Precipitation of cetyl (hexadecyl) pyridineum chloride using mono and divalent oxyanions

M.K. Purkait [a,*], S. DasGupta [b], S. De [b]

[a] Department of Chemical Engineering, Indian Institute of Technology Guwahati, Guwahati 781039, India
[b] Department of Chemical Engineering, Indian Institute of Technology Kharagpur, Kharagpur 721302, India

ABSTRACT

Experimental investigations have been carried out to observe the performance of precipitation behavior of potassium permanganate (KMnO$_4$) and potassium dichromate (K$_2$Cr$_2$O$_7$) in cetyl (hexadecyl) pyridineum chloride (CPC) solution. As in the case of Al^{3+}-dodecylbenzenesulfonate systems [P. Somasundaran, K.P. Anathapadmanabhan, M.S. Celik, Langmuir 4 (1988) 1061–1063], the precipitation is found to be caused by interaction of CPC micelles with oxyanions. The counter oxyanions have a strong tendency to bind themselves to the surface of cationic CPC micelles. This lowered the free oxyanion concentration in solution. Therefore, to start precipitation, higher oxyanion concentration is required for higher CPC concentration. The effects of temperature, concentrations of both counter ions and CPC on the precipitation have been studied in detail. It has been observed that at CPC to KMnO$_4$ concentration ratio of 1.0 (concentration of CPC and KMnO$_4$ is 400 ppm), the percentage precipitation of CPC is around 99.3 at 30 °C. The percent precipitation of CPC decreases to about 94% when temperature increases to 70 °C at the same condition. The extent of CPC precipitation increases at the same experimental condition when K$_2$Cr$_2$O$_7$ is used instead of KMnO$_4$.

1. Introduction

Surfactants are used in several separation processes like; micellar enhanced ultrafiltration, reverse micellar extraction of proteins, separation using aphrons, and mineral flotation in froth flotation. Both cationic and anionic surfactants are used in different surfactant mediated separation processes. Irrespective of type of surfactant, it is important to recover surfactant from these processes for the process to be economical and eco-friendly.

Precipitation of anionic surfactants (sodium dodecyl benzenesulfonate (SDDBS) and sodium dodecane sulfonate (SDDS) in presence of Al^{+3} has been studied by Somasundaran et al. [1]. Precipitation of anionic surfactant has also been studied using polyvalent cations like Ca^{+2} [2]. Talens et al. observe that with Al^{+3}, SDDBS forms precipitate but sodium lauryl sulfate (SDS) and α-olefin sulfonate (AOS) forms flocculate [3]. Micellar flocculation of anionic surfactants in presence of Al^{+3} [4], Fe^{+3} [5] and hydrocarbons [6] has been studied in connection with its effects on enhanced oil recovery (EOR), adsorptive micellar flocculation (AMF), recovery of surfactants from surfactant based separation processes like, micellar enhanced ultrafiltration (MEUF), surfactant enhanced carbon regeneration (SECR), etc. In micellar flocculation of anionic

surfactant, binding of cations to the aggregated anionic surfactant molecules (micelles) changes the surface potential of anionic micelles and forms large flocs due to micellar coagulation, which are than separated by coarse filtration. In AMF, pollutants presents in aqueous medium are adsorbed on flocs and then separated. In case of precipitation, as soon as precipitation started, micelles are disrupted to maintain the critical micellar concentration (CMC), releasing more cations and accelerating the precipitation process [3]. The performance of the precipitation process is highly dependent on the solubility of the complex formed by the cations and anionic micelles. Many workers [3–6] have been studied different conditions like metal ion concentration, pH, etc., for the extent of precipitation of anionic surfactant like SDS (sodium dodecyl sulphate), AOS (α-olefinsulfonates), etc. Porras and Talens [7] have studied the removal of pesticide 2,4-dichloro phenoxy acidic acid from aqueous solution by adsorptive micellar flocculation (AMF) using AOS. In our recent published paper, CPC is used a cationic surfactant for MEUF of dye and different aromatic alcohols. The precipitation of CPC has been successfully done by forming insoluble cetyl pyridineum iodide salt [8–10]. Studies on recycling of CPC using precipitation or other method are still scant.

