(\text{CH}_3)_3\text{N}^+$ in CTAB possesses positive charge on a highly electronegative atom Nitrogen, therefore it is a highly electron attracting functional group. As a result of which it exerts a tremendous electrostatic pull towards HCrO_4^-

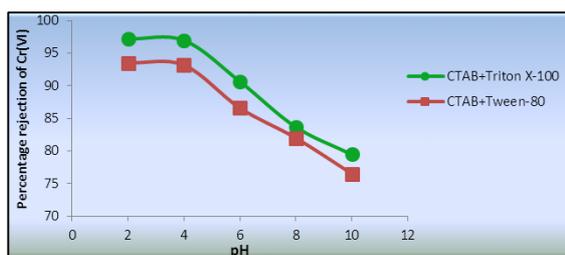


Fig 2: Influence of pH on the percentage rejection of Cr (VI) with CTAB+Triton X-100 and CTAB+Tween-80 at constant temperature =25°C, shaking rate=160 rpm, filtration time = 60 minute and initial feed Cr (VI) ion concentration =20 mgL-1.

3.3. Influence of filtration time

The study of feed flow rate at shaking rate 160 rpm, feed metal ion concentration 20 mg/L was made and samples were collected from 10 to 100 minutes with varying interval of 10 minutes, TMP and permeate flux were noted. Permeate flux seemed to decrease slightly and TMP increases with the increase in run time as there was negligible concentration polarisation. Maximum % Rejection of Cr(VI) was found to be 96.95% and 92.89% with CTAB +Triton X-100 and CTAB + Tween-80 respectively was observed at 60th minute which remained almost constant thereafter as shown in Figure 3. At this time, system tends to reach equilibrium. After reaching equilibrium, a noticeable increase in concentration polarisation causes increase in TMP which forcibly transfers micelles through the membrane pores. Hence, a reduction in permeate flux was noted, indicating membrane fouling due to concentration polarization^[14].

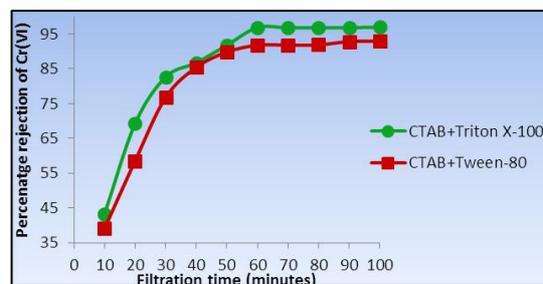


Fig 3: Influence of filtration time on the percentage rejection of Cr (VI) with CTAB +Triton X-100 and CTAB+Tween-80 at constant temperature =25°C, shaking rate=160 rpm, Temperature= 25°C, pH=2 and initial feed Cr (VI) ion concentration =20 mgL-1.

3.4. Isotherm study

The equilibrium study was carried out using three adsorption Isotherms namely, Langmuir, Freundlich and DKR adsorption isotherms. Isotherm parameters for the Langmuir, Freundlich and DKR models for Cr (VI) adsorption on mixed surfactant of CTAB are enlisted in Table 1. If EDR value is between 8 and 16 kJ/mol, the biosorption process is by chemical ion-exchange and if $\text{EDR} < 8$ kJ/mol, the sorption process is of a physical nature^[15]. In our study all EDR values are less than 8 kJ/mol for Cr (VI) adsorption with both the mixed surfactant systems. The adsorption energy EDR, for adsorption of Cr (VI) was found to be 1.626 kJ/mol and 0.88 kJ/mol with CTAB +Triton X-100 and CTAB +Tween -80 mixed micelle system. These results indicate that Cr (VI) interaction with

both the mixed micelles are via physical forces i.e. physisorption mechanism. r_{max} , maximum adsorption capacity obtained from Langmuir model were found to be 7.633 mg g⁻¹ and 9.259 mg g⁻¹. On application of Freundlich model in our study, the resulted n values settled between 1 to 10, which infers favourable adsorption process [16]. The

correlation coefficient $R^2 \geq 0.99$ for Langmuir isotherm and $R^2 \geq 0.87$ for Freundlich model. Evidently, the data best fit in the Langmuir model. This suggests that Cr (VI) sorption is monolayer and there is homogeneous distribution of active sites on the surface of CTAB+Triton X-100 and CTAB +Tween -80 micelles [17].

