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Coagulation–flocculation studies of tannery wastewater using combination of alum with cationic and anionic polymers

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A B S T R A C T

A study was conducted to treat the tannery wastewater through coagulation–flocculation–sedimentation. Alum was used as coagulant with cationic and anionic polymers as coagulant aid. The results were subsequently compared with the study in which alum was used alone for the treatment. Jar test apparatus was used to conduct research work. The results of the study revealed that the combination of alum with cationic polymer C-492 [molecular weight (MW) = 6 million Dalton; charge density (CD) = 40%] resulted in effluent turbidity removal of 97%, total suspended solids (TSS) removal of 93.5%, total chemical oxygen demand (TCOD) removal of 36.2% and chromium removal of 98.4%. Sludge production was 40 mL/L and cost of chemicals to treat one cubic meter of wastewater was $ 0.07. For this combination the optimum dose of alum was 100 mg/L as $Al_2(SO_4)_3$ with 5 mg/L of C-496. The combination of alum with suitable anionic polymer A-100 (MW = 15 million Dalton; CD = 16%) resulted in effluent turbidity removal of 99.7%, TSS removal of 96.3%, TCOD removal of 48.3% and chromium removal of 99.7%. Sludge production was 30 mL/L and cost of chemicals to treat one cubic meter of wastewater was $ 0.08. The results of the above combinations were compared with those when alum was used alone for the treatment. The comparison revealed that use of coagulant aid reduced sludge volume by 60–70% and cost of chemicals by 50% for comparable removal efficiencies.

1. Introduction

Strict enforcement of environmental regulations in Pakistan has forced tanning industry to improve its effluent quality and comply with the environmental standards. The wastewater from tanning processes is quite strong with respect to certain pollutants. It contains high organic, solids and chromium contents [1]. A substantial portion of these pollutants is in particulate form and thus can be removed at the primary step of wastewater treatment. Enhanced removal of pollutants at the primary step of wastewater treatment using coagulation–flocculation–sedimentation is normally referred to as chemically enhanced primary treatment (CEPT).

A number of workers have applied CEPT for the treatment of tannery wastewater using metal salts alone and obtained appreciable removals of various pollutants. Haydar and Aziz [2] reported alum as a suitable metal salt for tannery wastewater with an optimum dose of 240 mg/L as $Al_2(SO_4)_3$ [416 mg/L as $Al_2(SO_4)_3{\cdot}14H_2O$]. They reported percentage removal of turbidity, total suspended solids (TSS), chemical oxygen demand (COD) and chromium within a range of 98.8–99.8%, 94.3–97.1%, 53.3–60.9% and 98.9–99.7%, respectively. At optimum dose alum generated an effluent with turbidity 2.3–15 NTU, TSS 30–60 mg/L, COD 720–1200 mg/L and chromium 0.2–0.8 mg/L. Additional sludge normally referred as CEPT sludge was 100 mL/L at optimum dose of alum. Similarly, Song et al. [3] used both alum and ferric chloride for the treatment of tannery wastewater and reported reasonable removals of TSS, COD and chromium at an optimum dose of 800–900 mg/L for both alum and ferric chloride. It has been observed that sludge produced by metal salts is normally porous, incompact and difficult to dewater having high moisture contents of 99–99.7% [4].

The combination of metal salts as coagulants with anionic polymers as a coagulant aid for CEPT of tannery wastewater also exhibited good removal efficiencies for various pollutants [5–7]. However, all these workers were silent about the specific details regarding molecular weight and charge density of the anionic polymers used. Polymers are waste specific and not all the polymers can be used for every wastewater. Thus it is essential to disseminate the specific details of the polymers as mentioned above. Nevertheless, the use of anionic polymers with metal salts has been reported to result in a number of benefits. It gives less sludge volumes. Bigger and compact flocs are achieved with greater settling rates, allowing use of higher surface overflow rates for primary settling tank [8–11]. The Sludge produced is low in moisture contents with good dewaterability [12]. Floc strength is quite high and can be ascribed to the strong bonds of polymer chains due to bridging [13].

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Table 1
Wastewater quality parameter for raw homogenized SLW wastewater.