In the present work, precipitation of CPC using monovalent (KMnO$_4$) and divalent (K$_2$Cr$_2$O$_7$) oxyanions has been studied. Although many factors which influence the rate of precipitation, such as presence of different ionic and polymeric impurities, salinity, pH and insoluble particles, etc. have not been studied here,

* Corresponding author. Tel.: +91 361 2582262; fax: +91 361 2582291.
E-mail address: mihir@iitg.ernet.in (M.K. Purkait).

the effects of temperature and the concentration of surfactant and counter ions on the extent of precipitation of both surfactant and oxyanions have been observed. Using precipitation technique, two observations have been made, namely, (1) separation or removal of $KMnO_4$ and $K_2Cr_2O_7$ using CPC micelles and (2) separation or removal of CPC using $KMnO_4$ and $K_2Cr_2O_7$. It is to be noted that for both the cases, $KMnO_4$ and $K_2Cr_2O_7$ are selected as monovalent and divalent oxyanion in order to observe the precipitation performance with varying ionic character. For removal of $Cr_2O_7^{2-}$ from aqueous medium, CPC may be a suitable one but for removal or reuse of CPC, selection of $K_2Cr_2O_7$ may not be recommended due its toxicity point of view. In that case, $KMnO_4$ will be ideal one. However, apart from the above two observations, the precipitation and/or flocculation of CPC are also explained using $KMnO_4$ and $K_2Cr_2O_7$.

2. Mechanism of CPC precipitation

A schematic diagram representing precipitation of cetyl pyridinium (CP) ion in presence of monovalent and divalent oxyanions has been shown in Fig. 1. The CP ion is present in three environments: (i) in micellar form; (ii) in monomer form; (iii) in precipitate form. Both the oxyanions exist as (i) bounded species with CP micelles; (ii) unbounded species; (iii) $(CP)_x$-oxyanion precipitate. x is the number of CP molecules bounded with a single oxyanion. For MnO_4^- and $Cr_2O_7^{2-}$, the value of x is 1 and 2, respectively. Counter oxyanions bind on the surface of oppositely charged CP micelles. Monomer CP ions form $(CP)_x$-oxyanion precipitates. As

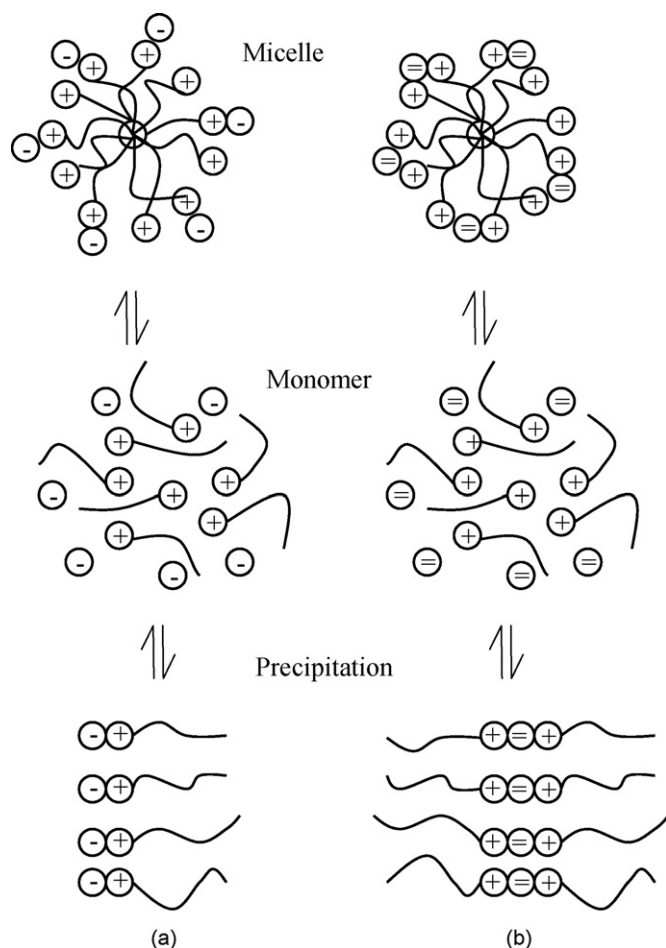soon as precipitation begins, micelles break down to maintain the concentrations of free CPC molecules at CMC. Thus oxyanions those are bound on the micelles go to the solution which increases the precipitation of CP ions.

