

Effect of COD/Sulphate Ratio on Anaerobic Digestion of Sulfate Rich Post Tanning Wastewater at Different F/M Ratios

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ABSTRACT

Anaerobic digestion of post tanning wastewater was performed in batch anaerobic digesters for the influence of various parameters such as COD/Sulphate ratio (0.62, 0.67 and 1.19 (w/w) %) and F/M ratio (0.3, 0.5, 0.7, 0.9, 1.1, 1.3 and 1.5 (w/w) %). Among the studied conditions, F/M ratio of 0.3 was found to be optimum for the removal of COD and Sulfate from the post tanning wastewater. The maximum removal of COD was found to be 53.1, 64.8 & 80.1% and reduction of sulphate was observed to be 32.7, 46.9 & 58.1% at COD/sulphate ratios of 0.62, 0.67 and 1.19 respectively. The reduction efficiency of COD and Sulphate were decreased with increase in COD/sulfate ratios from 0.62 to 1.19 in anaerobic digester, a similar trend was observed for F/M ratio from 0.3 to 1.5% in anaerobic digestion. The production of methane gas at COD/sulphate ratio of 0.62, 0.67 and 1.19 were observed to be 189, 246 and 310 ml/g of COD removed. The maximum concentration of sulfide production was achieved for the sample which reached sulphate concentration of 426 mg/L at COD/sulfate ratio of 0.62, and the anaerobic digestion process was inhibited at this sulfide concentration. These results show that the post tanning wastewater with 0.62 COD/sulfate ratios shows process inhibition in AD.

Keywords: COD/sulfate ratio; F/M ratio; Post tanning wastewater; Sulfate containing wastewater.

I INTRODUCTION

In recent years, anaerobic processes have become popular for the treatment of many organic rich industrial wastewaters. However, there are some industries pulp and paper processing, molasses fermentation, sea food processing, potato starch factories, tannery, edible oil refineries, pharmaceutical production, petrochemical processing, and wine distilleries are not easily amenable by anaerobic treatment due to the presence of a high concentration of sulfate and/or sulfide (Lens et al., 1998;). The application of anaerobic treatment to these wastewaters has been found to be problematic due to the formation of hydrogen sulphide (H₂S). The production of H₂S in anaerobic digesters results from the action of sulphate-reducing bacteria (SRB) which utilize sulphate as terminal electron acceptor and compete with acetogens and methanogens with several key substrates including propionate, butyrate, ethanol, acetate and H₂/CO₂. The reduction of sulphate during the anaerobic treatment of wastewaters are generally undesirable action which substantially reduces the methane yield and reason for the many operating issues such as corrosion, odour and microorganism toxicity caused by H₂S. The direct discharge of tannery wastewater creates environmental threat for its high concentration of dyes, surfactants, sulfonated oils, chromium salts, solid waste fragments, waste skin trimmings and many other inorganic compounds (Durai, G; 2011; Zupancic, G.D, 2010). Generally, post-tanning involves neutralisation and washing steps, followed by re-tanning, dyeing and fat liquoring of hides/skins. The use of sodium bisulphite, sodium metabisulphite and sodium thiosulphate are the

prime reason for the high concentration of sulphate ions in the post-tanning wastewater. Moreover, the post tanning waste water contains high concentration of organic matter, suspended solids, sulphide and chromium. (Haydar.S et al., 2009). The high concentrations of organic ammonical nitrogen, collagen, and nitrogen functional tanning agents made post tanning wastewater more recalcitrant in nature (El-Sheikh, M.A, 2011).

There are parameters such as quantity of inoculum added and the amount of waste (based on the Chemical Oxygen Demand (COD) content) were investigated to understand the appropriate food (substrate) to Microorganisms (inoculum) (F/M) ratio (Gonzalez-fernandez, C. et al 2009;). Particularly, the substrate concentration has been considered as an important factor which influences the efficiency of the anaerobic digestion process (Lianhua et al., 2010; Sanchez et al., 2001). At very low substrate concentrations, there is a risk of low metabolic activity and very low quantity of biogas production. In contrast, if the substrate concentration is high, that might lead to a substrate overload condition in which intermediate products may accumulate, resulting in product inhibition of the process (Tanimu et al., 2014; Zhang et al., 2014). Generally COD to sulphate ratio is a parameter widely used to control biological sulphate reduction, as well as the methanogenic process. The Sulphate reduction and methane formation may take place simultaneously in anaerobic digestion process. Both sulphate reducers (SRB) and methane formers (MPB) may use hydrogen and acetate as a sources for electron donor which leads to a competition for organic substrate exists between SRB and MPB (ref). The consumption of organic content is strongly

