Physical and Chemical Separation of Oil and Suspended Solids from Palm Oil Mill Effluent

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Abstract: The measured characteristics of palm oil mill effluent (POME) from 5 palm oil mills before discharge into the first settlement pond were temperature 72°C, pH 4.2, TS, SS, COD and O&G 71.6, 47.3, 120.1 and 28.4 g/l, respectively. The separation of oil and suspended solids (SS) by settling at 30- 55°C for 24 hrs showed that the POME was separated into the top layer and bottom layer but no oil layer was observed. The highest percentage of top layer (56 %) was obtained after settling the POME at 55°C with the removal of O&G, SS and COD 77.1 %, 94.4 % and 85 %. The separation of O&G and SS

from POME by centrifugation showed that the suitable speed and time for the separation was 7,000 rpm (6,240 x*g*) for 15 min. The removal of TS, SS, O&G, and COD from the POME by centrifugation was 91.6, 95.4, 97.1 and 94.2 %, respectively. While the separation of O&G and SS from the POME by chemical coagulants ($Ca(OH_2)$, $FeSO_4$ and alum) showed that $FeSO_4$ 1.0 g/l gave the highest removal of O&G (82.5 %), SS (88.5 %) and COD (61.1 %) after 10 hrs settling.

Keywords: Palm oil mill effluent, POME, oil separation, oil and grease, suspended solid.

Introduction

Palm oil is a rapidly growing industry in Southern Thailand with over 50 palm oil mills in operation. The palm oil mill generated effluents have BOD 50,000- 60,000 mg/l, COD 80,500-115,000 mg/l, SS 18,500-52,000 mg/l, O&G 16-2,500 mg/l and pH 4.05-4.02 [1]. Palm oil mill wastewater treatment often uses simple biological processes including open ponds for anaerobic and aerobic treatments. It usually takes more than 200 days to reduce the BOD of the effluent to 100 mg/l [1]. For is reason, physical and chemical methods were examined for pretreatment of POME before biological treatment.

It has been reported that centrifugation of the effluent after the separator and the mixed effluent could be separated into 3 layers. The oil recovery by this method was 5-30 %. Furthermore,

COD and O&G of the final effluent were reduced approximately by 50 % and 85 % [2]. The oil from palm oil mill effluent was separated by centrifugation at 10,000 g at 5^{o}C which showed 50 % reduction in O&G [3]. It has been reported that the use of alum as a coagulant had removal efficiencies of 96 % O&G [4].

Therefore, this paper describes the pretreatment of POME by physical and chemical methods before biological treatment in the open ponds system. The physical methods used to separate oil and suspended solids from POME in this experiment were flotation and centrifugation. Some chemicals were also used to coagulate the suspended solids.

Materials and Methods

1. Palm oil mill effluent (POME)

POME samples were collected from inlet POME (combined wastewater) to the first settlement pond of the local palm oil mills. All collected samples were mixed well, divided into 2l portions and kept at 4°C until required. Sufficient quantities would be withdrawn from the cold storage and allowed to reach to room temperature (28-30°C) before use.

2. Determination of characteristics of POME

Determination of BOD, COD, TS, SS, O&G and pH were carried out in accordance with the Standard Method for the Examination of Water and Wastewater [5]. Three replicates were analysed for one sample.

3. Separation of oil from POME by physical methods

3.1 Separation of oil from POME by flotation

Effect of time and temperature on POME separation was studied by allowing the POME sample (100 ml) to settle in 100 ml cylinder at different temperatures (30^{o}C, 37^{o}C, 45^{o}C and 55^{o}C). The % removal of O&G, TS, SS and COD from the middle layer were then determined.

3.2 Separation of oil from POME by centrifugation

The POME sample (50 ml) was centrifuged at 5,000 rpm (3,184x*g*), 7,000 rpm (6,240x*g*) and 10,000 rpm (12,735x*g*) at 30°C for 20 minutes. The quantity of oil in the top layer, middle layer and bottom layer was analyzed as well as the % removal of O&G, TS, SS and COD from the middle layer. After obtaining the suitable speed the centrifugation time was varied from 5-20 minutes.

4. Separation of oil from POME by chemical methods

Lime ($Ca(OH)_2$), $FeSO_4$, and alum (commercial grade) were used to coagulate POME. The POME sample (100 ml) was adjusted to pH 6.0 by addition of 6.0 N NaOH. Then the chemical coagulants were added at different doses (0.5, 1.0, 1.5 and 2.0 g/l). Chemical coagulation process consisted of 1 minute of rapid mixing, 30 minutes of slow mixing and settling for 10 hrs at room temperature (30^{o}C). The changes of color, pH and volume of top layer were observed. Top layer was collected to analyze for % removal of TS, SS, O&G and COD.