3. Experimental

3.1. Chemicals

Technical grade cetyl (hexadecyl) pyridinium chloride (CPC) has been taken from SISCO Research Lab. Pvt., Ltd., India as cationic surfactant and is used as received. Further purification of CPC is irrelevant for two reasons: (1) cost of CPC will be very high if it is purified again and (2) some amount of impurities may eventually be mix up with CPC during further purification. Nevertheless, minimum assay content of received CPC is 98% having sulphated ash content of maximum 0.1%. The critical micellar concentration of CPC is 322 ppm in aqueous medium at 25 °C and aggregation number is 136 [11]. Potassium permanganate ($KMnO_4$) and potassium dichromate ($K_2Cr_2O_7$) are procured from Merck (India) and used as received.

3.2. Methods

In the precipitation experiments, solutions (100 ml) of surfactant (CPC) and counter oxyanion have been prepared by dissolving accurately weighed amount of CPC with potassium permanganate ($KMnO_4$) and potassium dichromate ($K_2Cr_2O_7$), separately, at different concentrations in distilled water. The pH of the solution is kept constant at 7 for all the experiments using hydrochloric acid and sodium hydroxide solution and is measured by pH meter supplied by Toshniwal Instruments Ltd. (India).

Each experiment is conducted by using a 150 ml bottle with stopper containing different concentrations of CPC and oxyanion solution in a constant temperature bath (supplied by Testing Instruments Manufacturing Company Ltd., India) for 30 min. After completion of precipitation, bottle is removed from the temperature bath and precipitate is collected very soon after filtration with the help of Whatman filter paper. Filtrates are taken at different temperatures and are diluted quickly after filtration to minimize error in the spectrophotometric analysis due to reappearance of precipitate as the solution cooled to room temperature. All the experiments have been conducted at three different temperatures (30, 50 and 70 °C) when $KMnO_4$ is used and four different temperatures (10, 30, 40 and 60 °C) when $K_2Cr_2O_7$ is used.

3.3. Analysis

The concentrations of CPC, $KMnO_4$ and $K_2Cr_2O_7$ have been determined by a spectrophotometer (make: Thermo Spectronic, USA; model: GENESYS 2). Pure CPC, $KMnO_4$ and $K_2Cr_2O_7$ solution is initially calibrated separately for different concentration in terms of absorbance units, which is recorded at the wavelength of 259, 526 and 355 nm, respectively. The extinction coefficients of all the compounds are obtained from the measurements of the pure components and are shown in Table 1. A standard method is used to

Fig. 1. Schematic of equilibrium existing in CPC-oxyanions system. (a) In CPC-$KMnO_4$. (b) In CPC-$K_2Cr_2O_7$.

Table 1
Extinction coefficients for CPC, $KMnO_4$ and $K_2Cr_2O_7$

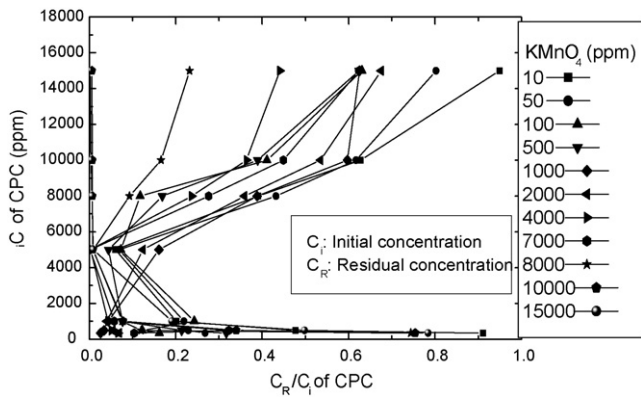| Wave length (nm)	Extinction coefficients (m^3/kg cm)		
	CPC	$KMnO_4$	$K_2Cr_2O_7$
259	3880	5.8	10.7
526	501	2128	1026
355	4016	9.2	2950

Fig. 2. Variation of residual CPC with overall CPC concentration at various $KMnO_4$ concentrations.

calculate the concentrations of CPC and the oxyanions from the solution [12].

4. Results and discussion

This section is divided into five parts. In first part, it is shown that CPC forms precipitate (not flocculate) with $KMnO_4$ and $K_2Cr_2O_7$. The precipitation behavior of CPC-$KMnO_4$ and CPC-$K_2Cr_2O_7$ systems are illustrated in the form of C_i and C_R. Where C_i and C_R are the initial (same as total) and residual concentrations of CPC. Second and third parts describe the variations of CPC and oxyanions precipitation, respectively, with different combinations of surfactant and oxyanion concentrations. Effect of temperature on the extent of precipitation is discussed in part 4. A comparison of CPC precipitation using $KMnO_4$ and $K_2Cr_2O_7$ has been made in the last part.