dependant on the COD/SO₄²⁻ ratio in organic substrate. (Isa et al., 1986). The ratio of COD/SO₄²⁻ about 0.67, was found to be theoretically enough for complete removal of organic matter as COD by sulphate reducing bacteria (Rinzema and Lettinga, 1988a). If the COD/sulfate ratio less than 0.67, then the amount of organic matter in the wastewater could be insufficient for complete reduction of the sulfate, and additional substrate should be added if the sulfate removal was the primary concern of the treatment.(RinzemaA.,1988). The active competition between MPB and SRB are found to be at COD/sulfate ratios of 1.7–2.7 (Choi and Rim 1991).

The present study focused on the treatment of sulphate rich post tanning wastewater by anaerobic digestion method. The main objective of this work was to study the influence of feeding strategy at different COD/sulfate and F/M ratio on sulfate and COD removal efficiencies.

II MATERIALS AND METHOD

(a) Sample collection (substrates and inoculums) and anaerobic digestion-Batch study

The post tanning wastewater was collected from the common effluent treatment plant (CETP) located in Chennai. The wastewater was collected in a plastic carboy and transported to Environmental Technology Division, Central Leather Research Institute and stored at 4°C until any experimental study. The dewatered sludge was collected from one of the anaerobic digestion plant and used as an inoculum for the experimental study. The batch experimental study of anaerobic digestion was performed for different COD/SULPHATE ratio (0.62, 0.69, and 1.2). The characteristics of initial wastewater at different COD/SULPHATE ratio were shown in table 1. The F/M ratio was also varied for 0.3, 0.5, 0.7, 0.9, 1.1, 1.3 and 1.5% respectively. The 120 mL serum bottles were taken for the batch experimental study with working volume of 80 mL. The serum bottles were closed by rubber cap and an aluminum seal after nitrogen gas purging at a rate of 15 mL/s (at ambient temperature; pressure, 0.8– 1.0kg/cm²) for 10 min for the anaerobic condition. The batch reactors were monitored at different retention time upto 108 h.

Table 1

Physico-chemical properties of post tanning wastewater

Parameters	EXP-1	EXP-2	EXP-3
pH	6.88	7.06	7.18
ORP (mV)	-302.96	324	-336.53
Chemical Oxygen Demand (COD) mg/L	3027.33	3097.33	3320
Sulphate (mg/L)	4853.33	4480	2753.66
COD/Sulphate ratio	0.62	0.69	1.20
Sulphide (mg/L)	43.33	38.66	26
VFA (mg/L)	1140	1190	1110

III PHYSICO-CHEMICAL PARAMETER ANALYSES

All the physico-chemical parameters were analysed according to the methodology described in standard methods of water analysis (APHA, 2005).The sulphate and sulphide parameters were immediately fixed after sample collection with 2N zinc acetate at alkaline condition to avoid sulphide oxidation. The fixed samples were centrifuged at 5000 rpm for 5min. The supernatant solution was taken for the analysis of sulphate and precipitate for total sulphides analysis by the iodometric method. The COD parameter was analyzed from the centrifuged sample (without zinc acetate fixation) after acidifying the sample with1N sulphuric acid to strip out sulphides as H₂S gas and fixed by addition of potassium dichromate. The closed reflux digestion was carried by HACHs COD digester (Loveland, USA) for the analysis of COD parameter. The ORP was recorded by Orion

920 (Thermo electronic instruments) and pH was measured using Hanna Instruments, (Model: HI 2212) pH meter. The generation of methane gas was measured by NaOH displacement method based on mariotte principle, i.e., the volume of 1 N NaOH solution displaced is equivalent to volume of methane gas generated. The generation of methane gas was recorded on daily basis.

$$M_{\max} = M_E S_L$$

(1)

Where M_{max} represents the methane yield (L g-COD⁻¹ removed),M_E is the total volume of methane gas (ml-CH₄) at the end of experiment and S_L is the amount of substrate removed (g-COD) at the end of experiment.