Parameter[a]	N[b]	Minimum	Maximum	Mean	S.D.[c]
pH	34	7.55	9.66	8.98	0.5
Total suspended solids (TSS)	34	568	2132	1232.7	277.4
Settleable solids (SS) (mL/L)	34	20	88	43.7	14.7
Total alkalinity (as $CaCO_3$)	34	520	1720	1227.3	311.2
Calcium hardness (as $CaCO_3$)	34	208	700	404	119.4
Chlorides	34	1000	4549	3067.2	354.7
Chromium	31	22.9	122.4	68.1	24.5
Total 5-day biochemical oxygen demand (TBOD)	34	390	1320	774.9	225.9
Soluble BOD (SBOD)	34	200	765	526.6	139.5
Total chemical oxygen demand (TCOD)	34	1760	3320	2442.4	376.9
Soluble COD (SCOD)	34	740	2040	1326.8	300.9

[a] All parameter except pH in mg/L if not specified.
[b] Number of samples.
[c] Standard deviation.

Little work has been reported on the application of metal salts with cationic polymers for CEPT of tannery wastewater. Thus the present study was conducted with the following objectives: (1) to evaluate the feasibility of using cationic polymers as coagulant aid with alum (2) to identify a suitable anionic polymer to be used as coagulant aid with alum and (3) to compare the sludge production, cost of treatment and efficiency of three different CEPT options i.e., (a) use of alum alone; (b) use of alum with cationic polymer as coagulant aid; and (c) use of alum with anionic polymer as coagulant aid.

2. Materials and methods

2.1. Sampling

Wastewater samples were collected from Saddiq Leather Works (SLW) situated in Sheikhupura, Pakistan. Samples were collected from the equalization tank, which was equipped with dome type aerators at its bottom for the purpose of mixing and homogenization. Various quality parameters of SLW wastewater were determined according to the procedures laid down in the Standard Methods [14] and are summarized in Table 1.

2.2. Polymers tested

The improvement in removal by alum while using polymers as a coagulant aid was studied by varying the alum dose and adding a fixed dose of a specific polymer. The optimal combination of alum and polymer doses, however, required varying both the alum and the polymers doses. This aspect of the work was left for a future study.

Cationic and anionic polymers were arranged free of cost from Cytec Industries Inc. (U.K). All these polymer were in dry form. In order to select a fixed dose of cationic polymer, the studies conducted by Haydar and Aziz [15] were used as a guideline. In these studies the treatment of tannery wastewater was carried out using cationic polymers alone, without any metal salts. After testing eleven different cationic polymers of varying molecular weight (MW) and charge density (CD), Haydar and Aziz recommended the use of a polymer with MW of 6 million Dalton (mDa) and CD of 40% at an optimum dose of 20 mg/L. Keeping in view this optimum dose, it was decided to use 5 mg/L of the above cationic polymer as coagulant aid in combination with varying doses of alum in the present study.

For the selection of anionic polymer dose, studies conducted by Haydar [16] were drawn upon. He examined 10 different anionic polymers for tannery wastewater and found that the use of these polymers alone resulted in marginal removal of turbidity with maximum removal occurring at a dose of 5 mg/L. Thus it was decided to use this dose for the present study. Among the anionic polymers tested by Haydar [16], two anionic polymers, one with CD of 16% and other with CD of 50% both having MW = 15 mDa were selected for the purpose of testing. Table 2 lists the polymer name/number, MW, CD and price per kg including transportation charges for all the polymers used in this study.

All the polymers were linear polyacrylamide with methyl acrylate copolymer for cationic group and acrylate copolymer for anionic group. For the purpose of administering a specific polymer dose, 0.1–0.2% polymer stock solution and for alum, 1% stock solution was prepared using distilled water. Alum is always available with some water of hydration which may vary within a range of 14–18 H_2O molecules. Due to this variation, doses of alum in this study were expressed both without water of hydration i.e., as $Al_2(SO_4)_3$ and as $Al_2(SO_4)_3{\cdot}14H_2O$, which is the most commonly available commercial alum composition.

2.3. Jar test methodology

Phipps and Bird Six Paddle Stirrer with a programmable unit and illuminated base was employed to simulate the coagulation–flocculation–sedimentation process in the laboratory. The jars had a capacity of 2-L and were equipped with a sampling port, 10 cm below the water line. The port allows sampling of the treated effluent after the jar test. In this study, standardized mixing speeds and durations were used which are reported in the literature [17–20].