4.1. Precipitation of CPC in presence of $KMnO_4$ and $K_2Cr_2O_7$

Fig. 2 shows the variation of precipitation (in terms of residual CPC) of CP-MnO_4 at different combinations of $KMnO_4$ and CPC in feed at 30 °C. For a given $KMnO_4$ concentration (up to 10,000 ppm), there is no precipitation at sufficiently high CPC (up to 15,000 ppm) concentration. This is due to the fact that the entire MnO_4^- ion is bound over the oppositely charged micellar surface. It seems to be flocculation. But in actual practice it is not since the nature of curve (Ref. Fig. 2) does not match as in the case of SDS with Al^{3+} [3]. Further, increase of MnO_4^- concentration, the number concentration of free MnO_4^- ion increases in solution that precipitate the free CPC molecule. Once the precipitation processes starts, micelles break down to maintain the free surfactant concentration at CMC level leaving solubilized MnO_4^- in the solution. And hence, precipitation process enhances. Similar trend has been observed when $K_2Cr_2O_7$ is used and is shown in Fig. 3. Hence both $KMnO_4$ and $K_2Cr_2O_7$ form precipitate with CPC.

4.2. Effect of CPC to oxyanion concentration ratio on the extent of CPC precipitation

Figs. 4 and 5 show the variations of percentage CPC precipitation with different combinations of feed CPC to $KMnO_4$ and CPC to $K_2Cr_2O_7$ concentration ratio, respectively, at 30 °C. It is observed from Fig. 4 that at CPC to $KMnO_4$ concentration ratio of 1.0, percentage CPC precipitation is maximum (around 99.3%) for 400 ppm of $KMnO_4$ at 30 °C. Percentage CPC precipitation increases up to the ratio of 1.0 and beyond that ratio percentage CPC precipitation decreases gradually as shown in Fig. 4. This result indicates that the stability of CP-MnO_4 precipitate depends both on the surfactant
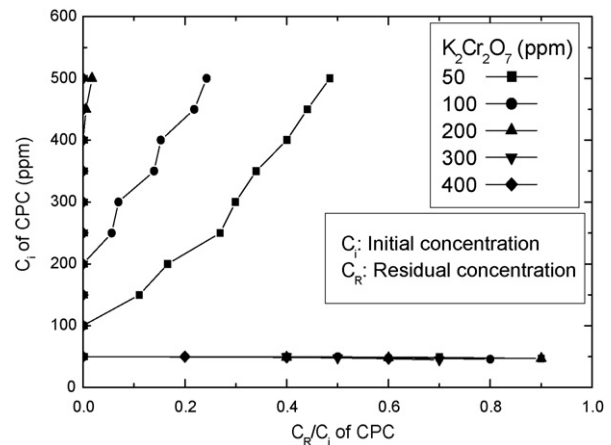

Fig. 3. Variation of residual CPC with overall CPC concentration at various $K_2Cr_2O_7$ concentrations.
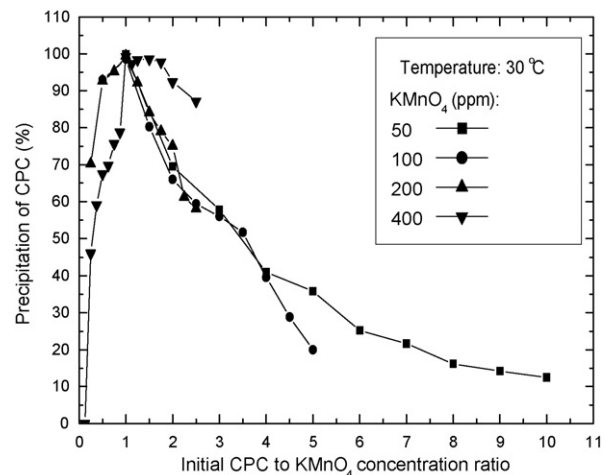

Fig. 4. Effect of CPC to $KMnO_4$ concentration ratio on the extent of CPC precipitation at 30 °C.