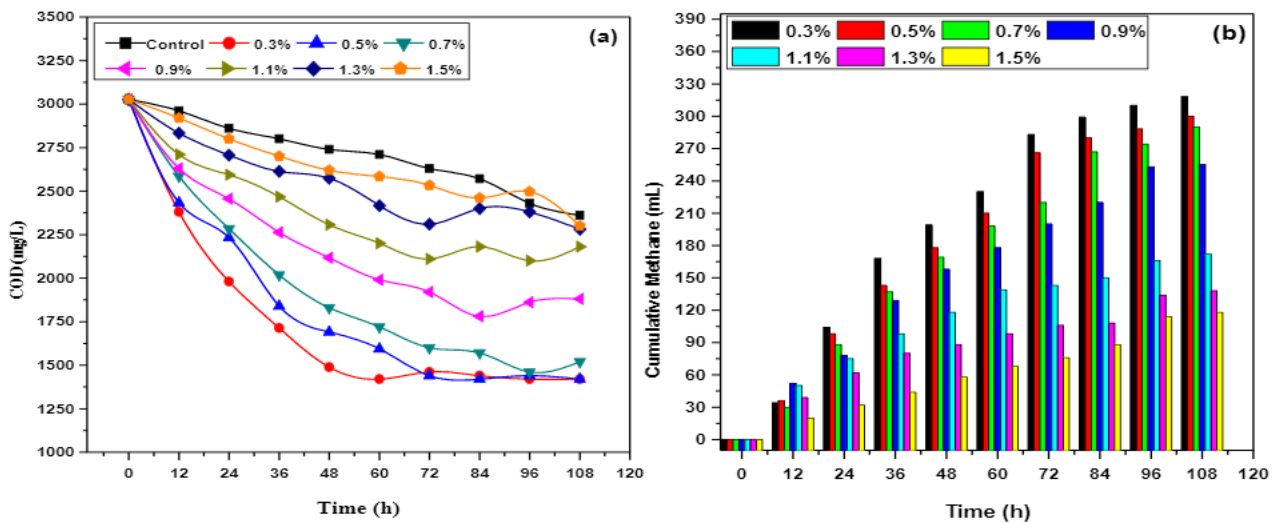
IV RESULTS AND DISCUSSION

In general, if there is a competition between MPB and SRB for a substrates available for anaerobic system, the treatability of wastewater will be highly significant by the presence of sulfate and it influenced much by COD/Sulphate ratio of the substrate (McCartney and Oleszkiewicz, 1991). The minimum COD/Sulphate ratio required for the

complete reduction as reported to be 0.67 (Lens et al., 1998). Hence, in this present investigation anaerobic batch reactors were operated for 108 hours at different COD/Sulphate ratios (0.62 0.69 and 1.2). The wastewater collected from the CETP has COD/Sulphate ratio of 1.2 minimum and maximum of 0.67. The effect of F/M ratio on COD and sulfate removal was also studied by varying F/M ratio from 0.3 % to 1.5 %.

V EFFECT OF COD REDUCTION AND METHANE FORMATION AT COD/SULPHATE RATIOS

The reduction of COD was evaluated for different F/M ratios (0.3, 0.5, 0.7, 0.9, 1.1, 1.3 and 1.5%) at



COD/Sulphate ratio of 0.62 and presented in Fig. 1a.

Fig. 1 Anaerobic digestion for post tanning wastewater at COD/Sulphate ratio 0.62 (a) COD removal (b) Cumulative methane formation in mL

The figure shows that the increase in F/M ratio from 0.3% to 1.5%, there was reduction in COD. The maximum amount of COD reduction was achieved for F/M ratio at 0.3 %. The maximum percentage of COD reduction was found to be 53% for 60h incubation period as shown in Fig 4. The other than 0.3 % of F/M ratios showed lower amount of COD removal; this may be explained by less availability of microorganism in the anaerobic reactors. Furthermore, the substrate utilization rate (SUR) was also increased with increase in addition

of inoculums. The cumulative yield of methane was recorded for each substrate as a function of time at different F/M ratios and reported in Fig.1b. The methane gas production for the blank samples always nil, hence the values of the blanks are not shown in Fig. 1b, 2b, 3b and 5. Irrespective of different F/M ratio, the maximum cumulative methane generation was observed to be 320 ml for the residence time of 60-84h and remains constant for the batch reactors which operated for maximum residence time of 108h.

