

Application of Starch Xanthates for Cadmium Removal: A Comparative Evaluation

MOHAMMAD JAWED and VINOD TARE*

Environmental Engineering, Civil Engineering Department, Indian Institute of Technology, Kanpur-208016, India

SYNOPSIS

Attempts to solve the problem of heavy metals removal and recovery from wastewaters have led to the development and application of several techniques. One of the techniques involves application of starch and cellulose-based products, especially xanthates. Both water-soluble and insoluble starch xanthate processes have been claimed to be successful based on the studies conducted with relatively simple wastewaters. The major objectives of this study have been to evaluate the effectiveness of insoluble starch xanthate (ISX) for the removal of cadmium, study the effect of complexing agents/ligands on the interaction of cadmium with ISX and soluble starch xanthate (SSX), and to make overall comparison of ISX and SSX processes for cadmium removal. Both SSX and ISX can bring down the aqueous phase cadmium concentration to less than 1 mg/liter (Indian effluent discharge standard) irrespective of the pH (in the range of 3 to 7), ionic strength, and initial cadmium concentration. The metal binding capacity of ISX is maximum in the pH range of 4 to 5 and is constant irrespective of equilibrium metal concentrations as well as different doses of ISX used at lower pH values (3 to 5). The metal uptake by ISX/SSX in the presence of EDTA is affected considerably. ISX shows higher uptake of cadmium in the presence of EDTA as compared to SSX. The insoluble xanthate process appears to have an edge over soluble xanthate process in terms of metal removal capacity, reliability, and ease of operation, particularly for cadmium.

INTRODUCTION

Humans have evolved in an environment containing metals and have developed protective means of defense against natural concentrations. Indeed, some metals have become essential for life. However, industry is pouring metals, particularly heavy metals, into our environment at an unprecedented and constantly increasing rate. As a result, technological society is exposing the population to some metals in unnaturally high concentrations, in unusual physical or chemical forms, and through unusual portals of entry. This trend is certainly likely to continue.

Various traditional (precipitation) and advanced technologies have been developed for the removal and recovery of heavy metals from wastewaters.¹

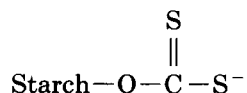
However, the precipitation process using conventional precipitants (hydroxides, carbonates, sulfides, etc.) cannot always give satisfactory removal to meet the pollution control limits, whereas advanced technologies may not be economically viable. Recently, starch-based products have been proposed as potential heavy metal scavengers. Out of several starch-based products, one school of thought²⁻⁷ prefers the use of insoluble starch products, particularly xanthates, while others⁸⁻¹⁰ prefer soluble over insoluble ones. At present, both soluble and insoluble products seem to be attractive.

The preference of soluble xanthate over the insoluble one or vice versa may depend on the type of metal ion and environmental conditions.¹¹ Hence, it is necessary to evaluate both (soluble and insoluble) xanthate processes with respect to a particular metal. The present investigation is directed with a particular metal ion cadmium. Cadmium is found to be effectively removed from pure as well as solutions containing metal ions Cu(II), Fe(II), Pb(II), Mn(II), Hg(II), Ni(II), Zn(II), etc.¹²

* To whom correspondence should be addressed.

EXPERIMENTAL METHODOLOGY

To study the interaction of starch xanthate and cadmium in aqueous environment, experiments were conducted using water-insoluble and water-soluble starch xanthates prepared from cornstarch (ICSX and SSX). The procedure for synthesis of these was similar to that employed by Tare and Chaudhari.¹¹ The structure of ICSX and SSX can be represented⁸ as



The basic difference between ICSX and SSX is in the preparation of base material from starch (viscose or crosslinked starch). Crosslinking of starch makes it insoluble in the aqueous phase. The aqueous environment was simulated to typical metal-bearing wastewaters by adjusting ionic strength (μ) and pH using appropriate salts and buffers, respectively. The experiments conducted with insoluble xanthates are classified as sorption experiments as they involve transfer of solute molecules from aqueous phase to the already existing solid phase (ICSX). The experiments with soluble xanthate are classified as precipitation experiments since the solid phase is formed after the interaction between soluble xanthate molecules and metal ions. A typical outline of the experimental procedure adopted for soluble and insoluble xanthate processes is shown in Figure 1.