Results and Discussion

1. Characteristics of palm oil mill effluent (POME)

The samples of inlet POME to the first settlement pond of 5 palm oil mills in Southern Thailand: Pure Palm Oil Co., Ltd. in Songkhla, Siam Modern Palm Oil Co., Ltd. in Krabi, Satoon Palm Oil Industry Co., Ltd. in Satun, Trang Palm Oil Co., Ltd. in Trang and Univanich Palm Oil Co., Ltd. in Krabi, were collected and analyzed. The characteristics of these POME samples are shown in Table 1.

Table 1. Characteristics of inlet POME to the first pond from 5 palm oil mills.

Factory	pH	Temp (^{o}C)	TS (g/l)	SS (g/l)	COD (g/l)	O&G (g/l)
Pure Palm Oil Co., Ltd.	4.2	60.0	82.1	65.5	160.7	25.1
Siam Modern Palm Oil Co., Ltd.	4.4	75.0	64.6	32.7	30.7	12.9
Satoon Palm Oil Industry Co., Ltd.	3.9	-	75.9	57.42	99.8	17.2
Trang Palm Oil Co., Ltd.	4.2	82.0	72.2	36.3	46.5	8.7
Univanich Palm Oil Co., Ltd.*	4.2	-	62.9	44.6	262.7	78.2
Mean	**4.2**	**72.3**	**71.6**	**47.3**	**120.1**	**28.4**
Std. Deviation	**0.2**	**11.2**	**8.0**	**14.0**	**94**	**28.5**

- not determined

The oil and grease (O&G) of inlet POME to the first pond from these palm oil mills were in the range of 8.70-25.05 g/l and the COD 30.7-262.7 g/l. A PORIM/RRIM survey of 40 palm oil mills in Malaysia indicated that the range of oil and grease from palm oil mill effluent was 1.30-86.43 g/l [6]. It has been reported that POME of 4 palm oil mills in Thailand contained O&G and SS 7.6 – 14.7 and 5.2 – 263 g/l, respectively [2]. On

average, wastewater from a palm oil mill contained COD and BOD of 70,647 mg/l and 35,160mg/l, SS and O&G of 17.5 and 11.1 mg/l, respectively [7]. For other authors who studied on palm oil mill effluent treatment using a pond system, the quantity of oil and grease from the system was 6,110 mg/l [8]. The main cause of differences in oil content in the wastewaters comes from the production process, preliminary wastewater treatment of each factory and time for taking the sample. Table 2 shows the characteristics of the POME of the same company at different sampling times to be used in different experiments. The results show some variation of each characteristic.

Table 2. Characteristics of inlet POME to the first pond used in flotation, centrifugation and coagulation experiments.

Parameters	Inlet POME for the different experiments		
	Flotation	**Centrifugation**	**Coagulation**
pH	3.81±0.02	4.15±0.01	4.18±0.01
Temperature (^{o}C)	53.00±2.65	50.00±1.30	53.33±1.53
TS (g/l)	58.93±0.29	47.81±0.22	52.9±0.03
SS (g/l)	39.48±0.65	26.19±0.41	44.13±0.46
O&G (g/l)	21.77±0.75	20.20±0.65	20.10±0.17
COD (g/l)	60.61±7.80	55.55±6.10	49.90±6.90

2. Separation of oil from POME by flotation

The characteristic of POME used in the flotation experiments contained O&G and SS 21.77 and 39.48 g/l, (as shown in Table 2). After one hour settling at 30-55°C all POME

samples were not separated. After 2 hrs most of the POME samples were separated in to 2 layers (top layer and bottom layer) except the POME settling at 30^{o}C. The percentage of top layer increased when settling time increased. The high temperature gave a higher percentage of top layer than low temperature (Figure 1). The POME that settled at 55°C gave the best percentage of top layer (56 %) at 24 hrs. It has been reported that POME from the sterilizer easily separated by normal settling but POME from the separator and mixed effluent were not successful through normal settling [2]. Meanwhile, [9] showed that the settling of POME from a decanter at 40°C had 38 % top layer at 19 hrs. The decrease of bottom layer was a result of dense coagulation of suspended solid. The analysis of the top layer showed that settling of the POME at 30^{o}C gave the lowest percentage removal of TS, SS, O&G and COD. While settling at 45 and 55^{o}C provided the best removal of TS, SS, O&G and COD when compared to the original POME (Figure 2). It was noticed that no oil layer was observed during settling. Probably, most of the oil in the wastewater was entrapped by plant cell debris in the bottom layer [3].