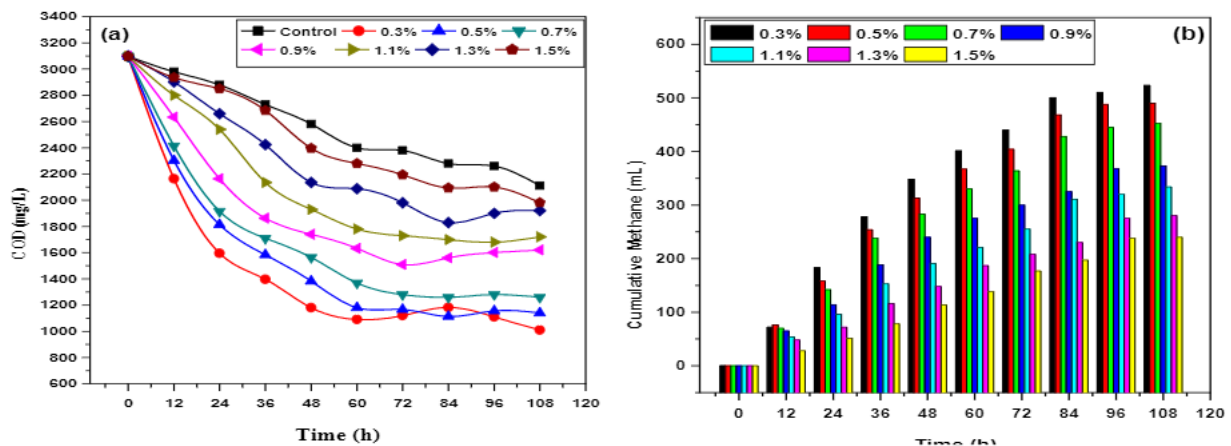


Fig. 2 Anaerobic digestion for post tanning wastewater at COD/Sulphate ratio 0.69 (a) COD removal (b) Cumulative methane formation in mL

The increase in F/M ratio may slow down the methanogen activity which resulting in decrease in generation of methane gas. The production of methane gas was found to be 0.204, 0.189, 0.185, 0.192, 0.185, 0.184 and 0.182 L of methane gas/ g of COD removed for the studied F/M ratios of .3, 0.5, 0.7, 0.9, 1.1, 1.3 and 1.5%. There are similar reports on the batch fermentation using wheat straw as a substrate at different I/S ratio Hashimoto (1989) and Chynoweth et al. (2001) reported that maximum conversion rate of substrate was obtained for the F/M ratio of 0.5.

Fig. 2a. Shows the reduction of COD for different F/M ratios from 0.3 to 1.5% at COD/ Sulphate ratio

0.69. Among the studied concentration of F/M ratio, ratio operated at 0.3% showed maximum removal of COD, 64.8 % at 60 to 72 h of incubation time. All the other ratios showed less COD removal shown in Fig 4. The Fig 2b & 5 Shows Cumulative methane gas generation at 0.3 % of F/M ratio was found to be 523 ml and 0.260 L of methane / g of COD removed. Irrespective of different F/M ratio, the maximum amount of methane gas generation was observed for the 72-84h of incubation time for all the F/M ratio values. The effect of COD reduction for different F/M ratio(0.3, 0.5, 0.7, 0.9, 1.1, 1.3 and 1.5%) were studied at COD/ Sulphate ratio of 1.2 as shown in Fig 3a.