For each jar test, 2-L of tannery wastewater was taken in the jars and rapidly mixed at 300 rpm for 1 min. The required doses of the coagulant and coagulant aid were added to the jars during the rapid mixing. A wooden stand with six test tubes having the required polymer dose was used to simultaneously administer the dose to all the jars [21]. The rapid mix was followed by tapered flocculation at 60 rpm for 5 min, 40 rpm for 5 min and 20 rpm for 10 min.

Table 2
Polymer name, charge, molecular weight, charge density and price per kg.

Sr. no.	Name/no.	Cationic/anionic	M.W. (million Dalton)	Charge density (%)	Price per kg including transportation charges ($)
1.	C-496	Cationic	6	40	4.98
2.	A-100	Anionic	15	7	3.33
3.	A-150	Anionic	15	50	3.33

Afterwards a settling time of 30 min was given before drawing the sample. Jar number 1 in all the jar tests was used as a control jar or "zero chemical jar". The coagulant or coagulant aid was not added to this jar to simulate plain sedimentation. Jar tests were performed using raw homogenized tannery wastewater without pre-settling in the following series.

2.3.1. Series 1

Studies conducted by Haydar and Aziz [2] showed alum to be a suitable coagulant for SLW wastewater. Therefore, in series 1, the suitable cationic polymer i.e., C-496 was combined with alum. Alum was added at the start of rapid mix and cationic polymer after a lapse of 30 s. This was done to neutralize the negative charge on colloids with alum and afterwards the polymer was used to condition the flocs formed. The dose of alum was varied from 0 to 140 mg/L. A fixed dose of 5 mg/L of the suitable cationic polymer, C-496, was added to all the jars except the "zero jar". Turbidity, TSS, TCOD and chromium determinations were made on the treated effluent to evaluate the performance of the coagulant and the coagulant aid. In order to ascertain the efficacy of C-496, two jar tests were initially run using turbidity as the test parameter. One jar test was with alum alone and the other with a combination of alum and a fixed dose of C-496.

2.3.2. Series 2

This series consisted of a preliminary jar test to select a suitable anionic polymer for use in combination with alum. Alum was added at the start of the rapid mix and the anionic polymer after a lapse of 30 s. The anionic polymer was added after alum, as suggested by Aguilar et al. [22] whose study was conducted on slaughterhouse wastewater and the results displayed greater efficiency due to negative charge of colloids. In the current study, the alum dose was varied from 0 to 180 mg/L and a fixed dose of 5 mg/L of the anionic polymer was added to all the jars except "zero jar". Turbidity of the treated effluent was measured to gauge the efficiency of the coagulant and the coagulant aid.

2.3.3. Series 3

Series 3 consisted of detailed jar test which was run on the basis of information obtained from series 2. The alum was combined with suitable anionic polymer to study the removal of TSS, TCOD and chromium from the treated effluent. The alum dose was varied from 0 to 180 mg/L with a fixed dose of 5 mg/L of the suitable anionic polymer selected from series 2.

3. Results and discussion

3.1. Series 1

The results of series 1 jar test have been graphically presented in Figs. 1–4. Fig. 1 shows the residual turbidity using alum with and without the use of C-496. It is evident from the figure that turbidity removal was enhanced by the use of C-496. A maximum removal of 97% with respect to raw homogenized wastewater occurred at an alum dose of 100 mg/L with 5 mg/L dose of cationic polymer C-496. The effluent turbidity at these doses was 35 NTU. At alum doses above 100 mg/L, there was no further appreciable reduction in turbidity.

Similarly it can be observed from Figs. 2–4 that a maximum removal of TSS, TCOD and chromium occurred at an alum dose of 100 mg/L with 5 mg/L of C-496. At these doses of coagulant and coagulant aid, removal of TSS was 93.5% (effleuent TSS = 70 mg/L), removal of TCOD was 36.2% (effluent TCOD = 1720 mg/L) and chromium removal was 98.4% (effluent chromium = 0.9 mg/L). The enforced effluent standards in Pakistan for TSS, COD and chromium are 200 mg/L, 150 mg/L and 1.0 mg/L, respectively. Thus, CEPT at the