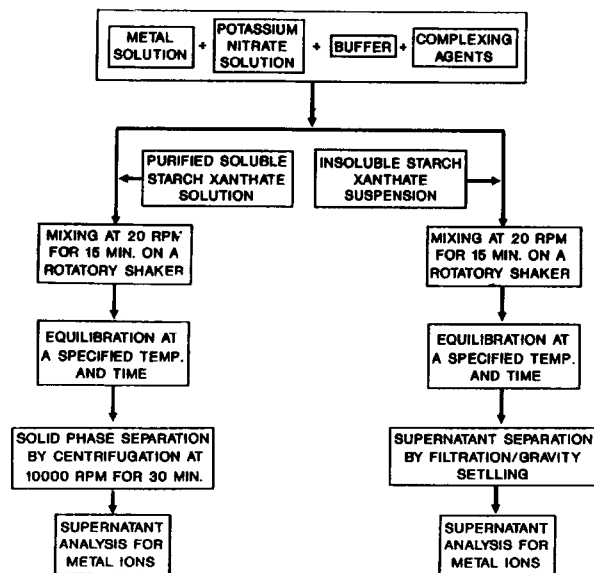


Figure 1 Typical outline of the experimental procedure adopted for soluble and insoluble xanthate processes.

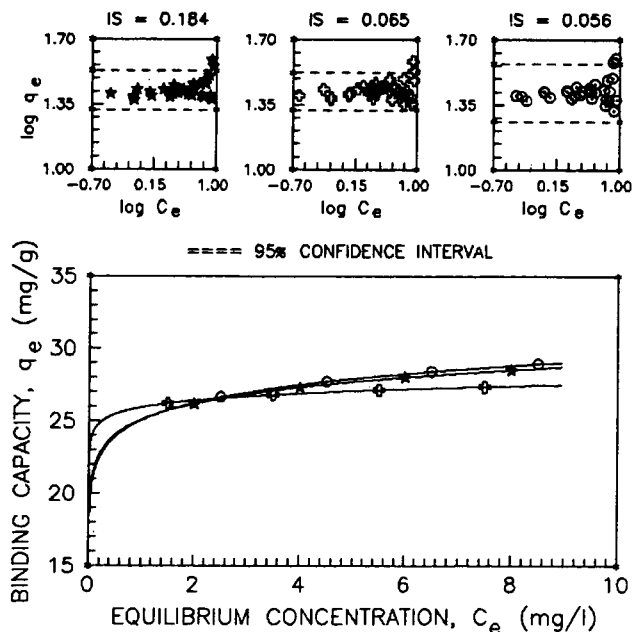


Figure 2 Equilibrium distribution of cadmium between aqueous phase and ICSX at pH 4.0 ± 0.1 .

Further details are presented elsewhere.^{11,13} Estimation of cadmium was done using the spectrophotometric method¹⁴ and atomic absorption spectrophotometer depending on the expected concentration and aqueous phase characteristics.

RESULTS AND DISCUSSION

Cadmium-Insoluble Starch Xanthate Interaction

The Cd-ISX interaction is characterized here as sorption interaction. The evaluation of the suitability of the sorption unit operation requires the consideration of two important physicochemical aspects of the process, the kinetics and the equilibria of sorption. The rate of uptake of metal ion by ISX was very rapid. Chaudhari¹³ experimentally demonstrated that the equilibrium for Cd(II)-ICSX system is achieved in less than 10–15 min. Considering this and allowing for different mixing and aqueous phase conditions that might exist in sorption equilibria studies, an equilibrium time of 2 h was used.

Distribution of Cadmium Between Aqueous Phase and ISX

Figures 2 and 3 are representative of the sorption isotherm data for Cd(II)-ICSX system. Nonlinear plots show best fit curve along with a few computed

