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Elimination of chemical sludge for treatment of tannery wastewater and its effect on biogas generation

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ABSTRACT

Disposal of sludge generated during treatment of tannery wastewater poses a major challenge owing to scarcity of land available for implementation of secured landfill facilities. Considering the magnitude of the problem, the present study focused on reduction of the volume of chemical sludge generated during physico-chemical treatment of wet-blue to finish tannery wastewater, by adopting plain sedimentation followed by anaerobic treatment for biogas generation. Apart from monitoring the effect of suspended solids on biogas generation, the effect of chromium concentration and COD/SO_4 ratio on biogas generation was also investigated in detail. During anaerobic treatment of composite tannery wastewater without plain sedimentation, biogas generation of 204 mL/g of $COD_{removed}$ was observed. Whereas, with 2 h settlement, the biogas generation observed was 255 mL/g of $COD_{removed}$. The amount of sludge generated during treatment of 1 m^3 of tannery wastewater with plain sedimentation for a period 2 h was only 0.5 kg/m^3, whereas with addition of chemical coagulant and coagulant aids, it was about 3.03 kg/m^3. Hence, for the tannery wastewater generated from wet-blue to finish tanning operations, plain sedimentation followed by anaerobic treatment is an environmental friendly option for elimination of chemical sludge.

Keywords: Biogas; Chemical oxygen demand; Chromium; Plain sedimentation; Suspended solids

1. Introduction

With stringent environmental regulations in place, disposal of sludge poses a big challenge and is a major bottleneck in wastewater treatment plants all over the world. The demand for development of technologies for minimization of sludge generation is of budding interest. Tannery wastewater is characterized by higher concentration of organic and inorganic pollutants [1,2]. Treatment of tannery wastewater by physico-chemical processes and membrane bioreactor for reuse was reported by Fettig et al. [3]; Lafrano et al. [4]. Enhanced coagulation process has been investigated for primary treatment of tannery wastewater for removal of pollutants such as total suspended solids (TSS), total chemical oxygen demand (TCOD) and chromium using aluminum sulfate as coagulants [5]. An integrated process of upflow anaerobic sludge blanket reactor, sequential batch reactor, electro oxidation and biological aerated filter was investigated for treatment of tannery wastewater by Liu et al. [6]. Treatment of tannery wastewater using hybrid upflow anaerobic sludge blanket reactor at two hydraulic retention times i.e., 70 and 60 h were studied, and 70 h HRT had shown better performance in removal of pollutants [7].

In India, tannery wastewater is treated by adopting physico-chemical treatment followed by biological treatment, either in individual effluent treatment plants (ETPs) or in common effluent treatment plants (CETPs). Overall, during the treatment process, a huge quantity of chemical and biological sludge is generated. Also, the cost of disposal of sludge is quite high and accounts for nearly 30–40% of the operating cost of wastewater treatment plants. Reduction of sludge generation is an urgent need in the tanning sector, owing to scarcity of land available for implementation of secured landfill facilities (SLF) for safe disposal of sludge.

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Reduction of sludge production from the wastewater treatment process rather than the post-treatment or disposal of the sludge was suggested by Hansen et al. [8]; Englande and Reimers [9]. Hyder and Aziz [10] reported that addition of cationic and anionic polymer as coagulant aid in conjunction with Aluminum Sulfate as coagulant reduced the chemical cost by 50% as well as sludge volume by 60–70%. Various sludge reduction technologies such as alkaline–thermal treatment, lysis-cryptic growth, activated sludge-ozonation process, chlorination-combined activated sludge process, sludge reduction by metabolic uncouplers for high dissolved oxygen activated sludge process have been reviewed for reduction of biological sludge [11,12]. The effect of ozone on sludge reduction was investigated for aerobic bio-oxidation of Phenolic wastewater and around 50% sludge yield was reduced with ozone dose of 30 mg O_3/L/d [13]. Influence of thermo-chemical pretreatment on sludge reduction in a bench-scale anoxic-aerobic membrane bioreactor was investigated for domestic sewage and around 33% reduction in sludge generation was reported [14]. The effect of sono-bio stimulation process was investigated on activated sludge process for minimization of biological sludge generation [15]. Around 50 and 22.5% of sludge yield was reduced by addition of 3,3,4,5-tetrachlorosalicylanilide (TCS) and tetrakis (hydroxymethyl) phosphonium sulfate (THPS) as metabolic uncoupler for the activated sludge cultures [16,17]. Preparation of activated carbon from waste activated sludge was explored towards sludge minimization option by Martin et al. [18]. Apart from bio-energy generation, incineration and co-combustion technologies are also gaining importance for management of bio-solids [19]. Most of the sludge minimization technologies concentrate on biological sludge reduction only. Very limited studies have been carried out for reduction of chemical sludge generated during physico-chemical treatment of wastewater.

