

## CHEMICAL COAGULATION OF SETTLEABLE SOLID-FREE PALM OIL MILL EFFLUENT (POME) FOR ORGANIC LOAD REDUCTION

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**RINGKASAN:** Kesesuaian proses pra-rawatan melalui pengenapan dan penggumpalan kimia telah dikaji atas 2 tujuan iaitu; pengembalian pepejal POME dan penurunan bebanan organik ke atas proses-proses rawatan berikutnya. Proses pengenapan graviti dan seterusnya pengoptimuman, penggumpalan dan pengelompokan kimia telah dikaji dengan menyeluruh. Bagi tujuan ini, gabungan penggunaan alum, polialuminium klorida (PAC),  $\text{FeCl}_3$  dan  $\text{FeSO}_4$  serta polielektrolit anion dikaji menggunakan modifikasi kaedah pengujian jar di mana, keberkesanan didasarkan kepada tahap penyingkiran  $\text{BOD}_5$ , COD dan SS. Keputusan menunjukkan bahawa keadaan optimum umumnya dicapai pada dos 150-200 ppm bagi  $\text{FeSO}_4$  dan  $\text{FeCl}_3$  sementara bagi alum dan PAC, ia dicapai pada kira-kira 300-350 ppm. Dos bahan penggumpal optimum didapati bergantung kepada kepekatan air sisa tersebut. pH proses penggumpalan didapati tidak banyak mempengaruhi tahap penyingkiran di antara pH 3-7. Ini bermakna, pengubahsuaian pH sebelum proses penggumpalan kimia boleh diabaikan tanpa banyak mempengaruhi keberkesanan penyingkiran.

**ABSTRACT:** The viability of pre-treatment process through sedimentation and coagulation was studied for two purposes i.e. valuable POME solids recovery and reduction of organic loading on the down stream treatment processes. The idea of gravity sedimentation was utilized to separate settleable solids from POME and subsequently, optimization of the combined coagulation and flocculation was closely studied. Here, combined use of Alum, polyaluminium chloride (PAC),  $\text{FeCl}_3$  or  $\text{FeSO}_4$  and anionic polymer were studied using modified jar test method where their efficiencies were gauged on the basis of the  $\text{BOD}_5$ , COD and SS removal. Results showed that optimization of coagulation and flocculation processes on settleable solid-free POME was generally reached at between 150-200 ppm of  $\text{FeSO}_4$  and  $\text{FeCl}_3$ , and 300-350 ppm of alum and PAC. The optimum coagulant dosages were found to be dependent on the strength of the wastewater. The coagulation pH was found to be having minimal effect on the process between pH 3-7. That means, pH adjustment prior to coagulation and flocculation might be omitted without detrimental effect on the process.

**KEYWORDS:** POME, coagulation, settleable solids, colloidal particles, jar test, removal efficiency, coagulant aid, solids recovery.

## INTRODUCTION

Coagulation and flocculation are the processes where compounds such as metal salts are added to effluents in order to destabilize colloidal materials. As a result, aggregation of small particles into larger, more easily removed floc takes place (Stephenson *et al.*, 1996). The process of coagulation is largely divided into surface charge neutralization of particles and floc formation (flocculation) by bridging the particles (Lefebvre and Legube, 1993). Unstabilized particles by charge neutralization are called primary floc (or coagulation floc) and floc enlarged by bridging are sometimes termed as secondary flocs (Stephenson *et al.*, 1996).

The effectiveness of the process is influenced by the coagulating agent, the coagulant dosage, the solution pH and ionic strength as well as the concentration and the nature of the organic compounds (Randtke, 1988).

Colloids are presented by particles over a size range of 1 nm ( $10^{-7}$  cm) to 0.1 nm ( $10^{-8}$  cm). These particles do not settle out on standing and cannot be removed by conventional physical treatment processes (Hammer and Hammer, 1997). Colloids present in wastewater can either be hydrophobic or hydrophilic. The hydrophobic colloids (clay etc.) pose no affinity for the liquid medium and lack stability in the presence of electrolytes. They are readily susceptible to coagulation. Hydrophilic colloids, such as proteins, exhibit a marked affinity for water. The absorbed water retards flocculation and frequently requires special treatment to achieve effective coagulation (Eckenfelder, 1989).

Colloids possess electrical properties that create a repelling force and prevent agglomeration and settling. Stabilizing ions are strongly adsorbed to an inner fixed layer that provides a particle charge that varies with the valence and number of adsorbed ions. Ions of opposite charge forms a diffuse outer layer which is held near the surface by electrostatic forces (Eckenfelder, 1989).

The effluent from the palm oil mills is highly polluting with a high BOD load, much of which is associated with finely divided colloidal or dissolved organic matter. It is also acidic and has a high oil content. However, it is non-toxic and biodegradable. A typical characteristic of POME is as shown in Table 1.