and $KMnO_4$ concentration. At higher $KMnO_4$ concentration (CPC to $KMnO_4$ ratio below 1.0), CP-MnO_4 precipitates are least stable. This may be due to excess MnO_4^- ions that hindered the formation of CP-MnO_4 precipitate. The decrease in percentage precipitate at higher CPC to $KMnO_4$ ratio (>1.0) is due to the fact that the $KMnO_4$
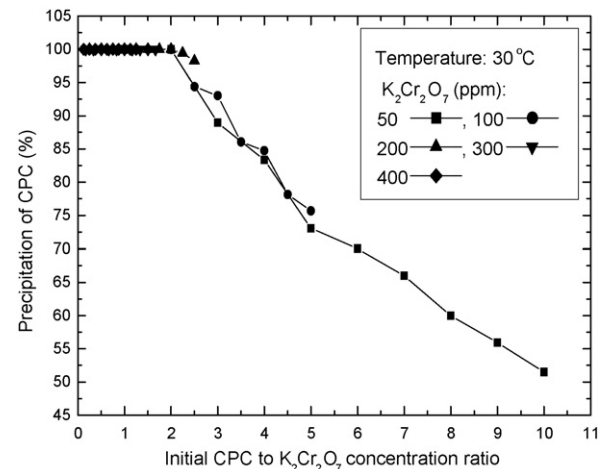

Fig. 5. Effect of CPC to $K_2Cr_2O_7$ concentration ratio on the extent of CPC precipitation at 30 °C.

Table 2
Precipitation of CPC with CPC to $K_2Cr_2O_7$ ratio observed in presence of different combinations of CPC and $K_2Cr_2O_7$

CPC (ppm)	$K_2Cr_2O_7$ (ppm)	$\frac{CPC}{K_2Cr_2O_7}$	Precipitation comes after (min)
500	250	2.00	10
500	70	7.14	10
500	50	10.00	10
500	35	14.28	15
5,000	250	20.00	15
1,000	35	28.57	15
10,000	250	40.00	20
10,615	260	40.82	20
10,890	148	73.58	Not even after 24 h
3,000	35	85.71	Not even after 24 h
10,140	110	92.18	Not even after 24 h
5,000	35	142.85	Not even after 24 h
10,480	73	143.56	Not even after 24 h
10,612	54	196.51	Not even after 24 h
7,000	35	200.00	Not even after 24 h
10,140	35	289.71	Not even after 24 h

Fig. 6. Effect of CPC to $KMnO_4$ concentration ratio on the extent of $KMnO_4$ precipitation at 30 °C.

present in the solution is not sufficient to precipitate the surfactant molecules. Same trends are observed at 50 and 70 °C and the percentage of CPC precipitation is around 95.5% and 94%, respectively, at CPC to $KMnO_4$ concentration ratio of 1.0.

Precipitation of CPC in presence of $K_2Cr_2O_7$ also depends on the feed CPC to $K_2Cr_2O_7$ ratio and is shown in Table 2 for different combinations of CPC and $K_2Cr_2O_7$ concentration. It is clear from Table 2 that beyond CPC to $K_2Cr_2O_7$ ratio of 40.82, no precipitation is observed. This may be due to the fact that up to that ratio $K_2Cr_2O_7$, surfactant molecules are precipitated and beyond that precipitates are not formed. It is observed from Fig. 5 that up to CPC to $K_2Cr_2O_7$ ratio of 2.0, percentage of CPC precipitation remains 100 for all the concentrations consider herein. Beyond that ratio, percentage of CPC precipitation decreases gradually. This is due to the instability of $(CP)_2Cr_2O_7$ precipitate as in the case of CP-MnO_4. From Figs. 4 and 5, it may be conclude that for a fixed amount of CPC, less amount (higher CPC to $K_2Cr_2O_7$ ratio) of $K_2Cr_2O_7$ is required than that required for $KMnO_4$ for maximum precipitation. Fig. 4 shows that the percentage of CPC precipitation at 30 °C remains maximum at CPC to $KMnO_4$ ratio of 1.0, whereas this ratio becomes 2.0 (as shown in Fig. 5) when $K_2Cr_2O_7$ is used instead of $KMnO_4$ at the same temperature.