During chrome tanning and retanning processes, chromium in the form of basic chromium sulfate is used, and chromium will be in the form Cr(III). The influence of COD/SO_4 ratio in Up-flow Anaerobic Sludge Blanket (UASB) treating domestic wastewater was reported by Subtil et al. [20], who found that a small variation in COD/SO_4 ratio (from 1.1 to 1.85) promoted a significant change in sulfate reduction as exemplified by the 45% to 10% of sulfate removal efficiency. The competition between the sulphate-reducing, methanogenic and syntrophic populations after development in reactors at varying influent COD/SO_4^{2-} ratios was studied and reported that, methanogens will degrade acetate and hydrogen and propionate was the preferred substrate for sulphate reducing bacteria [21]. The effect of influent COD and upward flow velocity on the behavior of sulphate-reducing bacteria was reported by Shayegan et al. [22]. The effect of substrate and operational parameters on the abundance of sulphate-reducing bacteria in industrial anaerobic biogas digesters using PCR was reported by Moestedt et al. [23]. The effect of operational parameters on the performance of hydrolytic and acidogenic process during co-digestion of tannery and dairy wastewater was reported by Berhe and Leta [24]. Mekonnen et al. [25] reported methane recovery and greenhouse gas emission mitigation in anaerobic sequential batch reactor (ASBR) for tannery wastewater. Anaerobic co-treatment of tannery wastewater and cow dung for biogas generation was studied using ASBR and reported that tannery wastewater can be co-digested with cow dung with a mix proportion of 80:20 for enhanced biogas generation [26].

Conventional coagulation and flocculation process not only removes the suspended solids but also precipitates the chromium present in tannery wastewater. Considering the cost involved in the procurement of coagulant chemicals and disposal of sludge generated during treatment of tannery wastewater, the present study focused on application of plain sedimentation followed by anaerobic treatment for biogas generation. In the present study, the performance of the anaerobic treatment of tannery wastewater was monitored for (i) biogas generation, with and without plain sedimentation and (ii) impact of chromium and COD/SO_4 ratio on biogas generation.

2. Materials and methods

2.1. Characterization of tannery wastewater and inoculum

Wastewater samples were collected from a CETP operating for treatment of tannery wastewater, where the tannery operation covers wet-blue to finish processes. Inoculum for the study was obtained from an anaerobic reactor operating for treatment of domestic sewage. Wastewater samples and inoculum samples were characterized as per Standard Methods 20th edition [27]. The elemental analysis of inoculum in terms of carbon, hydrogen, nitrogen, sulphur on dry weight basis was done using the elemental analyzer, CHNS-O (Model- Euro EA 3000). Similarly, after plain sedimentation, settled sludge samples were analyzed for elemental analysis.

2.2. Plain sedimentation and quantification of sludge generation

Tannery composite wastewater samples were subjected to plain sedimentation without addition of chemical coagulant or coagulant aid. Settleability of suspended solids (SS) was monitored at regular intervals of time i.e., every half an hour for a period of 2 h. The percentage settlement of SS w.r.t. time is reported. Particle size distribution was measured for composite wastewater and after 2 h of plain sedimentation, with the help of particle size analyzer. In the conventional physico-chemical treatment, alum, lime and poly electrolyte are used for removal of SS during treatment of composite wastewater and the same was adopted in the present study. After settlement, the percentage removal of SS was arrived at. Based on the results obtained, the amount of sludge generated for treatment of 1 m^3 of tannery wastewater was arrived at. The sludge quantification was compared with plain sedimentation for a period of 2 h.