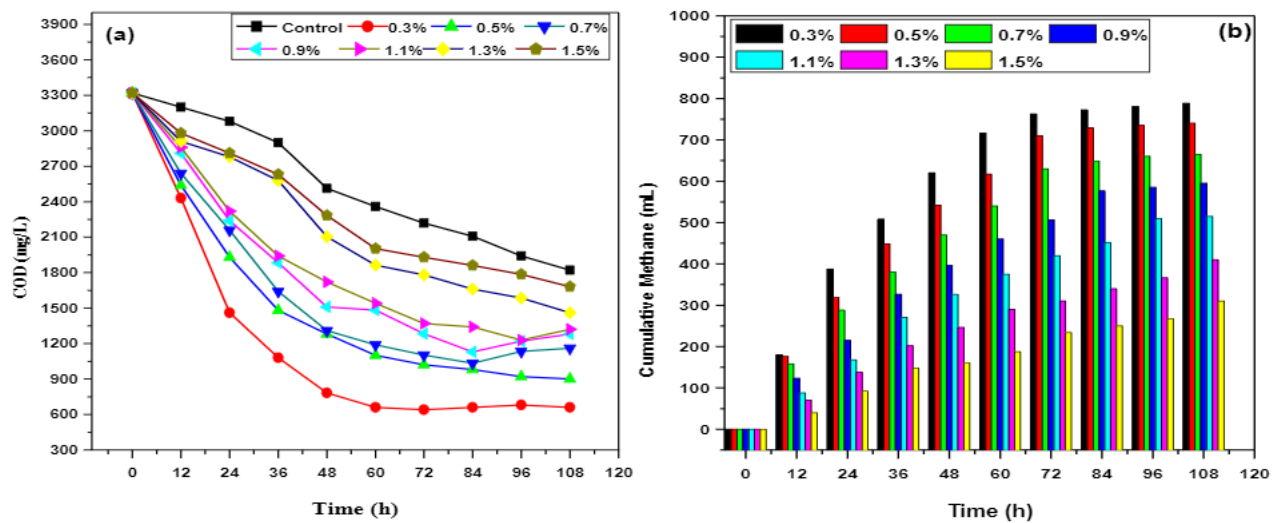


Fig. 3 Anaerobic digestion for post tanning wastewater at COD/Sulphate ratio 1.2 (a) COD removal (b) Cumulative methane formation in mL

The maximum COD reduction was observed for the F/M ratio operated at 0.3 %, the maximum removal of COD at 0.3 % of F/M ratio was found to be 80 % at incubation time of 48 to 60 h.as shown in Fig 4. The Cumulative yield of methane gas generation was observed to be 788 ml with 0.310 L of methane / g of COD removed for 0.3% F/M ratio (Fig. 3b) (Guerrero, L et al., 2013). Either the increase or decrease in value of F/M ratio of 0.3 % showed decrease in amount of methane gas production as shown in Fig 5. This

illustrate that the optimum F/M ratio for the treatment of post tanning wastewater is 0.3 %. Irrespective of F/M ratio, the maximum methane generation was observed for all the F/M ratio after the residence time of 60 to 72h. A similar response was observed by Jeong T.Y, 2008 for the wastewater operated at high COD/sulphate ratios with maximum methane production. Further, COD removal efficiency was increase with respect to increase in COD/sulphate ratios from 1.1 to 116(Izumi et al., 2010; Liu et al., 2009).

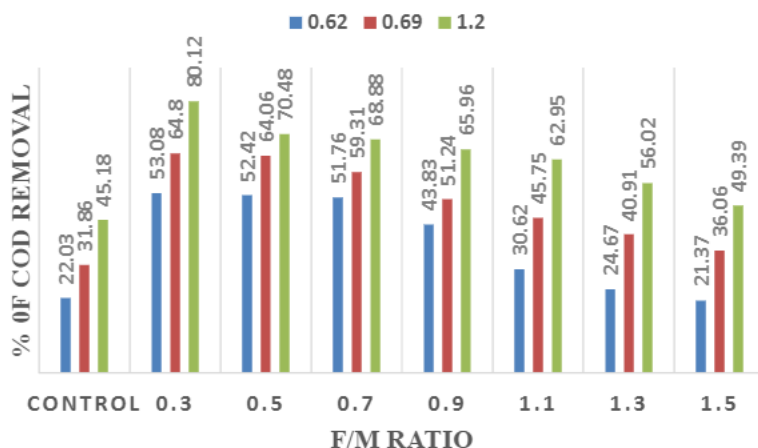


Fig. 4 Percentage of COD removal at different COD/Sulphate ratio and different F/M ratios for the treatment of nost tanning wastewaters

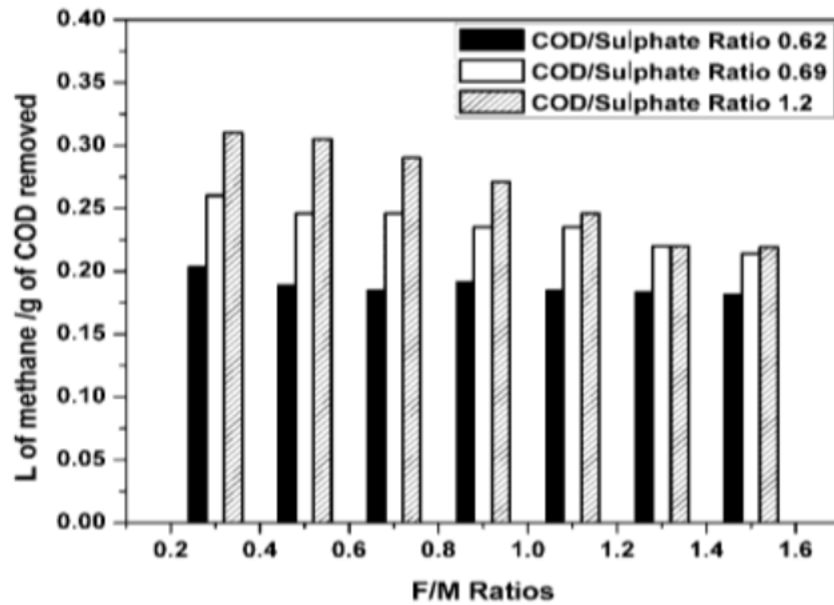


Fig. 5 Profile for the Methane gas generation in L /g of COD removed at different COD/Sulphate and F/M ratios for the treatment of post tanning wastewater