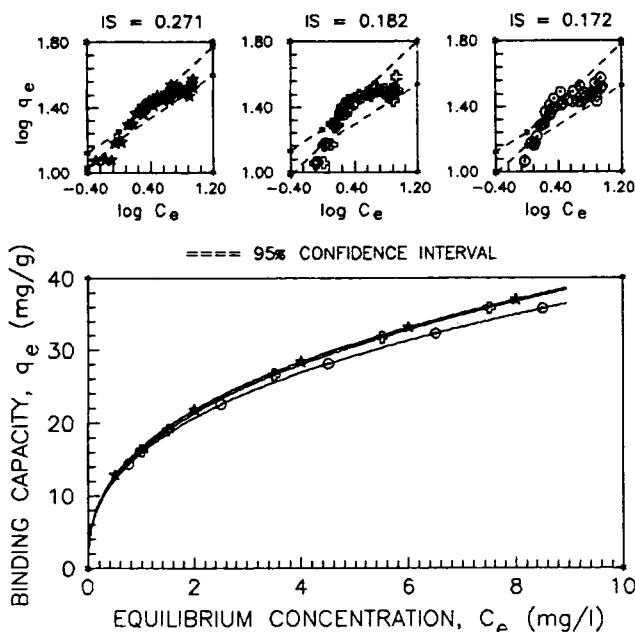


Figure 3 Equilibrium distribution of cadmium between aqueous phase and ICSX at pH 7.0 ± 0.2.

points whereas linearized plots depict the experimental data. All Cd-ICSX experiments were conducted in triplicate. The present data gave a better fit to the Freundlich equation, which is expressed as

$$q_e = K_f C_e^{1/n} \tag{1}$$

where K_f and n are constants, q_e is the mass of sorbate per unit weight of sorbent (metal binding capacity of ISX), and C_e is the equilibrium sorbate concentration in solution. The estimated isotherm parameters, along with the values of coefficient of correlation (R), standard error of estimate (σ), number of observations (N), and range for the 95% confidence interval are presented in Table I.

It can be observed from the linearized plots of the estimated q_e values (Fig. 2 and Table I) that the sorption intensity, $1/n$ is negligible at pH 3, 4, and 5. This was deduced by testing¹⁵ the hypothesis that the slope given by the linearized form of Eq. (1) is zero. This means that the metal binding capacity of

Table I Estimated Freundlich Isotherm Parameters and Relevant Statistical Information

pH	Cd(II) conc. (mg/l) (approx.)	μ	K_f	$\frac{1}{n}$	95% Confidence Interval			R	N
					$\log K_f$	$\frac{1}{n}$	σ		
± 0.1	10	0.141	19.55	≈ 0	1.16–1.39	≈ 0	0.049	—	36
± 0.0		0.045	18.56	≈ 0	1.21–1.32	≈ 0	0.027	—	32
3 ± 0.0		0.036	18.01	≈ 0	1.18–1.32	≈ 0	0.024	—	30
± 0.2	30	0.036	23.56	≈ 0	1.31–1.42	≈ 0	0.027	—	35
± 0.1	10	0.184	27.20	≈ 0	1.32–1.53	≈ 0	0.046	—	33
± 0.1		0.065	26.90	≈ 0	1.32–1.52	≈ 0	0.043	—	36
4 ± 0.1		0.056	27.58	≈ 0	1.26–1.57	≈ 0	0.055	—	34
± 0.2	30	0.056	28.84	≈ 0	1.37–1.53	≈ 0	0.029	—	33
± 0.1	10	0.161	26.81	≈ 0	1.34–1.56	≈ 0	0.032	—	34
± 0.1		0.088	29.01	≈ 0	1.33–1.56	≈ 0	0.055	—	33
5 ± 0.1		0.079	26.94	≈ 0	1.33–1.51	≈ 0	0.040	—	33
± 0.1	30	0.079	29.68	≈ 0	1.37–1.56	≈ 0	0.045	—	34
± 0.2	10	0.193	19.46	0.260	1.27–1.31	0.227–0.290	0.030	0.975	36
± 0.2		0.124	22.11	0.144	1.31–1.37	0.096–0.195	0.038	0.828	36
6 ± 0.25		0.115	22.38	0.105	1.32–1.39	0.052–0.152	0.033	0.769	36
± 0.3	30	0.114	20.62	0.144	1.29–1.34	0.120–0.167	0.039	0.960	36
± 0.1	10	0.271	16.70	0.383	1.20–1.25	0.332–0.429	0.047	0.971	36
± 0.2		0.182	16.24	0.394	1.17–1.26	0.310–0.470	0.074	0.931	36
7 ± 0.2		0.172	15.96	0.379	1.16–1.24	0.304–0.448	0.057	0.950	36
± 0.2	30	0.172	18.08	0.193	1.24–1.30	0.155–0.217	0.049	0.961	36

ICSX at pH 3, 4, and 5 is constant and independent of equilibrium sorbate concentrations in the C_e range of interest. However, this is not true at higher pH (6 and 7).