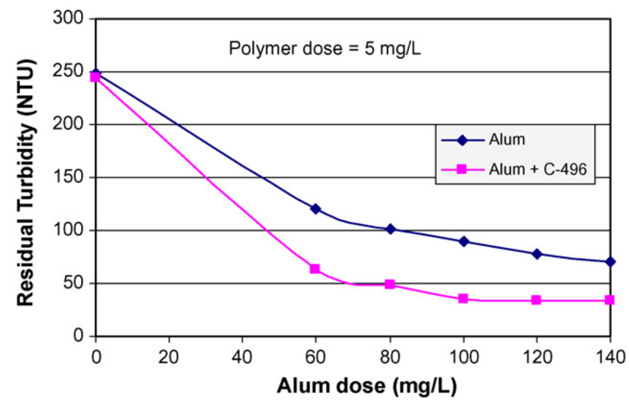

Fig. 1. Effect of alum and cationic polymer C-496 dose on residual turbidity.

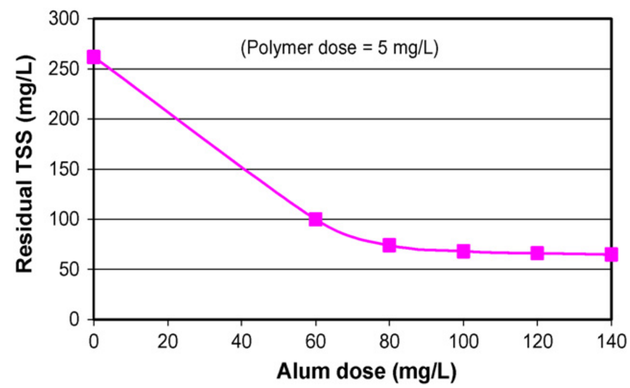

Fig. 2. Effect of alum and cationic polymer C-496 dose on residual TSS.

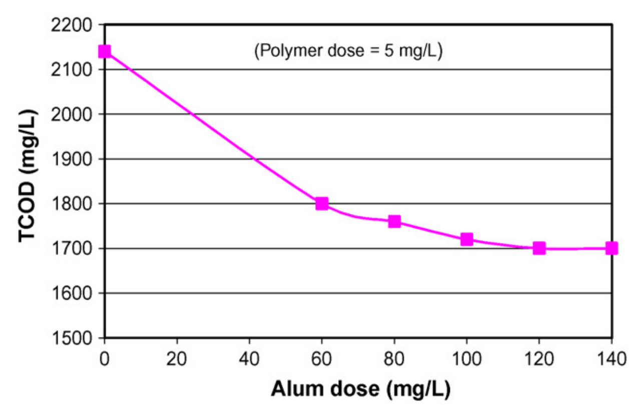

Fig. 3. Effect of alum and cationic polymer C-496 dose on residual TCOD.

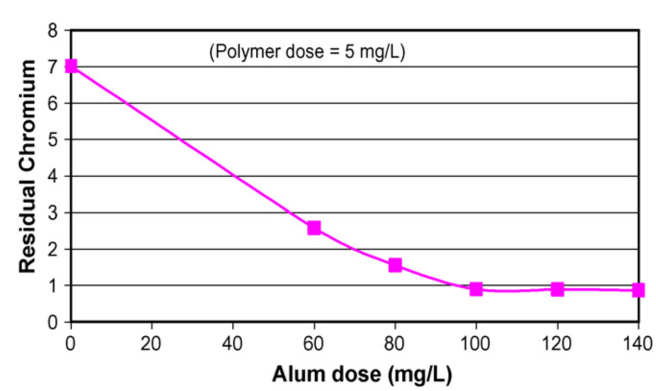

Fig. 4. Effect of alum and cationic polymer C-496 dose on residual chromium.

optimum doses of alum with a fixed dose of cationic polymer was successful in meeting effluent standards for TSS and chromium. COD was high and required a secondary treatment, like the activated sludge process, to meet the effluent standards [23]. The soluble COD for the sample used for the jar test was 1720 mg/L which shows that all COD remaining after CEPT was in the soluble form. The possible constituents of this soluble COD may be dissolved fats from hides and dyes used in the tanning process. Amongst these constituents, dyes are toxic.