VI REDUCTION OF SULPHATE AND FORMATION OF SULPHIDE IN ANAEROBIC DIGESTION AT DIFFERENT COD/SULPHATE RATIOS

the system is operated at high COD/Sulphate ratios, MPB grows with increase in solution pH due to the formation of ionized products such as HS⁻ and S²⁻. (Biswas, T. 2012). In Anaerobic digestion process the sulphate ions was reduced to sulphide ions by Sulphate reducing bacteria.

At low COD/Sulphate ratios, due to the availability of high sulphate concentration SRB may grow actively and generates H₂S which are found to be many reason for the decrease in solution pH. When

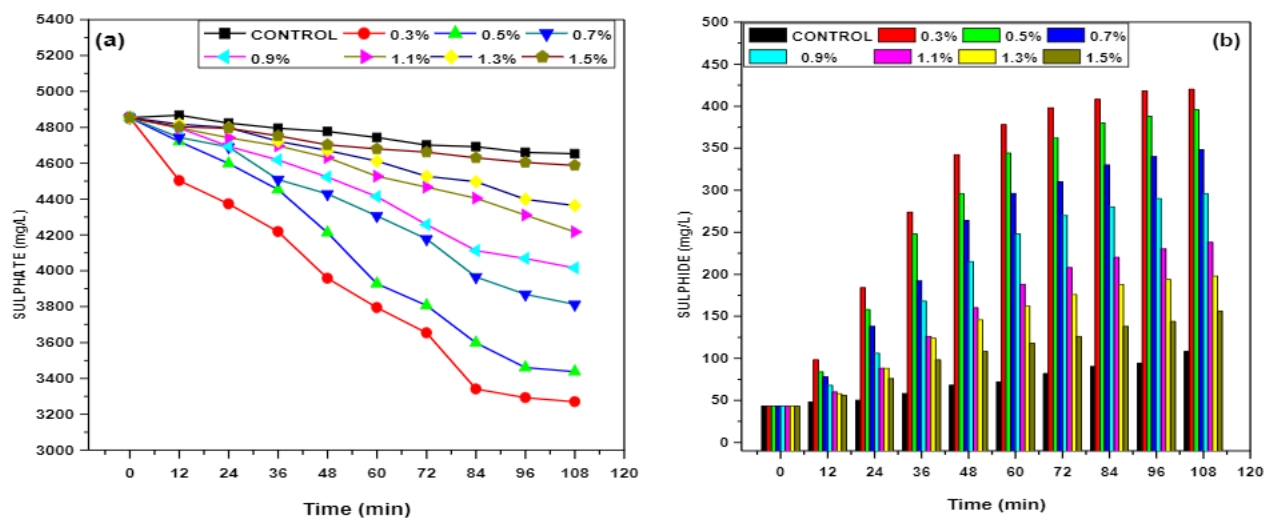


Fig. 6 Reduction of sulphate and formation of sulphide concentration in anaerobic treatment of post tanning wastewater at COD/Sulphate ratio 0.62 (a) Sulphate reduction (b) Sulphide formation

Fig 6a shows change in sulphate concentration for the anaerobic digestion operated at COD/Sulphate ratio of 0.62. The results revealed that the maximum sulphate reduction was observed for the system operated at 0.3% F/M ratio. The reduction

of sulphate concentration was decreased from 4853mg/L to 3270 mg/L with 32% of sulphate reduction (Fig 9). Further, Fig 6b shows the formation of sulphide concentration during anaerobic digestion. The concentration of sulphide

was found to be 426, 402, 358, 302, 248, 198 and 156 mg/l for the system operated at different F/M ratio (0.3, 0.5, 0.7, 0.9, 1.1, 1.3 and 1.5%). The anaerobic digestion process was found to be inhibited with increase in sulphide concentration for the studied experiments operated at different F/M ratios.

Anaerobic digestion experiment was carried out at COD/Sulphate ratio of 0.69 and presented in Fig.7. The change in concentration of sulphate is shown in Fig.7a and the formation of sulphide concentration are presented in Fig.9.