The finely divided nature of the suspended solids militates against efficient solids separation and the large proportion of colloidal and dissolved solids present minimizes the effectiveness of solids separation as a means of reducing BOD using conventional sedimentation processes. However, in terms of meeting the BOD:COD and suspended solids discharge standards, it is essential that a high proportion of these solids are removed before attempts are made to remove soluble BOD.

**Table 1.** Typical characteristics of POME (Ma, 1995)

Parameter (mg/L)	Range	Mean
BOD (3 Days @ 30 °C)	10250 - 47500	2500
COD	15500 - 106360	53630
TS	11450 - 164950	43635
SS	410 - 60360	19020
O&G	130 - 86430	8370
NH <sub>3</sub> -N	0 - 110	35
T-N	180 - 1820	770
pH	3.8 - 4.5	4.1

By virtue of their particle size, the suspended solids in POME are unlikely to settle readily unaided. Aid therefore, must be provided in the form of chemical coagulation and flocculation. This is accomplished by a combination of physical and chemical processes which thoroughly mix the chemicals with the wastewater and promote the aggregation of wastewater solids into particles large enough to be separated by sedimentation, floatation, media filtration or straining. The strength of the aggregated particles determines their limiting size and their resistance to shear in subsequent processes.

For particles in the colloidal and fine-supra colloidal size ranges (less than 1-2 microns), natural stabilizing forces (electrostatic repulsion, physical separation by absorbed water layers) predominate over the natural aggregating forces (*van der Waals*) and the natural mechanism (Brownian movement) which tends to cause particle contact. Coagulation of the fine particles involves both de-stabilization and physical processes that disperse coagulants and increase the opportunity for particle contact.

Design of chemical treatment facilities for removal of suspended solids must take into account the types and quantities of chemicals to be applied as coagulants, coagulant aids and for pH control and the associated requirements for chemical handling and feeding and for mixing and flocculation after chemical addition.

In spite of its short detention time and low capital cost, chemical coagulation has found little application in the treatment of POME. This is a result of a number of factors including insufficient proof of the effectiveness of the technique, the high cost of chemicals for coagulation as well as for pH adjustment. Besides, problems associated with dewatering and disposing of generated sludge and high concentration of residual cation level which remain in the supernatant also limit operation of this process in actual scale.

The main purpose of this study is to scrutinize the coagulation process capability to bring about mass reduction in organic content of the POME supernatant. Settleable solids will be first removed through sedimentation to avoid erroneous results in the coagulation process. Removal efficiencies for parameters BOD, COD and SS will be the main aspects to gauge the process performance.

## MATERIALS AND METHODS

Sedimentation test was carried out in a glass column 22 cm in diameter and 30 cm height. The required proportions of fresh POME and distilled water were mixed and shaken properly before being poured into the column and stirred again using a glass rod. The relatively clear layer levels of the supernatant over the solids settlement layer were taken as interface heights and were measured against time.

POME supernatants at 3 different concentrations were used throughout this study except for the sedimentation test. They were prepared as described in the sedimentation test above which was first diluted with a dilution factor of 4 (to be denoted concentration 1), 5 (concentration 2) and 8 (concentration 3). After mixing followed by a standing period of 30 minutes, the supernatants were carefully siphoned out and used in a series of jar test experiments.

Optimization of coagulation and flocculation processes were carried out using Jar test stirring apparatus operating at 65 rpm with a 3 minute stirring time allocated after coagulant addition. Four types of coagulants were used *vis.* ferrous sulfate ( $\text{FeSO}_4$ ), ferric chloride ( $\text{FeCl}_3$ ), aluminium sulfate (alum) and polyaluminium chloride (PAC) and their dosages were varied between 0 to 600 ppm. The coagulants optimum dosages determination was carried out at constant pH of 4.5 and its performance was determined by the degree in reduction of BOD, COD and SS. pH influence was studied after determination of the optimum dosages. Variation in pH within the range of 2-9 was carried out using either 6M NaOH or 3M  $\text{H}_2\text{SO}_4$  to obtain the optimum pH value for each coagulant at their optimum dosages. The jar test stirring apparatus was stopped immediately after the addition of hydrolyzed polyacrylamide based anionic polyelectrolyte as coagulant aid.

Samples were taken after 2 hours of standing to simulate the effect of average settling tank retention time design (1-3 hours). Here, 50 ml of the supernatant was pipetted out making sure the tip of the pipette submerged about 1 cm from the surface to avoid floating particles.

In this experiment, coagulant aid served as secondary floc promoting agent to speed up settling of coagulated particles. The effect of coagulant aid dosage was also demonstrated by varying the dosage between 0 to 3 ppm at optimum coagulant dosages and pHs.