3.2. Series 2

The objective of series 2 jar tests was to identify a suitable anionic polymer that could be combined with alum for CEPT of tannery wastewater. For this purpose three jar tests were conducted. One was conducted with alum alone as coagulant. In the second jar test alum was combined with anionic polymer A-150 and in the third jar test alum was combined with anionic polymer A-100. The results of these jar tests have been presented in Fig. 5. It can be seen in the figure that the curves for alum alone and alum with A-150 overlapped showing that A-150 did not help in enhancing the removal of turbidity. The use of alum with A-100 showed improvement in the removal of turbidity. Moreover, the addition of A-100 resulted in the formation of big flocs while no flocs were observed with A-150. The possible reason to this effect may be the high CD of A-150, which instead of conditioning the flocs reversed the neutralized charge on the small flocs produced by alum. Thus it can be concluded that anionic polymer of low CD (16%) could be effective when used in combination with alum. Moreover, the optimum dose of alum from Fig. 5 appears to be 160–180 mg/L with 5 mg/L dose of A-100. At these doses the percentage removal of turbidity was 99.4% (effluent turbidity = 7.25 NTU).

3.3. Series 3

This consisted of a detailed jar test with suitable anionic polymer i.e., A-100 in combination with alum. The results are presented in Figs. 6–8. Fig. 6 shows the effect of varying alum dose on residual TSS with a fixed dose of 5 mg/L of anionic polymer A-100. It can be seen that maximum percentage removal of 96.3% for TSS occurred at an alum dose of 160 mg/L (effluent TSS = 40 mg/L). No further appreciable reduction in TSS could be observed at higher dose of alum. Similarly, in Fig. 7 maximum percentage removal of 48.3% for TCOD occurred at an alum dose of 180 mg/L (effluent TCOD = 1280 mg/L). Maximum percentage removal of 99.7% for chromium, in Fig. 8, could be observed at an alum dose of 160 mg/L (effluent chromium = 0.2 mg/L). Thus it can be concluded that optimum alum dose when used in combination with anionic polymer is 160 mg/L with 5 mg/L of A-100 (MW = 15 mDa; CD = 16%). It can be seen that the effluent standards for TSS and chromium were met at the optimum doses of coagulant and coagulant aid while COD was high and required secondary treatment. The soluble COD of the wastewater sample used for series 3 jar test was 1320 mg/L. It shows that some portion of soluble COD was also removed at the optimum doses. This could be due to the adsorption of soluble COD on $Al(OH)_3$ gel formed due the use of alum.

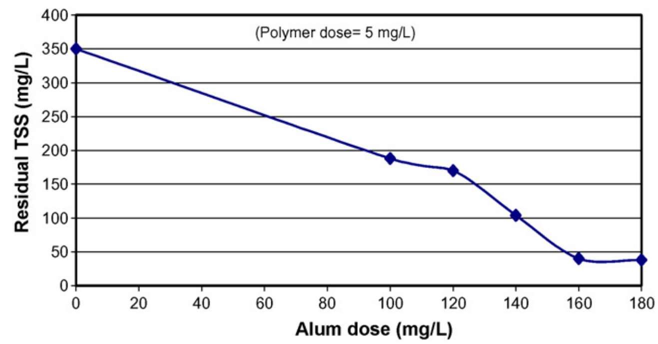

Fig. 6. Effect of alum and anionic polymer A-100 dose on residual TSS.

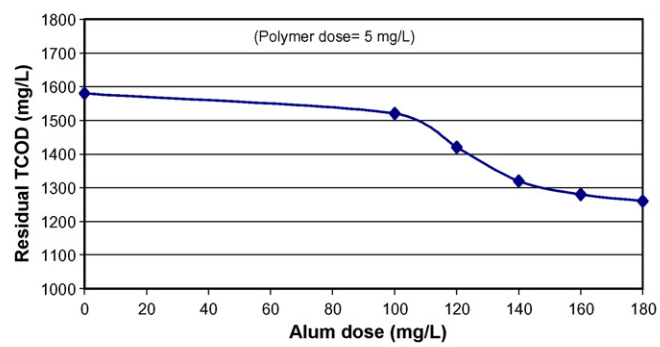

Fig. 7. Effect of alum and anionic polymer A-100 dose on residual TCOD.