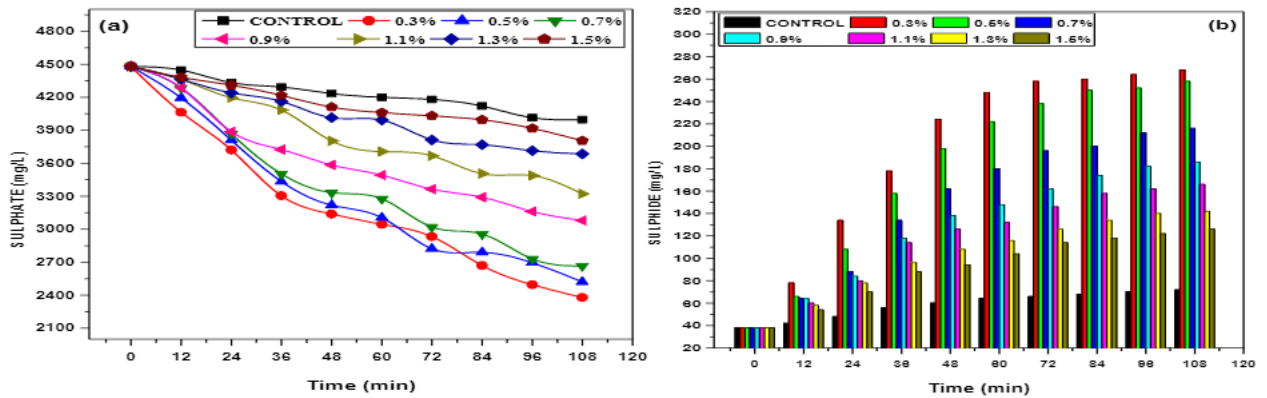


Fig. 7 Reduction of sulphate and formation of sulphide concentration in anaerobic treatment of post tanning wastewater at COD/Sulphate ratio 0.69 (a) Sulphate reduction (b) Sulphide formation

The figures illustrated that the concentration of sulfate was observed to be decreased from 4480 mg/L to 2380mg/L at an F/M ratio of 0.3. The maximum percentage of sulphate reduction was found to be 46.83 % for the AD system operated at 0.3% F/M ratio. Further, the increase in F/M ratio from 0.3 to 1.5% decreased the sulphate removal percentage. The calculated percentage of sulphate removal are 43.7, 40.5, 31.2, 25.9, 17.8, and 15%, for the F/M ratio of 0.5, 0.7, 0.9, 1.1, 1.3 and 1.5% respectively. The formation of sulphide concentration at F/M ratios from 0.3 to 1.5% was observed to be 278, 268, 226, 196, 186, 162 and 146mg/L respectively (Fig. 7b). The maximum concentration of sulphide for the AD operated at 0.3% F/M ratio produced more than 200 mg/L in

48h of incubation time. Hence, it failed on anaerobic digestion due to sulphide toxicity to the microorganisms. However, the system operated at 0.5% F/M ratio produced sulphide concentration above 200 at 60h incubation time without sulphide inhibition. This may be due to the high microorganism growth is balanced the sulphide toxicity.

The influence of COD/sulphate ratio operated at 1.2 for different F/M ratio for the reduction of sulphate is shown in Fig 8&9. The maximum decrease in concentration of Sulfate was found to from 2753 mg/L to 1328 mg/L for a studied F/M ratio of 0.3 and the percentage of sulphate was calculated to be 58.1 % (Fig.9).