4.3. Effect of CPC to oxyanion ratio on the extent of precipitation of oxyanions

Variations of percentage oxyanions precipitation with different combinations of feed CPC to oxyanions ratio have been shown in Figs. 6 and 7 at 30 °C for $KMnO_4$ and $K_2Cr_2O_7$, respectively. It may be observed from Fig. 6 that the percentage of $KMnO_4$ precipitation increases sharply with CPC to $KMnO_4$ ratio and reaches around 100% at the ratio of 1.0. Beyond that ratio, percentage CPC precipitation remains unaltered. This is because of the fact that beyond the CPC to $KMnO_4$ ratio of 1.0, all $KMnO_4$ molecules get precipitated in the experimental concentration range of CPC and $KMnO_4$. Same trend have been observed for 50 and 70 °C, and maximum precipitation is observed at a ratio of 1.5 and 3.0, respectively.

It may be observed from Fig. 7 that the percentage of $K_2Cr_2O_7$ precipitation increases with CPC to $K_2Cr_2O_7$ ratio. Beyond the ratio of 2.0, precipitation remains unaltered up to a certain limit and dissolves thereafter. For example, for the feed $K_2Cr_2O_7$ concentration of 50 ppm, 86% precipitation is observed at the ratio of 2.0 and beyond that ratio percentage $K_2Cr_2O_7$ precipitation remains almost unaltered from ratio 2.0 to around 10. Beyond the ratio of 10, no precipitation is found. Similar trend is observed for the feed $K_2Cr_2O_7$
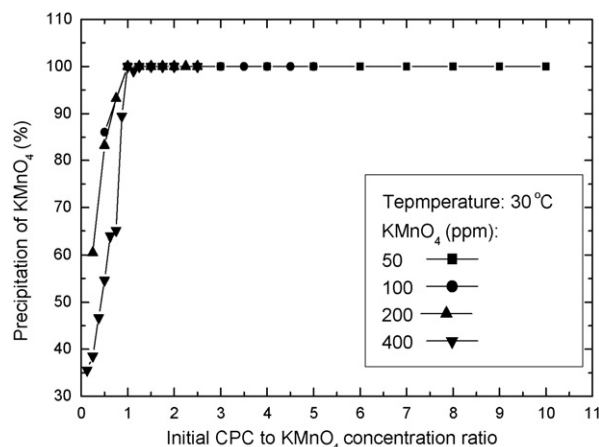
concentration of 100, 300 and 400 ppm. This is because of the fact that up to the ratio of 2.0 all the CPC molecules are precipitated (Ref. Fig. 5). Further addition of CPC (ratio beyond 2.0), concentration of $K_2Cr_2O_7$ is insufficient to precipitate all the CPC molecules and the precipitate is dissolve thereafter. Higher the $K_2Cr_2O_7$ concentration in the feed results lower precipitation of $K_2Cr_2O_7$ due to greater extent of free $K_2Cr_2O_7$ molecules in the solution.

4.4. Effect of temperature on the precipitation

Temperature variation is one of the important parameter that changes the extent of CPC and $KMnO_4$ precipitation and is shown in Figs. 8 and 9 at the CPC to $KMnO_4$ ratio of 1.0 for 50 and 200 ppm of $KMnO_4$, respectively. It is observed from both the figures that percentage precipitation of CPC and $KMnO_4$ decreases with increasing temperature. It may be observed from Fig. 8 that the percentage of CPC precipitation decreases from about 99.8 to 47.5 and percentage of $KMnO_4$ precipitation decreases from about 100 to 94.3 when temperature increases from 30 to 70 °C. Same trend is observed for 200 ppm of feed $KMnO_4$ at CPC to $KMnO_4$ ratio of 1.0 and is shown in Fig. 9. In that case, CPC precipitation decreases from about 99 to 85% when temperature increases from 30 to 70 °C. Lower precipitation of CPC along with $KMnO_4$ at higher temperature is due to the fact that solubility of CP-MnO_4 precipitate increases with temperature causes increase in the concentration of the free CPC and $KMnO_4$ in the solution which results a decline in the extent of precipitation.
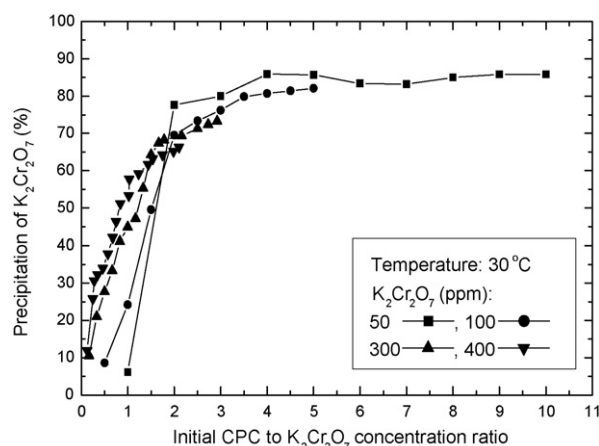

Fig. 7. Effect of CPC to $K_2Cr_2O_7$ concentration ratio on the extent of $K_2Cr_2O_7$ precipitation at 30 °C.