3.4. Sludge production

Sludge handling and treatment constitute a major portion of both capital and operational cost in wastewater treatment. Additional sludge volumes result with CEPT as compared to plain sedimentation due to two reasons. Firstly, additional sludge is formed due to enhanced removal of suspended and colloidal particles when compared with plain sedimentation. Secondly, a portion of additional sludge volume is due to chemical sludge, which is formed especially in case of metal salts due to the formation of metal hydroxide. The additional volume of sludge produced in CEPT

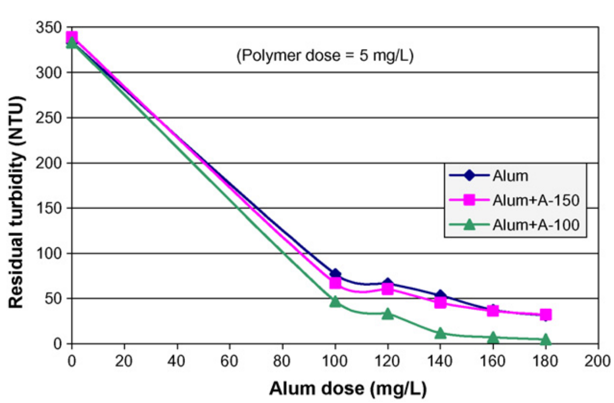

Fig. 5. Effect of alum and anionic polymer dose on residual turbidity.

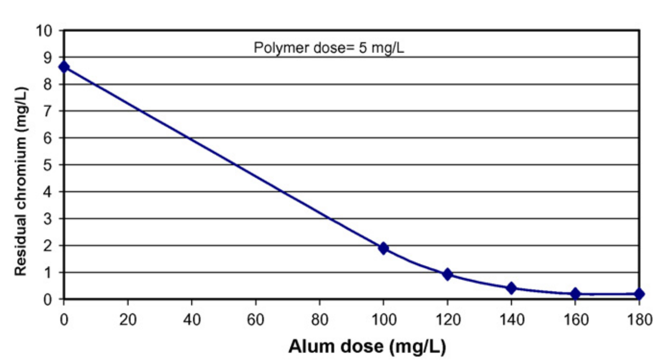

Fig. 8. Effect of alum and anionic polymer A-100 dose on residual chromium.

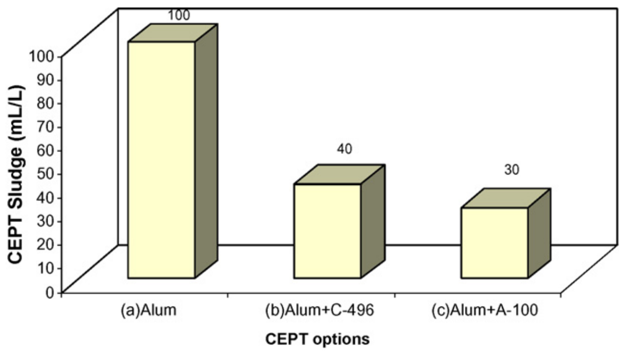

Fig. 9. Sludge production in different CEPT options.

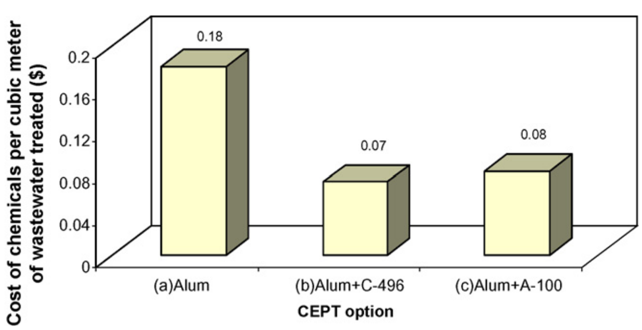

Fig. 10. Cost of chemicals to treat one cubic meter of SLW wastewater for different CEPT options.

has been referred to as CEPT sludge here. This CEPT sludge volume was measured in series 1 and series 3 jar tests using graduations on the jars. Sludge production for SLW wastewater was reported by Haydar [16] using alum alone as the coagulant. Fig. 9 shows the sludge production in mL/L for the jar having optimum dose for three different CEPT options i.e., (a) when alum was used as sole coagulant; (b) when alum was used in combination with cationic polymer; and (c) when alum was used in combination with anionic polymer.

It is evident from Fig. 9 that the use of alum alone resulted in increased sludge volume and thus sludge handling cost. At the optimum dose of alum i.e., 240 mg/L as $Al_2(SO_4)_3$ [416 mg/L of alum as $Al_2(SO_4)_3{\cdot}14H_2O$] the sludge produced was 100 mL/L [16]. The use of coagulant aids (cationic and anionic polymers) significantly reduced the sludge volumes. There was a reduction of 60% and 70% in sludge volumes for option (b) and (c), respectively when compared with option (a).