Table 1: Langmuir, Freundlich and DKR model parameters for the Cr (VI) adsorption onto mixed micelles of CTAB

Metal ion treatment with binary surfactants	Isotherm model						
	Langmuir			Freundlich			DKR
Cr(VI)	Γ_{max} (mg g ⁻¹)	K_L (L mg ⁻¹)	R^2	n	K_F (L mg ⁻¹)	R^2	E_{DR} (kJ/mol)
CTAB+ Triton X-100	7.6335	0.2298	0.9959	1.6835	1.5631	0.8759	1.626
CTAB+ Tween-80	9.2592	0.1527	0.9915	1.4925	1.3551	0.9680	0.888

3.5. Thermodynamic study

Adsorption thermodynamic was studied at different temperatures 25°C, 30°C, 35°C, 40°C and 45°C for calculating thermodynamic parameter such as ΔG° , ΔH° and ΔS° . It was found the removal percentage of hexavalent Chromium was maximum at 25°C. The adsorption efficacy decreased on increasing the temperature from 25°C to 45°C. The negative value for enthalpy ΔH° is accounted for the system is extremely exothermic in nature leading to release and supply of energy for transference of hydrophobic tail of non-ionic and advance conformational as well as morphological changes in surfactants structure at the interface [18]. In general, the sorption process is classified to be film-diffusion controlled when E_{Act} is below 16 kJ mol⁻¹, particle-

diffusion controlled when E_{Act} is 12–50 kJ mol⁻¹, and chemical-reaction controlled when E_{Act} is greater than 40 kJ mol⁻¹ [19]. The Activation energy E_{Act} and pre-exponential factor A for Cr(VI) adsorption show respectively 50.219 kJ/mol, 914378.6 with CTAB+Triton X-100 and 29.669 kJ/mol, 6148.725 with CTAB+Tween-80. Thus, it can be stated that Removal of Cr⁶⁺ on CTAB+Triton X-100 and CTAB+Tween-80 micelles is particle diffusion controlled. An increase in the number of active centres corresponds to a higher value of pre-exponential factor A . Decrease in the energy of the active centres is attended by a growth in the value of E_{Act} . It thus becomes clear why high activation energy is connected with a high value of A [20].

Table 2: Thermodynamic and thermokinetic parameters of Cr (VI) adsorption with CTAB+Triton X-100 and CTAB+Tween-80

Mixed surfactant system	Temperature (°C)	ΔG° (kJ mol ⁻¹)	ΔH° (kJ mol ⁻¹)	ΔS° (Jmol ⁻¹ ·K ⁻¹)	E_{Act} (kJ mol ⁻¹)	A (s ⁻¹)
CTAB + Triton X-100	25	-6.909	-33.647	0.0894	50.219	914378.6
	30	-6.642				
	35	-6.197				
	40	-5.659				
	45	-5.156				
CTAB + Tween 80	25	-6.656	-17.393	0.0363	29.669	6148.725
	30	-6.314				
	35	-6.162				
	40	-6.108				
	45	-5.857				

Generally, when the ΔG° value is in the range of 0 to -20 kJ.mol⁻¹, occurrence of physical adsorptions is apparent [21]. ΔG° value for CTAB+Triton X-100 and CTAB+Tween-80 in Cr⁶⁺ adsorption were found to be -4 kJ/mol and -6 kJ/mol, this infers to the idea that adsorbate-adsorbent bind via weak vander waals forces. The negative value of enthalpy and Gibbs free energy thus indicate that the sorption of Cr(VI) on mixed micelles is spontaneous and exothermic in nature. The reaction is accompanied with positive entropy in both the selected system, which reveals increased randomness and disorderliness at the interface. The mechanism relies mainly on attractive electrostatic interaction between negative chromate ions and positively charged mixed micelles of cetyl trimethyl ammonium bromide functional group. However, CTAB+Triton X-100 and CTAB+Tween-80 micelles indicate that physical adsorption is the predominant mechanism in the sorption process of Hexavalent Chromium.

Conclusion

Cr(VI) is a noxious inorganic contaminant that has been proved to be potentially carcinogenic. The removal of Cr(VI) by CTAB+Triton X-100 and CTAB+Tween-80 mixed

surfactant systems showed percentage rejection of 97% and 94% respectively. The optimization study revealed that at constant surfactant molar ratio (non-ionic: cationic) of 0.1, pH 2, filtration time 60 minutes and initial feed metal ion concentration 20mg L⁻¹ can be considered as optimum. Change in pH caused profound effect on the adsorption of Cr(VI) and both the mixed micelles displayed better performance in acidic medium. Langmuir model ($R^2 > 0.99$) better fit the adsorption data than Freundlich model ($R^2 > 0.8$). However, the adsorption of Cr(VI) on mixed micelles of CTAB+Triton X-100 and CTAB+Tween-80 is homogeneous and occurs by formation of monolayer coverage. Low values of E_{DR} values obtained from DKR adsorption isotherm shows the Cr(VI)-micelle interaction is through electrostatic attraction. The negative value of Gibbs free energy and enthalpy indicate the sorption process to be exothermic and feasible. Vant hoff plots show extremely high values of apparent activation energy E_{Act} , 50.219 kJ/mol and 29.66 kJ/mol respectively by CTAB+Triton X-100 and CTAB+Tween-80 micelles. This represents that the rate of Cr(VI) adsorption by CTAB+Triton X-100 was found to be

slower than with CTAB+Tween-80. Conclusively, the interaction of Cr(VI) with mixed micelles was purely through electrostatic attractions and the mechanism followed was predominantly via. Physisorption. Thus, use of CTAB+Triton X-100 and CTAB+Tween-80 through micellar enhanced ultrafiltration (MEUF) technique showed enhanced performance and can be employed as an efficient remediation method in removal of hexavalent Chromium.

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