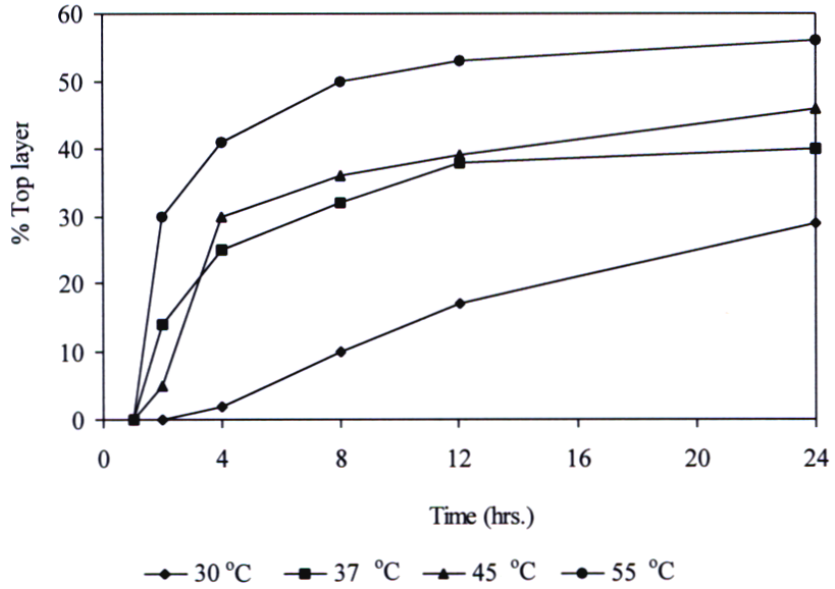

Figure 1. Effect of settling temperature and time on the % top layer of palm oil mill effluent.

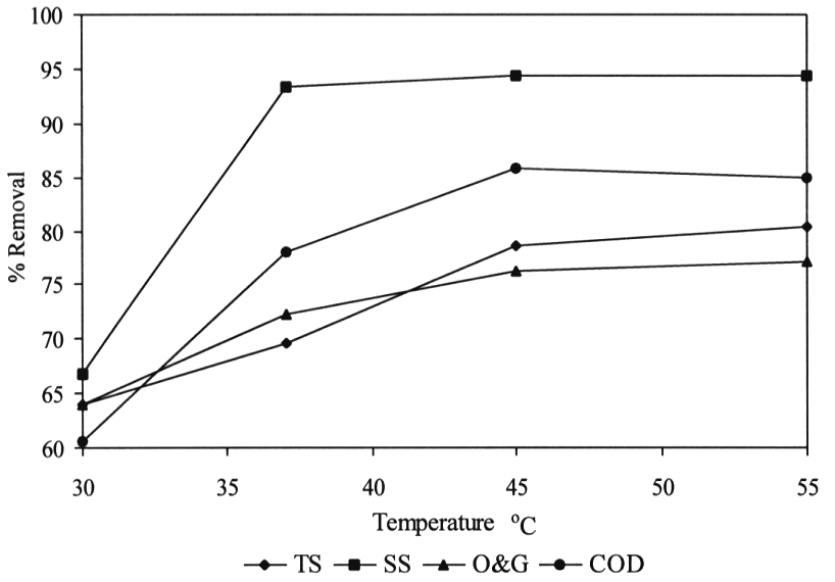

Figure 2. Effect of settling temperature on the % removal of TS, SS, O&G and COD from the top layer after 24 hours.

3. Separation of oil from POME by centrifugation

A centrifuge is a device for separating particles from a solution according to their size, shape, density and viscosity of the medium and the rotor speed [10]. The characteristics of the POME used in the centrifugation experiments are shown in Table 2. It contained O&G and SS 20.2 and 26.2 g/l, respectively.