Cadmium Binding Capacity of ISX under Varying Aqueous Phase Composition

The sorbate-sorbent interaction in the sorption process is influenced by solution (aqueous) phase composition. The two important parameters that characterize the aqueous phase composition are pH and ionic strength, μ . The aqueous phase speciation is strongly dependent on pH. pH also dictates the extent of protonation of several functional groups on the sorbent. The ionic strength controls the mobility of ions and can influence the equilibrium constants by influencing the activities of the ions. In the present study, ionic strength was adjusted by the addition of potassium nitrate (KNO_3) and the pH was maintained by using citrate-phosphate buffer. The ionic strength values reported are computed based on equilibrium speciation obtained from modified MINEQL.¹⁶

Figures 2 and 3 show the typical variation of cadmium binding capacity of ICSX at different pH values (4 and 7) with three different ionic strength values. The ionic strength has very little or no effect on the cadmium binding capacity. However, the aqueous phase pH influences the cadmium binding capacity of ICSX significantly. From Table II, it is clear that the cadmium binding capacity of ICSX is maximum in the pH range of 4 to 5. It is also evident that the metal binding capacity of ICSX is almost independent of ICSX dose and/or equilibrium cadmium concentration at pH values ≤ 5 . However, metal binding capacity seems to be increasingly dependent on ICSX dose and/or equilibrium cadmium

concentration as the pH value increases above 5. At pH < 4 , the xanthate may be converted to an unstable form⁸ thereby causing lower uptake of metal, although the metal may be available in the most suitable form for sorption. At pH ≥ 5 , the metal may not be available in suitable form for sorption. Generally, at higher pH values, cadmium is available in much more complexed form¹⁷ thereby hindering sorption process and reducing the cadmium binding capacity of xanthate.

Effect of Complexing Agents/Ligands on the Interaction of Cadmium with SSX and ICSX

Complexing agents/ligands are generally used in electroplating and other metal industries to maintain high metal concentrations in solution. The most common chelating agents, which are reported to have significant adverse effects¹ on metal removal efficiency, using various processes, are EDTA and cyanide. Hence, it was thought to be necessary to see the effect of EDTA on the removal of cadmium by xanthate process.

EDTA (ethylenediaminetetraacetic acid) is an organic ligand. It forms strong complexes with cadmium. To study the effect of EDTA on the xanthate process for the removal of cadmium, experiments were conducted in duplicate in the pH range of 3 to 7 with three different concentrations of EDTA (0.1, 1.0, and 10.0 mmol/liter) using two different doses of ICSX and SSX (0.140 and 0.280 mmol/liter for ICSX, 0.165 and 0.330 mmol/liter for SSX). Figure 4 shows the effect of EDTA on the interaction of cadmium with ICSX and SSX. Control contains all other components (i.e., metal solution, potassium nitrate, buffer, and ICSX/SSX) except EDTA. The metal reduction in the control is considered to be 100% for comparison purposes. From Figure 4 it is clear that ICSX removes higher percentage of cadmium as compared to SSX in the pH range of 4 to 5. At pH 3, metal uptake is quite higher for SSX. At pH > 5 , the metal uptake by ICSX and SSX in the presence of EDTA is severely affected. Particularly, at pH 6 and 7, no significant removal of cadmium was observed even at lower concentration of EDTA (0.1 mmol/liter). As the concentration of EDTA is increased from 0.1 to 10.0 mmol/liter, the metal removal is reduced considerably. These results suggest that the Cd-EDTA complexes are stronger than Cd-ICSX/SSX. As the EDTA concentration and pH are increased, Cd-EDTA complexes become much stronger, thereby causing little or no significant removal of cadmium. ICSX/SSX doses seem to have no significant effect on the removal of cad-

Table II Variation of Cadmium Binding Capacity of ICSX with pH

pH	Cadmium Binding Capacity of ICSX, q_e (mg/g) at Equilibrium Cadmium Concentration, C_e (mg/liter) ^a		
	0.1	1.0	10.0
3	16.60 (1.27)	17.88 (0.25)	19.33 (1.00)
4	22.55 (1.30)	25.36 (0.40)	28.58 (0.71)
5	21.31 (1.18)	25.01 (0.63)	29.44 (1.58)
6	14.74 (2.95)	21.29 (1.29)	31.45 (2.89)
7	6.78 (0.14)	16.34 (0.31)	39.38 (1.03)

^a Values in parenthesis show standard deviation in q_e .