2.3. Experimental setup for anaerobic treatment of tannery wastewater

With and without plain sedimentation, the tannery composite wastewater was subjected to anaerobic treatment. Experiments were carried out in batch reactors of 650 mL capacity. The inoculum and composite wastewater were taken into the batch anaerobic reactors and micro and macro nutrients were added. Biogas generation was monitored by water displacement method. After ceasing of biogas generation, the anaerobic reactors were opened, and the contents were thoroughly mixed. After mixing the contents, the samples were analyzed for pH, oxidation reduction potential (ORP), bicarbonate alkalinity, volatile fatty acids (VFA) and COD. All analysis was done as per Standard Methods 20th edition [27].

2.4. Effect of chromium and COD/SO_4 ratio on biogas generation

Tannery wastewater contains chromium which is released from the chrome tanning process. In the chrome tanning process and re-chroming process, chromium in the form of Basic Chromium Sulfate ($Cr(OH)SO_4$) is used [28]. In India, chrome recovery systems (CRS) have been implemented and are mandatory for tanneries processing raw-to-finish/raw-to-wet blue tanning process. The concentration of chromium in chrome tanning wastewater is in the range of 3000–5000 mg/L. Apart from this, re-chroming process is also carried out in tanneries and the concentration of chromium after the re-chroming process wastewater is in the range of 400–800 mg/L. Chromium is not recovered from this (re-chroming) process. This sectional stream is mixed with the remaining wastewater, which results in the presence of chromium in the composite tannery wastewater. In conventional tannery wastewater treatment, during physico-chemical treatment, the residual chromium entering CETPs will be removed prior to biological treatment [29,30]. However, in the present study, chemical coagulant or coagulant aid was not added; only plain sedimentation was adopted for removal of suspended solids. Addition of basic chromium sulfate (BCS) during chrome tanning process leads to the presence of chromium content in composite tannery wastewater, and increased sulfate concentration. It is well known that the COD/SO_4 ratio plays a major role in anaerobic treatment process for generation of biogas [31,32]. Hence, studies were carried out to investigate the effect of Cr and COD/SO_4 ratio for biogas generation after plain sedimentation. The inhibitory effect of Cr and COD/SO_4 ratio on biogas generation was monitored. After ceasing of biogas generation, the contents were analyzed as per Standard Methods 20th edition [27].

3. Results and discussion

3.1. Characteristics of tannery wastewater and inoculum

Characteristics of tannery wastewater and inoculum are presented in Table 1. The quantity of chemicals used in each process and volume of water used in the tanning processes vary between tanneries. Due to this, the concentration of the pollutants present in the wastewater will also vary. It was observed from the results that the COD/SO_4 ratio was 1.85–2.08. The total chromium concentration was 30–90 mg/L, indicating chromium was contributed by the re-tanning operation. In India, chrome recovery systems have been implemented for recovery of chromium from main chrome tanning process and not from re-chroming sectional streams. Hence, the composite tannery wastewater (i.e., wet-blue to finish leather process) contains chromium, which was evident from the characteristics of the wastewater in the present study also. The biodegradation potential of wet-blue to finish wastewater at various concentrations of suspended solids, chromium and COD/SO_4 ratio was studied under anaerobic condition. Effect of these parameters on biogas production was monitored. The pH of the inoculum was around 8.2 and concentration of total suspended solids and volatile suspended solids was 38,000–42,000 mg/L and 30,000–33,000 mg/L respectively. The elemental analysis of the inoculum and sludge after plain sedimentation (2 h) in terms of carbon, hydrogen, nitrogen and sulphur, on dry weight basis, was done using the elemental analyzer. The oxygen content was calculated as suggested by Sosnowski et al. [33]. The elemental analysis results are shown in Table 2. It was observed from the results obtained from elemental analysis, primarily the CHNS contribution to sludge is due to organic matter attached to the grit material only.