After centrifugation of the POME at different speeds at 30^{o}C for 20 min the POME was separated into 3 layers: top (partial oil), middle (partial supernatant) and bottom (partial sediment) layers. The results showed that increasing the centrifugal speed did not significantly effect ($p < 0.05$) the volume of the top layer (Table 3). However, the volume of the middle layer was increased significantly and the volume of the bottom layer was decreased significantly. The top layer and the bottom layer each contained O&G 49-50 % of the total O&G in the POME, while the middle layer contained almost no O&G. More than 90 % removal of TS, SS, O&G and COD was obtained by centrifugation even at the lowest speed (3,184 x*g* or 5,000 rpm) (Figure 3).

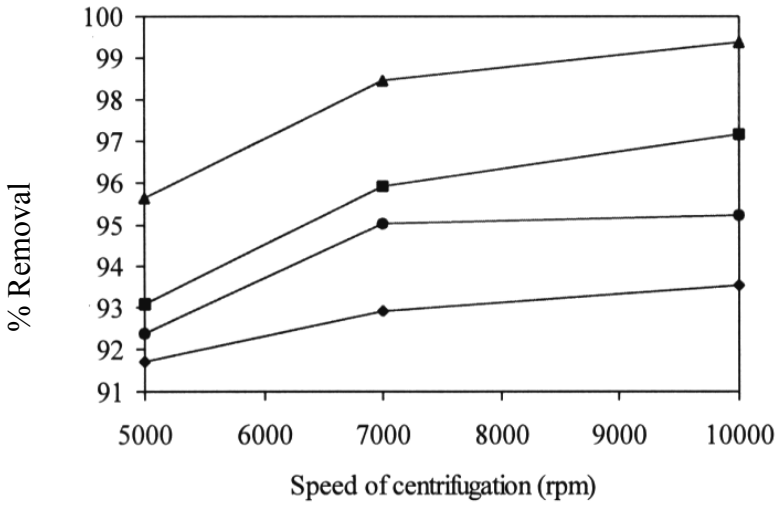

Figure 3. Effect of centrifugal speed on % removal of TS, SS, O&G and COD from the palm oil mill effluent (in middle layer).

Table 3. Effect of centrifugal speed on oil separation from palm oil mill effluent (at 30°C for 20 min).

Oil separation	**Speed**		
	5,000 rpm (3,184 *xg*)	**7,000 rpm (6,240 *xg*)**	**10,000 rpm (12,735 *xg*)**
Top layer (oil)			
Volume (%)	6.50[a]	6.80[a]	6.90[a]
Concentration of oil (g/ml)	0.152[b]	0.147[b]	0.146[b]
Quantity of oil* (g)	0.99[a]	1.00[a]	1.00[a]
Middle layer (supernatant)			
Volume (%)	52.20[a]	84.70[b]	86.80[c]
Concentration of oil (g/ml)	0.001[c]	0.0006[b]	0.0005[a]
Quantity of oil (g)	0.07[b]	0.06[b]	0.04[a]
Bottom layer (sediment)			
Volume (%)	11.30[c]	8.50[b]	6.30[a]
Concentration of oil (g/ml)	0.085[a]	0.113[b]	0.152[c]
Quantity of oil (g)	0.96[a]	0.96[a]	0.96[a]

Note: the same superscript-letter in the same row means no significant difference ($p<0.05$).

* Quantity of oil (g) = Volume (%) × Concentration of oil (g/ml)

Figure 3 showed that centrifugation at 7,000 rpm (6,240x*g*) and 10,000 rpm (12,735x*g*) did not have much significant difference on the percentage removal of TS, O&G and COD in the middle layer. However, the centrifugal speed had significant effect on the percentage removal of SS. The suspended solid and colloidal particles were effectively separated from the soluble constituents of the effluent after centrifugation. The centrifugation of POME at 10,000 rpm (12,735x*g*), 30^{o}C gave the reduction of TS, COD, O&G and SS 93.5, 95.2, 99.4 and 97.1% respectively. However, [3] reports that centrifugation of POME at 10,000x*g* 5^{o}C gave reduction in the TS, COD and O&G of the middle layer 46, 40 and 50 %. H-Kittikun [2] reported that centrifugation of the effluent after the separator and the mixed effluent recovered oil 5-30 % and COD and O&G of the final effluent were reduced approximately by 50 % and 85 %.