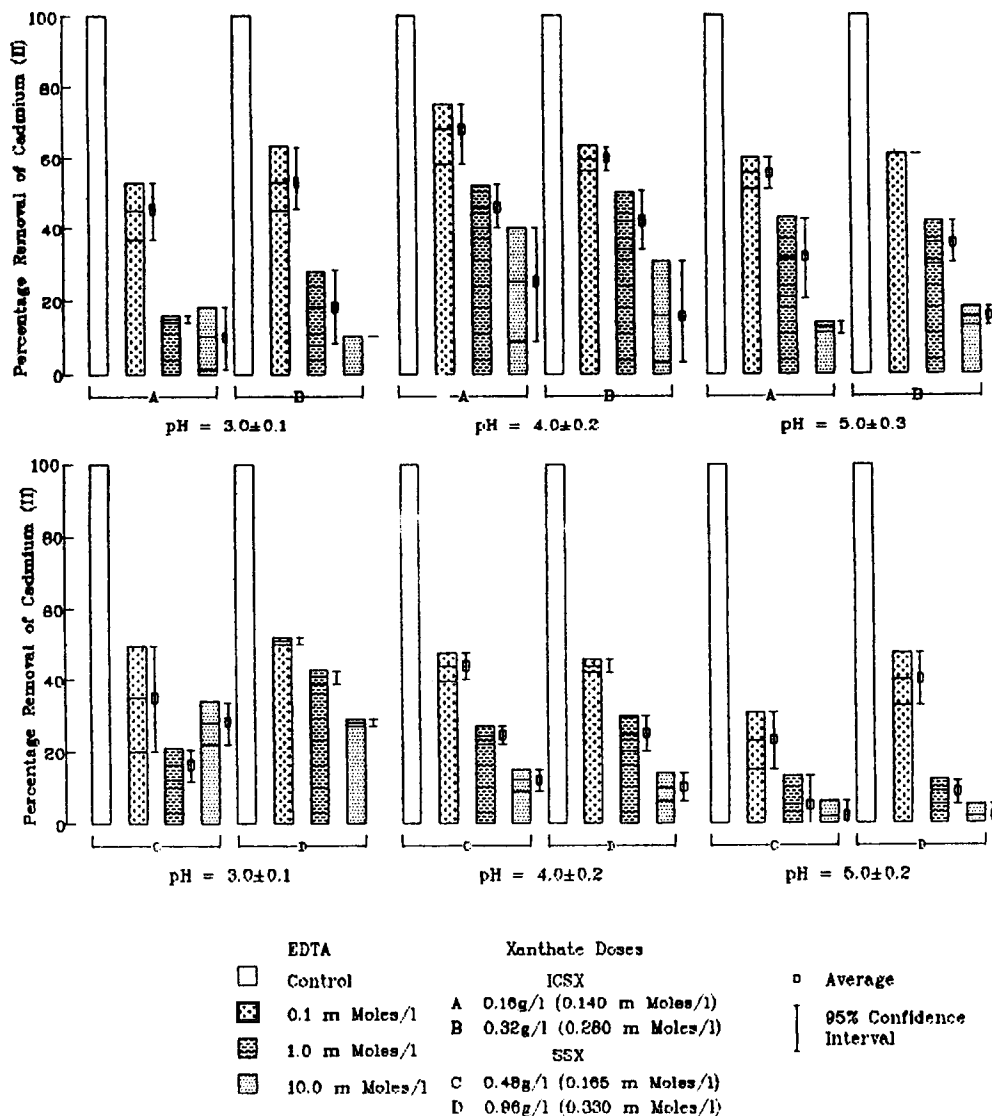


Figure 4 Effect of EDTA on the interaction of Cd-ICSX/SSX.

mium in the presence of EDTA (95% confidence intervals are overlapping, Fig. 4). However, at this stage it cannot be concluded that ICSX/SSX doses have no effect on the removal of cadmium. Detailed investigation is required with very high doses of ICSX/SSX particularly in the comparable range of EDTA concentrations used (i.e., doses of ICSX/SSX should be considered in the range of 0.1 to 10.0 mmol/liter).

Soluble Versus Insoluble Xanthate Process

As long as the wastewater is free from complexing agents/ligands or the concentration of these is below a certain level, the soluble as well as insoluble xanthate processes appear to produce wastewater having

cadmium metal concentration less than 1 mg/liter. Results on SSX process have been adopted from earlier research.¹⁷ For overall comparison of the two processes, it is essential to consider the following aspects.