Table 1
Characteristics of tannery wastewater and inoculum

Sl. No	Parameter	Range
Characteristics of wet-blue to finish wastewater		
	pH	5.5–6.5
	Chemical oxygen demand (COD, mg/L)	4000–6000
	Biochemical oxygen demand (BOD, mg/L)	1000–1700
	Sulfate as SO_4 (mg/L)	2200–3000
	Suspended solids (mg/L)	2500–3500
	Total chromium as Cr (mg/L)	30–90
	Biodegradability index	0.25–0.28
Characteristics of inoculum		
	pH	7.5–8.2
	Suspended solids(mg/L)	38000–42000
	Volatile suspended solids (mg/L)	30000–33000
	Total chromium as Cr (mg/L)	BDL

Note: BDL – Below Detectable Limit

Table 2
Elemental analysis of the inoculum and sludge after settled sludge after 2 h of plain sedimentation

Sl. No.	Parameter	Value	
		Inoculum	Settled sludge after 2 h of plain sedimentation
1	Carbon content, %	30.17	10.24
2	Nitrogen content, %	4.51	3.12
3	Hydrogen content, %	3.84	3.95
4	Sulphur content, %	19.84	11.21
5	Oxygen content, %	42.44	71.48

3.2. Settlement of suspended solids during plain sedimentation

During plain sedimentation, the rate of settlement of SS w.r.t. time was monitored and is presented in Fig. 1. With

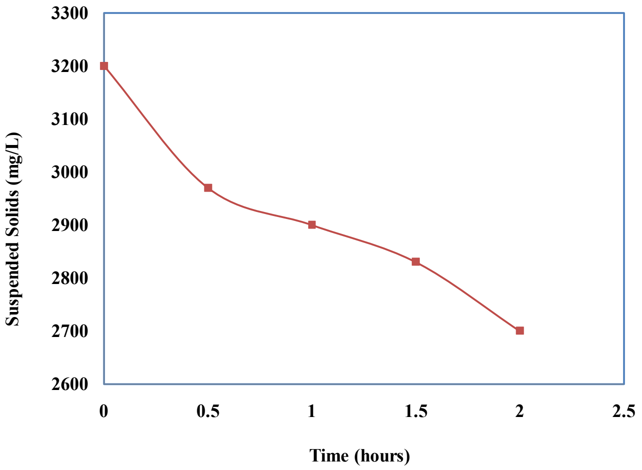

Fig. 1. Settlement of solids during plain sedimentation.

increasing settlement time, the percentage settlement of SS also increased, but there was no change in the pH. Also, as seen from Fig. 1, 7.2% of settlement of SS was observed within half an hour of settlement period, which implies that during half an hour settlement period coarse particles of bigger size could have settled down. By increasing the settlement time, SS removal efficiency increased only from 7.2 to 15.6%. It implies that in case of plain sedimentation, particle size plays a more significant role than the settlement time. However, in the present study, with plain sedimentation period of only 2 h, the removal of SS was shown to be dependent on particle size distribution. The particle size distribution analysis of composite wastewater without settlement and settlement after 2 h is presented in Figs. 2 and 3. As shown in Fig. 2, in raw composite tannery wastewater, the particle size was in the range of 122.4 (d·nm) to 1484(d·nm) and after 2 h settlement the particle size was in the range of 105.7 (d·nm) to 1281(d·nm). It was evident from the particle size distribution curves presented in Figs. 2 and 3 that large size particles were able to settle down during the plain sedimentation process. Particles of size over and above 1000 d·nm settled down partly or fully during the plain sedimentation process. During plain sedimentation, discrete spherical particles settlement takes place and it is usually employed for removal of grit. Plain sedimentation reduced the concentration of particles in suspension before the wastewater was subjected to anaerobic treatment process [34].

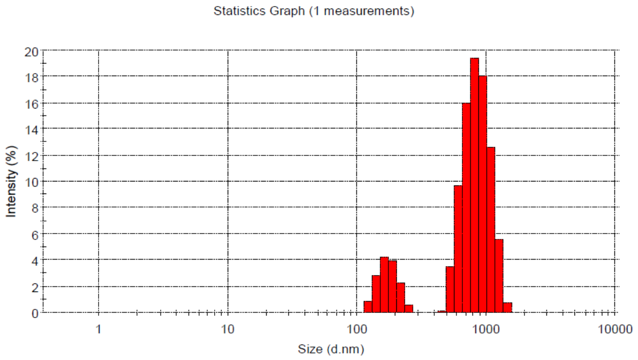

Fig. 2. Particle size distributions in raw tannery wastewater.