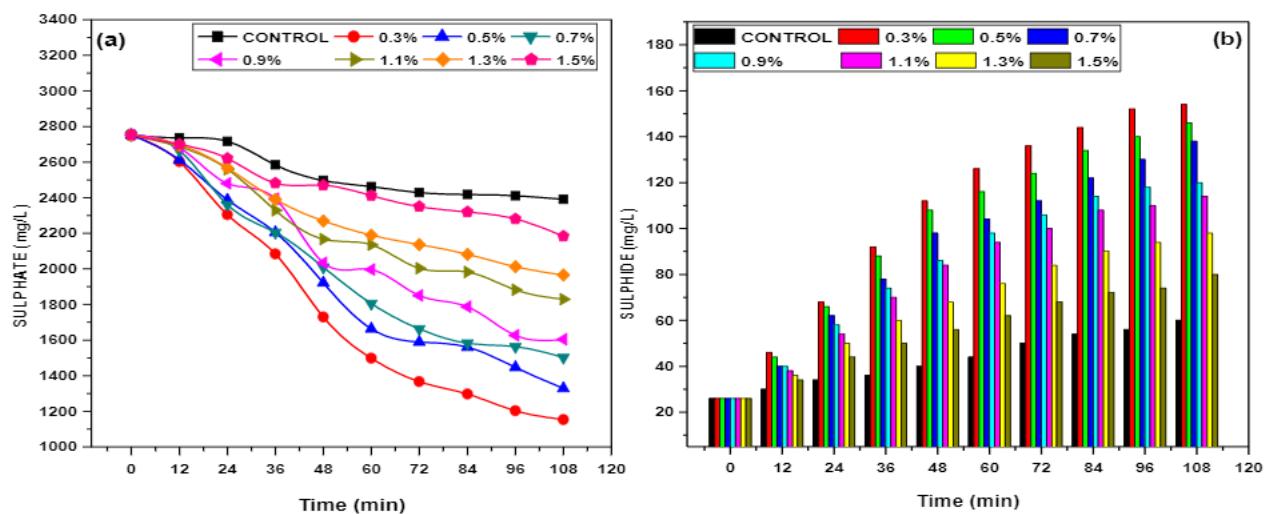


Fig. 8 Reduction of sulphate and formation of sulphide concentration in anaerobic treatment of post tanning wastewater at COD/Sulphate ratio 1.2 (a) Sulphate reduction (b) Sulphide formation

As the increase in F/M ratio from 0.3 to 1.5% decrease the percentage sulphate removal and the values are 51.8, 45.4, 41.7, 33.5, 28.6 and 20.1% respectively for the F/M ratio of 0.5, 0.7, 0.9, 1.1, 1.3 and 1.5%. The formation of sulphide concentration is presented in Fig 12b for the different F/M ratio. The concentration of sulphide was found to be 154, 146, 138, 120, 114, 98 and 80 mg/L for the F/M ratio of 0.5, 0.7, 0.9, 1.1, 1.3 and 1.5% respectively. The results illustrated that the

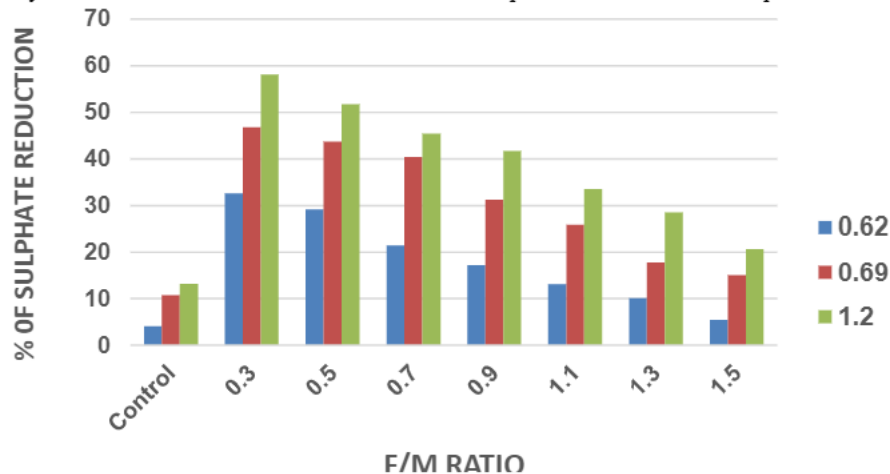


Fig. 9 Percentage of Sulphate removal at different COD/Sulphate and F/M ratios for the treatment of post tanning wastewater

VII CONCLUSION

The post tanning wastewater from CETP'S shows different COD/Sulphate ratios from 0.62 to 1.2%, for all COD/Sulphate ratios the AD shows 0.3% F/M ratio was the optimum to remove COD and Sulphate. Anaerobic digestion of COD/Sulphate ratios for 0.62 shows H₂S production in all F/M ratios, due to H₂S concentration the anaerobic digestion process were inhibited and the inhibition of AD was observed. Anaerobic digestion of COD/Sulphate ratios for 0.69 and 1.2 shows not much process inhibition in AD. So, anaerobic digestion of COD/Sulphate ratios for 0.62 need an alternative pre-treatment method to proceed AD. Otherwise post tanning wastewater with below 0.62 COD/Sulphate ratios were treated separately.

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increase in COD/sulphate ratio from 0.62 to 1.2 increased the removal of sulphate for the F/M ratio operated at 0.3 %. However, the increase in F/M ratio decreased the removal of sulphate ion from the post tanning wastewater (Krapivina M et al., 2007). A similar reports on the reduction of sulphate with increase in COD/sulphate ratio by Archilha (2010) reported that the production of sulphide was found to be high for the system operated in low COD/sulphate ratio.

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