The sludge in option (b) and (c) was mainly composed of organic and inorganic solids, aluminum hydroxide gel, polymer and chromium. Among these components aluminum [24], cationic polymer [25] and chromium [26,27] are toxic. The cationic polymer was toxic at the dose administered while the anionic polymer was not [25]. Thus the sludge contained toxicity in the form of aluminum, the cationic polymer and chromium and needed to be disposed of carefully.

3.5. Cost of chemicals for CEPT

Based on the suggested dose of alum alone for CEPT of tannery wastewater as reported by Haydar [16] and the doses of alum and cationic/anionic polymers found in this study, the cost of chemicals for the treatment of one cubic meter of tannery wastewater was also evaluated. The cost of polymers per kg has been given in Table 2 and cost of alum per kg was $ 0.43. A cost comparison for three CEPT options has been shown in Fig. 10.

Table 3
Comparison of raw wastewater characteristics, treated effluent and percentage removals for various parameters for three CEPT options.

	CEPT options		
	Alum [2] (a)	Alum + C-496 (b)	Alum + A-100 (c)
Turbidity (NTU)			
Raw	1370	1184	1302
Treated	2.3	35	7.23
Removal (%)	99.7	97	99.4
TSS (mg/L)			
Raw	1508	1078	1104
Treated	54	70	40
Removal (%)	96.4	93.5	96.3
TCOD (mg/L)			
Raw	2400	2700	2480
Treated	1120 (1200)[a]	1720 (1720)[a]	1280 (1320)[a]
Removal (%)	53.3	36.2	48.3
Chromium (mg/L)			
Raw	77	56.9	70.3
Treated	0.8	0.9	0.2
Removal (%)	98.9	98.4	99.7

[a] Soluble COD.

Fig. 10 shows that the combination of alum with cationic polymer is the most economical CEPT option with a chemical cost of $ 0.07 per cubic meter of wastewater treated. It was followed by combination of alum with anionic polymer with a chemical cost of $ 0.08 per cubic meter of wastewater treated. The chemical cost of alum, if used alone, was almost double than the rest of the two CEPT options. Thus the use of alum alone is expensive and will generate extra sludge and result in more financial burden in terms of sludge handling.

3.6. Comparison of removal efficiencies of three CEPT options

A comparison of the raw wastewater characteristics, treated effluent and percentage removals of various parameters has been presented for the three CEPT options in Table 3. It can be seen in Table 3 that for turbidity, TSS and TCOD, the percentage removal for option (a) and (c) are better than option (b). For the removal of chromium, the results were quite close for all three options. However, all the above mentioned differences are minor with respect to removal efficiency and the quality of treated effluent. Therefore, from Table 3 it can be concluded that with respect to the removal of various pollutants, the three options are comparable. It may further be noted that the removal of some portion of soluble COD was observed in option (a) and (c) while TCOD in option (b) is equal to soluble COD. Therefore, CEPT remove all particulate COD and only soluble COD is left behind.

4. Conclusions

It can be concluded from the present study that the use of cationic and anionic polymers as coagulant aid with alum is highly feasible for CEPT of tannery wastewater. The use of alum alone is neither economical with respect to the chemical cost nor with respect to the resulting higher sludge volumes. With 5 mg/L of cationic polymer C-496 [MW = 6 mDa; CD = 40%], the optimum dose of alum was found to be 100 mg/L as $Al_2(SO_4)_3$ [173.6 mg/L as $Al_2(SO_4)_3{\cdot}14H_2O$] for maximum removal of pollutants. Using 5 mg/L of anionic polymer A-100 [MW = 15 mDa; CD = 16%], the optimum dose of alum was found to be 160 mg/L as $Al_2(SO_4)_3$ [294.73 as $Al_2(SO_4)_3{\cdot}14H_2O$] for obtaining maximum reduction in pollutants. A comparison of this study with that when alum was used alone for the same tannery wastewater revealed that for the comparable removal efficiencies for various pollutants, the use of coagulant aids reduced the chemical cost per cubic meter of wastewater treatment by almost 50%. Furthermore, it also reduced the amount of sludge volume by 60–70% thereby reducing the sludge handling cost.

Acknowledgements

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