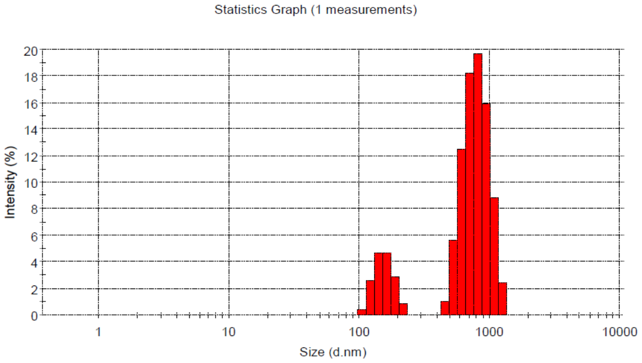

Fig. 3. Particle size distribution in after 2 h of plain sedimentation.

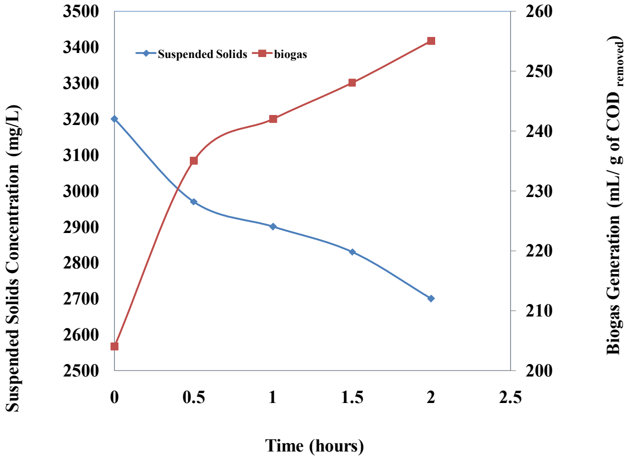

Fig. 4. Comparative analysis of biogas generation without and with plain sedimentation.

3.3. Effect of suspended solids on biogas generation

In batch reactors, composite tannery wastewater was subjected to anaerobic treatment (i) without plain sedimentation and (ii) after plain sedimentation for a period of 2 h. Biogas generation was monitored daily. Comparative analysis of biogas generation, without and with plain sedimentation, is depicted in Fig. 4. As shown in Fig. 4, in case of control reactor (composite tannery wastewater without plain sedimentation), biogas generation of 204 mL/g of COD $_{removed}$ was observed, whereas with half an hour and 2 h settlement, the biogas generation observed was 235 and 255 mL/g of COD $_{removed}$ respectively. When compared to treatment without plain sedimentation, increase in biogas generation was 15.2% with half an hour of plain sedimentation, whereas by increasing the settlement time to 2 h, only a marginal increase of 8.5% was observed. Hence, the removal of solids, especially inert in nature, has a synergistic effect on biogas generation. The behavior of plain settling during treatment of tannery wastewater was investigated and reported that 76.1% suspended solids were removed

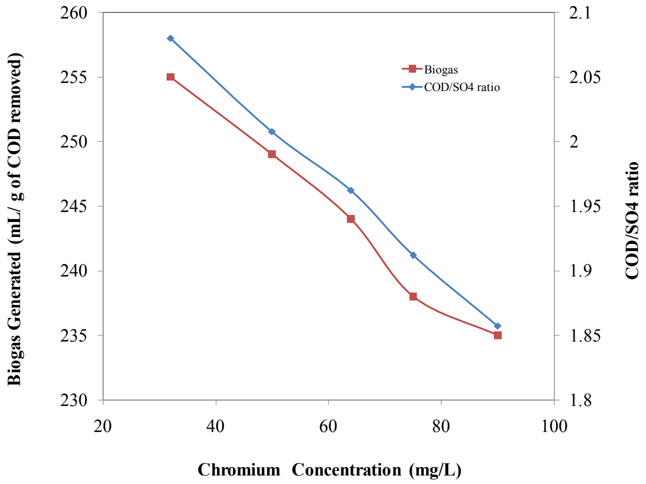

Fig. 5. Effect of chromium and COD/SO_4 ratio on biogas generation.

after 3 h of plain settling. The sludge volume was 28% of the volume of the mixed wastewater [1]. The main feature of physical process is separation of suspended solids from the liquid and that of the biological process is degradation of decomposable organic matter under anaerobic conditions. When the rate of bacterial growth is considered, the retention time of the solids becomes an important parameter. Biological treatment of wastewater basically reduces the pollutant concentration through microbial coagulation and removal of non-settleable organic colloidal solids [35].