Table 3
Comparison of CPC precipitation in presence of $KMnO_4$ and $K_2Cr_2O_7$

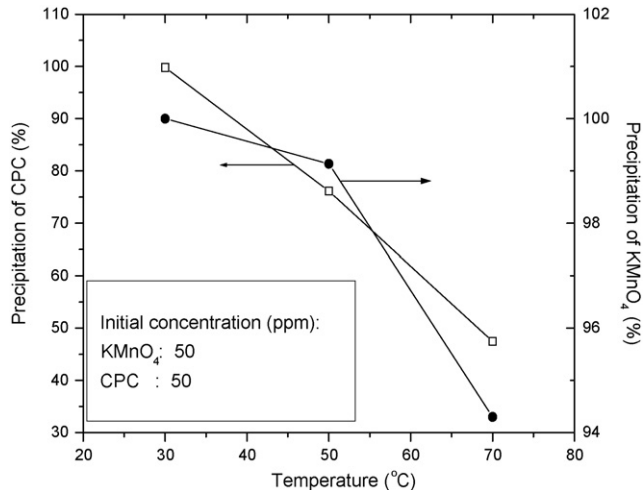| Temperature (°C)	%CPC precipitation	%$KMnO_4$ precipitation	Temperature (°C)	%CPC precipitation	%$K_2Cr_2O_7$ precipitation
30	99.26	99.97	30	100.0	55.25
50	95.56	99.65	40	99.73	33.12
70	94.08	99.39	60	96.95	25.59

Fig. 8. Variation of CPC and $KMnO_4$ precipitation with temperature for 50 ppm of CPC and $KMnO_4$ each.

Fig. 10 shows the variations of CPC and $K_2Cr_2O_7$ precipitation with feed CPC concentration at different temperature and 400 ppm of $K_2Cr_2O_7$. It may be observed from the figure that up to 40 °C, the percentage precipitation of CPC remains 100% and is almost independent of temperature and feed CPC concentration but decreases 4–5% when temperature increases to 60 °C. A 400 ppm of $K_2Cr_2O_7$ used in this case is sufficient to precipitate 500 ppm of CPC and hence 100% precipitation is observed. These precipitates are insoluble up to 40 °C. Beyond that temperature, increase in solubility of $(CP)_2$-Cr_2O_7 precipitate causes decline in CPC precipitation.

It may also be observed from Fig. 10 that the precipitation of $K_2Cr_2O_7$ increases with feed CPC concentration and decreases with increasing temperature. Percentage of $K_2Cr_2O_7$ precipitation increases from 41.1 to 69.5 when CPC concentration increases 50 to 500 ppm. On the other hand, percentage of $K_2Cr_2O_7$ precipitation decreases from 69.5 to 42.8 when temperature increases from 10

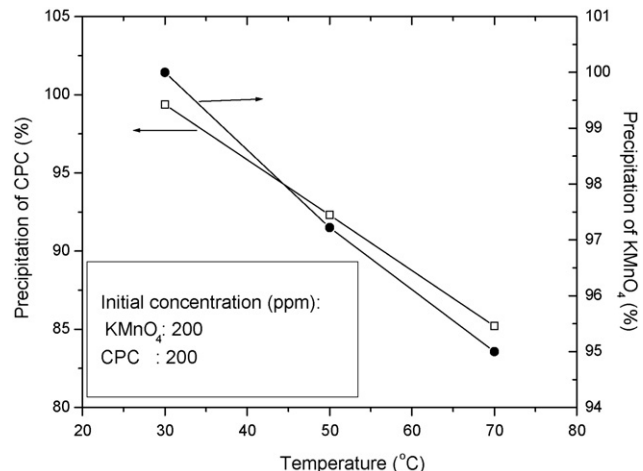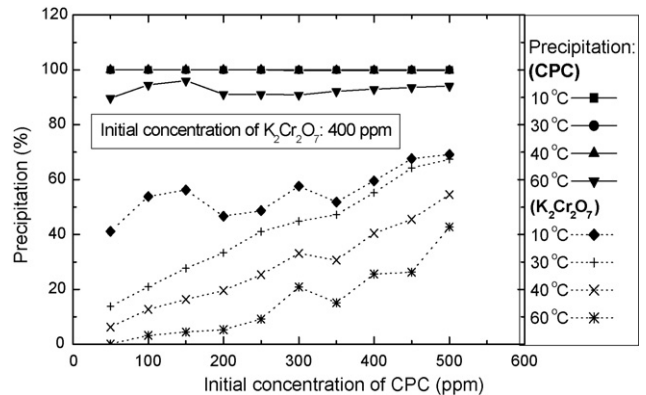**Fig. 9.** Variation of CPC and $KMnO_4$ precipitation with temperature for 200 ppm of CPC and $KMnO_4$ each.