The effect of centrifugal time on the separation of O&G and suspended solids from the POME was performed at 7,000rpm (6,240x*g*). The results (Figure 4) show that the centrifugal time at 10-20 min did not have any effect on the volume of the top layer ($p < 0.05$). The volume of the middle layer increased with time, while the volume of the bottom layer decreased significantly (Table 4). The removal of oil from the supernatant was time dependent. The longer the centrifugation time, the more oil removal. The optimal time for oil separation from the POME by centrifugation was 15 min. If the top and bottom layer were removed, it could get rid of most of O&G and

COD of the POME (more than 95%). The results showed that no significant difference ($p < 0.05$) on the percentage removal of TS, SS, O&G and COD in the middle layer when the POME was centrifuged at 7,000 rpm (6,240 x*g*) for 15 and 20 min.

Table 4. Effect of centrifugal time on oil separation from the palm oil mill effluent at 7,000 rpm (6,240 xg) (at 30°C, 20 min).

Oil separation	Time of centrifugation (min)			
	5	**10**	**15**	**20**
Top layer (oil)				
Volume (%)	5.20[a]	6.30[b]	6.50[b]	6.90[b]
Concentration of oil (g/ml)	0.18[c]	0.15[b]	0.15[b]	0.14[a]
Quantity of oil* (g)	0.94[a]	0.945[a]	0.98[b]	1.02[b]
Middle layer (supernatant)				
Volume (%)	78.80[a]	82.90[b]	85.60[c]	86.80[d]
Concentration of oil (g/ml)	0.0016[d]	0.0013[c]	0.0007[b]	0.005[a]
Quantity of oil (g)	0.13[d]	0.11[c]	0.06[b]	0.04[a]
Bottom layer (sediment)				
Volume (%)	16.00[d]	10.80[c]	79.90[b]	6.30[a]
Concentration of oil (g/ml)	0.06[a]	0.09[b]	0.12[c]	0.15[d]
Quantity of oil (g)	0.94[a]	0.95[a]	0.96[b]	0.96[b]

Note: the same superscript-letter in the same row means no significant difference ($p<0.05$).

* Quantity of oil (g) = Volume (%) × Concentration of oil (g/ml)

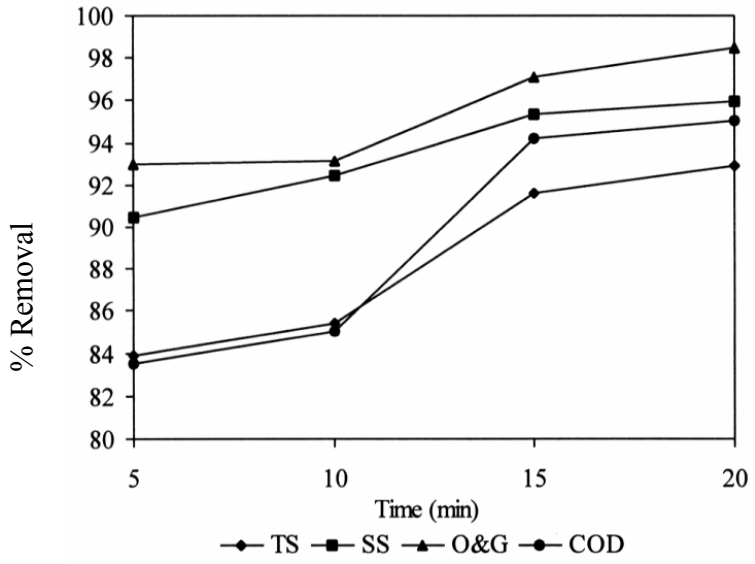

Figure 4. Effect of centrifugal time on % removal of TS, SS, O&G and COD in middle of POME at 7,000 rpm (6,240 xg).

4. Separation of oil from POME by chemical method

Chemical coagulants were added to enhance the removal efficiency of sedimentation. $Ca(OH)_2$, $FeSO_4$ and alum (commercial grade) at different dosages were used to separate O&G and SS from the POME. The POME used in this experiment had TS, SS and O&G 44.13, 52.91 and 20.10 g/l (Table 2).

The results are shown in Table 5. After adding the coagulants and settling for 10 hrs the POME was separated into 2 layers: top layer (partial supernatant) and bottom layer (partial sediment). No oil layer was observed on the top layer. When $Ca(OH)_2$ was used as the coagulant the result showed that the pH and the color intensity of the POME increased with the

increasing concentration. On the other hand, the pH and the color intensity of the POME decreased with the increasing addition of $FeSO_4$ and alum. The optimum dosage of $Ca(OH)_2$ and $FeSO_4$ addition was 1.0 g/l which gave 78.5 % and 82.5 % removal of O&G, respectively, while the pH of POME changed from 6.0 to 7.46 and 5.44. Alum at the concentration of 0.5 g/l gave % removal of O&G the same as $FeSO_4$ (1.0 g/l), but alum gave the color intensity of POME more than $FeSO_4$ and the pH of POME was 5.27. The control with no addition of chemicals gave 50.6 % removal of O&G. Addition of 1.0 g/l of $FeSO_4$ to the POME gave the highest percentage removal of TS, SS, O&G and COD in the top layer as 70.84 %, 88.52 %, 82.46 % and 61.13 %, respectively (Figure 5). The result showed that $FeSO_4$ was the most suitable for treatment of POME.