Primarily, plain sedimentation studies were carried out for wet-blue to finish tannery wastewater only. Settlement of SS w.r.t. time was monitored. pH of wet-blue to finish tannery wastewater was in the range of 5.5–6.5. Hence, during settlement of solids, chromium content present in the wet-blue to finish tannery wastewater will not be precipitated. For removal of chromium, pH > 8.3 is needed. However, in the present study, neither adjustment of pH nor was coagulant or coagulant aid added. In order to confirm the chromium concentration in the sludge, the sludge samples were analyzed using Atomic Absorption Spectroscopy after plain sedimentation. Chromium was below the detectable limit.

For the purpose of comparison, the composite tannery wastewater was subjected to physico-chemical treatment using alum, lime and poly electrolyte for removal of SS. After physico-chemical treatment, tannery wastewater was subjected to anaerobic treatment and biogas generation of 280 mL/ g of $COD_{removed}$ was observed. At the end of the digestion period, the VFA/alkalinity ratio was in the order of 0.2–0.4 and oxidation reduction potential was – 350 to –450 mυ, which signifies the prevalence of anaerobic conditions.

3.4. *Effect of chromium and COD/SO_4 ratio on biogas generation*

As shown in Table 1, the composite tannery wastewater contained 30–90 mg/L of chromium.

Composite samples were collected at different time intervals (days) since the nature and concentration of the pollutants vary from tannery to tannery, which is reflected in the nature of the composite wastewater. The effect of Cr concentration on biogas generation was studied along with change in COD/SO_4 ratio in the composite tannery wastewater. Biogas generation was monitored, and results are presented in Fig. 5. As shown in Fig. 5, when the Cr content increased from 32 to 90 mg/L, a reduction of around 8% in biogas generation was observed. Toxicity effect of Cr(III) in anaerobic digestion process was evaluated and reported by Alkan et al. [36]. It was reported that 1140 mg/L of Cr concentration was toxic when it was injected to anaerobic digestion process in a step-wise manner. In the same study, the shock application of Cr(III) concentration in the range of 400–500 mg/L to anaerobic digestion process was also evaluated and it was observed that the performance of anaerobic digestion process had been affected.

Mechanism responsible for reduction and removal of Cr(VI) on active biomass of anaerobic sludge granules was investigated and reported that, Cr(VI) reduction and removal was attributed to combined effect of biological activity and physical characteristics of the anaerobic sludge granules [37]. The effect of heavy metals such as Hg, Cd and Cr(IIII) on biogas production during anaerobic treatment of sewage sludge was investigated. It was reported that the presence of heavy metals inhibited the production of biogas and the order of toxicity was Hg > Cd > Cr(III) [38]. Effect of metals such as Pb, Cd, Cu, Zn, Cr(III), Ni, N, K, Mg and Ca in food wastes on biogas generation was investigated and reported that, the negative effect of heavy metals on biogas production was attributed not only to the concentrations but also the oxidation state, pH of the substrate, and interaction with other compounds [39].

It is very difficult to conclude straight away whether the reduction of biogas generation is mainly attributable to the presence of higher concentrations of chromium or due to a low COD/SO_4 ratio, which is another significant factor that controls biogas generation in anaerobic digestion process. In the initial characteristics presented in Table 1, the COD/SO_4 ratio was 2.08 and when the COD/SO_4 ratio decreased, a decrease in generation of biogas was observed. When the COD/SO_4 reduced to 1.86, around 8% decrease in biogas generation was observed. Anaerobic treatment of sulfate rich wastewaters and the precautions to prevent sulfide toxicity in methanogenic reactors was demonstrated by Pol et al. [40]. When the COD/SO_4 ratio is higher than 10, it can be treated in a methanogenic system without any system failures [40]. If the COD/SO_4 ratio is above 0.67, then hypothetically, sulfate can be reduced with the available COD [41]. If the ratio falls below 0.67, then the amount of organic matter is insufficient for a complete reduction of the sulfate and extra substrate has to be added. A similar observation was made in case of enhanced amount of chromium concentration in the wastewater also. Anaerobic digestion of sulphate-rich post-tanning wastewater at different COD/SO_4 and F/M ratios was investigated and reported that COD and SO_4 removal efficiency was increased with increase in COD/SO_4 ratio from 0.62 to 1.20 [42].