Fig. 10. Variation of CPC and $K_2Cr_2O_7$ precipitation with initial CPC concentration at different temperature.

to 60 °C. Increasing $K_2Cr_2O_7$ precipitation with CPC concentration is due to more number of CPC molecules in feed mixture. Lower precipitation of $K_2Cr_2O_7$ at higher temperature is due to decrease in solubility of $(CP)_2$-Cr_2O_7 precipitate as discussed earlier.

4.5. Comparison of CPC precipitation in presence of $KMnO_4$ and $K_2Cr_2O_7$

A comparison of CPC and oxyanion precipitation has been shown in Table 3 using 400 ppm of both CPC and oxyanion concentration in feed at different temperatures. It may be observed from Table 3 that the percentage of CPC and both mono and divalent oxyanions precipitation decreases with increasing temperature. The reason for the above information has already been discussed earlier. It may also be observed from Table 3 that the percentage of CPC precipitation is somewhat less when $KMnO_4$ is used instead of $K_2Cr_2O_7$ at 30 °C. This is because of the fact that at a certain temperature and CPC to oxyanion concentration ratio, less amount of divalent counter ion ($Cr_2O_7^{2-}$) is sufficient for the complete precipitation of oppositely charged CPC than that required for the monovalent counter ions (MnO_4^-). Therefore, CPC precipitation is more in case of $K_2Cr_2O_7$ than that of $KMnO_4$ at the same concentration level.

On the other hand, $K_2Cr_2O_7$ precipitation is very less than that of $KMnO_4$ at the same experimental condition. This may be due to the fact for 400 ppm of CPC leaser amount of $K_2Cr_2O_7$ rather than $KMnO_4$ is required for precipitation. Excess $K_2Cr_2O_7$ remains in the solution which decreases the numeric value of percentage of precipitation of $K_2Cr_2O_7$.

5. Conclusion

Precipitation of CPC has been studied using $KMnO_4$ and $K_2Cr_2O_7$. The effect of temperature, concentrations of CPC and oxyanions on the extent of precipitation are studied in detail. It is observed that the precipitation of CPC and oxyanions decrease with increasing temperature. The temperature study may help in assessment of the optimum conditions for precipitation of CPC surfactant under

unique thermal environments. Precipitation of CPC is more in presence of divalent counter ions ($K_2Cr_2O_7$) rather than mono valent ($KMnO_4$) one. From the experimental observation, three major conclusions can be made.

Firstly, within the concentrations range considered herein, CPC forms precipitate with $KMnO_4$ and $K_2Cr_2O_7$ but not does form flocculate as in the case of SDS and AOS with $Al_2(SO_4)_3$.

Secondly, precipitation technique may be an useful process for the removal of high concentration of Cr(VI) and Mn(VII) from waste water in the form of $K_2Cr_2O_7$ and $KMnO_4$ using CPC as cationic surfactant. It is observed that CPC to $KMnO_4$ concentration ratio of 1.0 and CPC to $K_2Cr_2O_7$ concentration ratio of 2.0, the precipitation are maximum. This ratio will be help full to know the amount of CPC required to treat a known concentration of $K_2Cr_2O_7$ and $KMnO_4$ in the waste water. Precipitates are separated by coarse filtration.

Finally, the experimental results of precipitation of CPC using $KMnO_4$ and $K_2Cr_2O_7$ may be helpful to think about an alternative method for the recycle or reuse of CPC surfactant used in different surfactant based separation techniques. In that case, $KMnO_4$ is always recommended considering the harmful effects of Cr(VI) and Mn(VII).

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