Chemical Requirements. Table III shows the chemical requirements for the synthesis of ICSX and SSX. It is clear from this table that the chemical requirements for the synthesis of ICSX is higher as compared to SSX. The synthesis process of ICSX involves three steps, namely, crosslinking, xanthation, and washing/drying, whereas SSX synthesis involves only the xanthation step. The two additional steps (i.e., crosslinking and washing/drying) for the synthesis of ICSX lead to higher chemical requirements.

Table III Comparative Evaluation of ICSX and SSX

Parameters	ICSX	SSX
Chemical requirements		
Cornstarch	100 g	62 g
NaOH	48 g	15 g
Carbon disulfide	13–45 mL	13 mL
NaCl	1.5 g	—
Epichlorohydrin	7.5 mL	—
KOH	6 g	—
Acetone	1.5–2.0 liters	—
Diethylether	150–250 mL	—
Yield	118.3 g	294.38 g (Viscose)
Cost ^a of chemicals required:	Rs. 156.00 per 100 g ICSX	Rs. 2.00 per 100 g viscose
Time required	90–110 h	3–4 h
Synthesis process	Cumbersome	Easy
Storage (at low temp., 6–8°C)	For longer duration (6–8 months) no effect on metal removal	For shorter duration (3 weeks) metal removal is reduced by 15–20%
Metal removal [mg of Cd(II) removed per g of ICSX/viscose or, mmol of ICSX/SSX]	High	Low
Process operation	Easy	Quite difficult
Sensitivity to dose	Nil	Yes

^a 1 U.S. \$ ≈ Rs. 15.50.

Yield of ICSX and SSX. The yield of ICSX and SSX (viscose) are 1.183 and 4.748 g/g of cornstarch used, respectively. The yield of SSX (viscose) is approximately four times greater than ICSX.

Relative Chemical Costs in Synthesizing ICSX/SSX. Based on the cost of chemicals required for the synthesis of ICSX and SSX, the total cost of chemicals required in synthesizing ICSX is 76 times higher than that for SSX (viscose). The washing step in the ICSX synthesis is responsible for the higher chemical's cost. However, it should be noted that such an extensive washing and drying step may not be essential at commercial scale. If the washing step is modified or eliminated from the synthesis process of ICSX, the cost of chemicals can be brought down considerably.

Time Required for Synthesis of ICSX/SSX. Generally, the synthesis of ICSX takes around 90–110 h whereas 3–4 h are sufficient for SSX (viscose) synthesis. Since the xanthation step for ICSX and SSX are almost the same, higher time requirements for ICSX is mainly due to crosslinking and washing and drying steps.

Storage. Generally, the final products (ICSX, viscose) are stored at low temperature (6–8°C). However, viscose shows 15–20% less metal uptake after 3 weeks of storage (even though degree of substitution does not vary considerably,^{18,19} whereas ICSX removes metal effectively even on prolonged storage (6–8 months).

Metal Removal by ICSX/SSX. A summary of the experimental results of Cd-ICSX/SSX interaction is shown in Table IV. The Cd-SSX results are adopted from Lokesh.¹⁷ When a comparison is made on the basis of milligrams of Cd(II) removed per gram of ICSX (or viscose) or mmoles of ICSX (or SSX), a unique feature is observed. At all pH values (3 to 7), milligrams of Cd(II) removed per gram or mmoles of ICSX is very high as compared to milligrams of Cd(II) removed per gram of viscose or mmoles of SSX. Hence, on the basis of metal uptake, ICSX is much superior to SSX.

Process Operation. The Cd-ICSX process is easy to operate in the sense that the solids can be separated by gravity settling. Hence, no additional efforts are required to separate the solids from the