The effect of pH and Sulfate concentrations on anaerobic treatment tannery wastewater was investigated and reported that, with sulfate concentration >8.45 g/L and pH < 6.4, up to 73% decrease in methane production was observed [43]. The effectiveness of natural zeolites in accelerating UASB reactor during startup was studied for high

Table 3
Comparison of conventional physico-chemical treatment vs plain sedimentation of tannery wastewater

Sl. No	Parameter	Conventional physico-chemical treatment	Plain sedimentation
	SS removed (%)	60	15.6
	COD removed (%)	40	10
	Biogas generated (mL/ gram of $COD_{removed}$)	280	255
	Primary sludge generated ($kg/m^{3)}$ of wastewater	3.03	0.50

Sulfate concentrated wastewater and reported that the process was stressful at COD/SO_4 ratio less than 3.0 and HRT of less than 3.3 h [44]. Lower COD/ SO_4 ratio of tannery effluent is an impediment in successful anaerobic treatment process [45]. Similar results were observed in the present study w.r.t COD/ SO_4 ratio.

3.5. Quantification of sludge generation

During conventional physico-chemical treatment, alum, lime and poly electrolyte are added. In the same manner, these coagulants and coagulant aids were added in the present study in order to assess the SS and COD removal efficiencies, with and without addition of chemical coagulants. After coagulation and flocculation, the SS and COD removal were monitored. Similarly, by adopting plain sedimentation for a period of 2 h, the amount of SS and COD removal was monitored. The results are presented in Table 3. It was observed that after conventional physico-chemical treatment, SS and COD removal efficiencies were 60 and 40% respectively, whereas, after plain sedimentation alone, SS and COD removal efficiencies were only 15.6 and 10% respectively. The additional SS and COD removal efficiency was attributed to addition of chemical coagulants and coagulant aids, which caused agglomeration of colloidal particles. However, as shown in Fig. 4, biogas generation of 255 mL/g of COD $_{removed}$ observed after plain sedimentation for a period of 2 h increased to 280 mL/g of COD $_{removed}$ after physico-chemical treatment. Based on the results obtained, the amount of sludge generated during treatment of 1 m^3 of tannery wastewater with plain sedimentation for a period 2 h was only 0.50 kg/m^3, whereas 3.03 kg/m^3 of sludge was generated with the addition of chemical coagulants and coagulant aids, by considering the solubility. Only an increase of 9.8% in biogas generation was observed after physico-chemical treatment was followed by anaerobic treatment. However, addition of chemical coagulants and coagulant aids generated an additional 2.53 kg/m^3 of chemical sludge, which increases the treatment cost and sludge disposal cost.

4. Conclusions

Most of the sludge minimization technologies concentrate on biological sludge rather than chemical sludge. Considering the cost involved for disposal of sludge generated during treatment of tannery wastewater, the present study focused on the elimination of chemical sludge generation. Plain sedimentation followed by anaerobic treatment of tannery wastewater for biogas generation and the combined effect of chromium and COD/SO_4 ratio on biogas generation was monitored. During anaerobic treatment of composite tannery wastewater without plain sedimentation, biogas generation of 204 mL/g of COD $_{removed}$ was observed, whereas with half an hour and 2 h settlement, the biogas generation observed to be 235 and 255 mL/g of COD $_{removed}$ respectively. Suspended solids, especially those inert in nature, have a synergistic effect on biogas generation. When the Cr concentration increased from 32 to 90 mg/L, about 8% reduction in biogas generation was observed. The amount of primary sludge generated during treatment of 1 m^3 of tannery wastewater with plain sedimentation for a period 2 h was only 0.5 kg/m^3, whereas with addition of chemical coagulants and coagulant aids, it was about 3.03 kg/m^3. Hence, for the tannery wastewater generated from wet-blue to finish tanning operations, plain sedimentation followed by anaerobic treatment is an environmental friendly option for elimination of chemical sludge generation.

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