Table 5. The effect of types and dosages of chemical coagulants on the oil removal from POME after 10 hour settling.

Dose (g/l)	%Top layer	Color of POME[a]	pH	%Removal of O&G
$Ca(OH)_2$				
0.5	55	+	6.48	76.64
1.0	60	++	7.461	78.46
1.5	25	+++	8.17	75.39
2.0	10	++++	9.95	73.45
$FeSO_4$				
0.5	60	+++	5.53	80.41
1.0	65	++	5.44	82.46
1.5	50	++	5.25	77.28
2.0	55	+	5.10	78.60
Alum				
0.5	65	+++	5.27	81.22
1.0	50	++	5.21	76.45
1.5	55	+	5.16	78.00
2.0	60	+	5.02	79.03
Control[b]	60	+++	5.65	50.56

[a] more + means darker color
[b] chemical addition

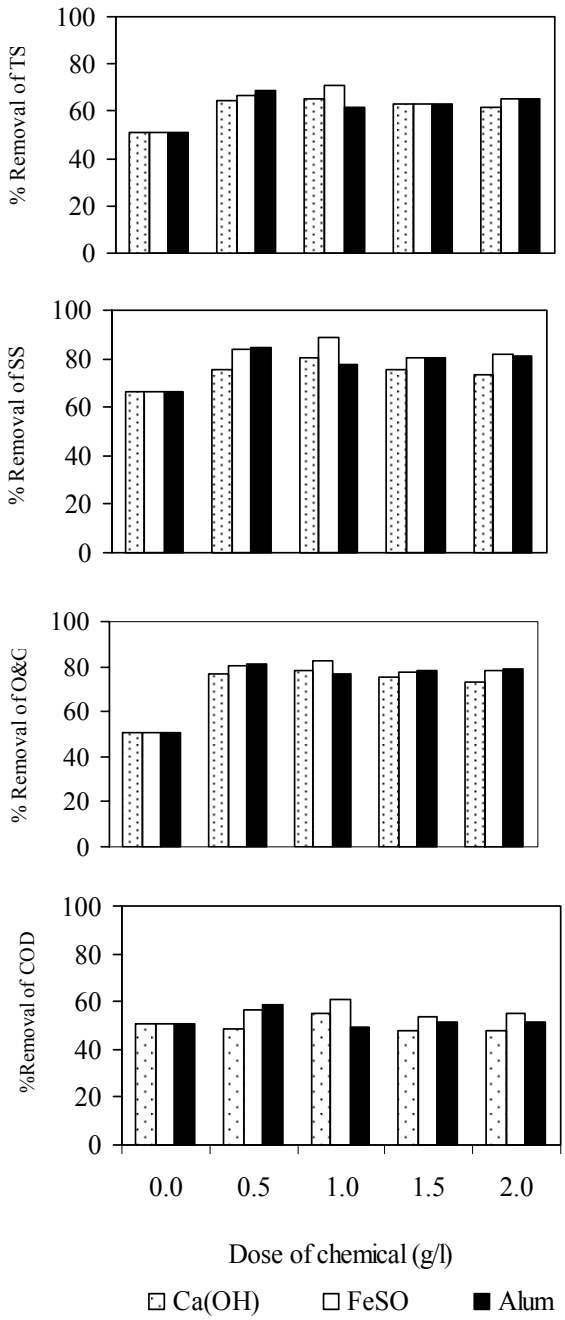

Figure 5. Effect of types and concentrations of chemical coagulant addition on % removal of TS, SS, O&G and COD from top layer of palm oil mill effluent after settling 10 hours.

Conclusions

The easiest way to separate O&G and suspended solids from palm oil mill effluent is settling but it is time consuming. The use of $FeSO_4$ as a coagulant also could remove O&G and some SS. However, the best way to separate O&G and SS from the POME is by centrifugation.

Acknowledgements

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