Table IV Summary of the Experimental Results of Cd-ICSX/SSX Interaction^a

pH	ICSX				SSX			
	Dose (g/liter)	Cd(II) Removal (%)	mg of Cd(II) Removed per		Dose (g/liter)	Cd(II) Removal (%)	mg of Cd(II) Removed per	
			g of ICSX	mmol of ICSX			g of Viscose	mmol of SSX
3.0 ± 0.3	0.12	27.67	22.42	25.62	0.80	56.02	5.00	16.67
	0.24	44.86	18.17	20.76	1.60	73.95	3.30	11.00
	0.36	69.65	18.81	21.49	2.40	76.05	2.26	7.54
	0.48	87.86	17.79	20.33	3.20	79.97	1.78	5.95
4.0 ± 0.3	0.12	33.81	29.50	33.71	0.87	65.97	5.41	18.12
	0.24	58.45	25.50	29.14	1.81	90.06	3.55	11.91
	0.30	74.50	26.00	29.77	2.68	87.96	2.34	7.85
	0.40	94.08	24.62	28.14	3.58	93.98	1.87	6.27
5.0 ± 0.6	0.06	17.35	29.67	34.23				
	0.16	40.55	26.00	29.71	1.00	78.01	5.57	18.57
	0.28	71.54	26.21	29.96	2.00	86.00	3.07	10.23
	0.40	91.72	23.52	26.88				
6.0 ± 0.5	0.12	35.66	30.25	34.57	1.10	79.97	5.19	17.30
	0.28	73.58	26.75	30.57	2.14	56.02	1.87	6.25
	0.44	88.31	20.43	23.35	4.21	71.00	1.20	4.02
	0.60	93.81	15.92	18.19	6.29	89.08	1.01	3.38
7.0 ± 0.1	0.12	38.29	32.42	37.05	1.10	78.99	5.13	17.09
	0.24	67.72	28.67	32.76	2.20	83.05	2.69	8.98
	0.36	81.99	23.14	26.44	3.30	86.97	1.88	6.27
	0.80	93.50	11.87	13.57	4.45	93.98	1.51	5.04

^a Initial Cd(II) conc. used for ICSX/SSX system = 7.00 – 10.00 mg/liters.

liquid. However, the Cd-SSX process forms a colloidal suspension (precipitate) in aqueous system that does not settle by gravity in a reasonable length of time. This is a major drawback with Cd-SSX process particularly for cadmium metal removal. In the earlier study¹⁷ and in the present study, centrifugation process was employed to separate the precipitate from liquid phase. This process may prove to be expensive at the industrial scale. As an alternate, it is necessary to coagulate the precipitate and separate from liquid phase by sedimentation.

Sensitivity of Cd-ISX/SX Process. The Cd-SSX process is highly sensitive to SSX doses. Above and below optimum dose, metal removal is severely affected. Lokesh¹⁷ observed that there exists an optimum dose of SSX for maximum cadmium removal that is a function of both pH and initial cadmium concentration. The variation in equilibrium aqueous phase cadmium concentration or the removal of cadmium with SSX concentration is more systematic (follows a typical trend) at lower

pH values (pH < 6), while at higher pH values, the equilibrium cadmium concentration in aqueous phase seems to be independent of SSX concentration in the range of SSX doses used. On the contrary, the Cd-ICSX process does not show any optimum dose.

In light of the preceding discussion, it appears that the insoluble xanthate process has an edge over the soluble xanthate process (Table III) in terms of metal removal capacity, reliability, and ease of operation, particularly for cadmium removal. However, the soluble xanthate process appears to be relatively less expensive as compared to insoluble xanthate.

CONCLUSION

Based on the results of the present investigation and the synthesis of the available scientific information derived from review of the relevant literature, the following conclusions may be drawn.

1. Irrespective of the pH (in the range of 3 to 7), ionic strength, and initial cadmium concentrations, it is possible to bring down the aqueous phase cadmium concentration to less than 1 mg/liter using starch xanthate.

2. At lower pH values (3 to 5), the ICSX's metal binding capacity is constant, irrespective of equilibrium metal concentration and different doses of ICSX used for metal removal. This is not true at higher pH (6 and 7).

3. The ionic strength has no effect on the cadmium binding capacity of ICSX as well as SSX.

4. The aqueous phase pH influences the cadmium binding capacity of ICSX significantly, showing maximum uptake of metal in the pH range of 4 to 5.

5. The metal uptake by ICSX/SSX in the presence of EDTA is affected considerably. This effect is less with ICSX as compared to SSX in the pH range of 4 to 5. However, this trend is reversed at lower pH ($\text{pH} \approx 3$), while at higher pH (6–7) both ICSX and SSX do not remove cadmium significantly.

6. The insoluble xanthate process appears to have an edge over soluble xanthate process in terms of metal removal capacity, reliability, and ease of operation, particularly for cadmium removal.

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