Parameters BOD, COD and SS of the sample were carried out using APHA standard methods (APHA, 1992). BOD was measured using the incubation period a 3 days at 30°C. Monitoring of the POME supernatant pH was performed using ORION (Model 410A) pH meter.

## RESULTS AND DISCUSSION

Based on the theory of gravity settling, the higher the concentration of the suspension is, the slower the settling rate. This can be explained by an increase in upward velocity of the displaced liquid (Metcalf and Eddy, 1991). This phenomenon is shown in Figure 1. The same pattern of settling resembles the study of sedimentation using calcium carbonate suspension (Coulson *et al.*, 1991).

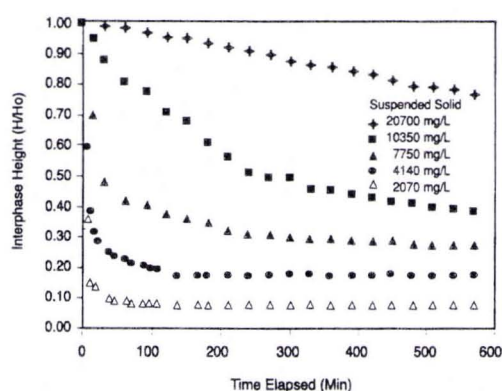


Figure 1. Settling of settleable POME solids at different concentrations

In a typical POME, the high content of its suspended solids forms a 'structure' and settling will occur at the compression of this structure. This is a characteristic of a hindered settling. Hindered settling occurs when inter particle forces are strong enough to hinder settling of the adjacent particles. Thus, the particles tend to remain at the position relative to each other and settle as a zone or blanket (Eckenfelder, 1989; Hammer and Hammer, 1997).

Settling of solid layers in POME occurs only at the compression of the lower structure due to continuous addition of weight on the upper layer during its settling from the supernatant. In general, the compression of the sediment can be represented by the equation below (Coulson *et al.*, 1991):

$$H_{t_1} - H_{\infty} = (H_{t_2} - H_{\infty})e^{-i(t_1-t_2)}$$

Where,

- $H_{t_1}$  = height of sediment at time  $t_1$
- $H_{\infty}$  = height of sediment at a very long time, say 24 hours
- $H_{t_2}$  = height of sediment at  $t$
- $i$  = constant

The separation process of POME settleable solids through gravity settling is a very slow process at its original concentration. As such, the retention time of the separation unit will increase to the extent that it is not economical to be built.

As described earlier, three different POME settleable solid-free supernatants were used in the jar test experiments in this study. The concentration of each of them, characterized by their BOD, COD and SS contents are as shown in Table 2 below.

**Table 2.** Mean BOD, COD and SS content for each sample concentration used

Sample	Parameter (mg/L)		
	BOD	COD	SS
Concentration 1	4749	9983	1700
Concentration 2	4099	7972	1325
Concentration 3	3483	5398	1081

The efficiency of the BOD, COD and SS removals at different coagulant dosages were thoroughly studied and the results are as depicted in Figure 2. The optimum removal efficiency of the coagulants is found to occur at different dosages. Alum and PAC for example, showing the best result at a dosage of about 350 ppm and 300 ppm respectively. As for iron salts, a comparatively lower dosage of about 200 ppm was found to be the optimum value. However, the performance of iron salts fell below the efficiency of PAC and alum even at similar concentrations.

The addition of coagulants exceeding their optimum dosages is found to give no additional positive effect especially for iron salts, which reduces the separation process. This might be due to counter ion re-stabilization causing the dispersion of the flocs and subsequently affecting the settling of the particles (Stephenson *et al.*, 1996).

In an aqueous solution, trivalent cations of aluminium and iron are similar in nature. When the salts are soluble in water, metal ions (M) become hydrated and undergo hydrolysis forming monomeric species:  $\text{MOH}^{2+}$ ,  $\text{M}(\text{OH})_2^+$ ,  $\text{M}_2(\text{OH})_2^{4+}$ ,  $\text{M}(\text{OH})_4^{5+}$ ,  $\text{M}(\text{OH})_3(\text{s})$  and  $\text{M}(\text{OH})_4^-$  (Dentel and Gossett, 1988). Under very acidic conditions, both  $\text{Al}^{3+}$  and  $\text{Fe}^{3+}$  exist in the solution. However, if the pH or the coagulant concentration is increased, hydrolysis takes place giving metal hydroxides,  $\text{M}(\text{OH})_3(\text{s})$ . The general trivalent metal ion hydrolysis reaction is as proposed by Ching *et al.*, (1994):



Polymeric metal hydroxides produced, generally having large surface area, amorphous and positively charged (Randtke, 1988). They are hydrophobic in nature causing them to attach to anionic organic particles and reduce their solubility (Dentel and Gossett, 1988; Ching *et al.*, 1994). Aluminium and iron have the tendency to produce non soluble complexes with polar molecules and oxygen containing functional groups such as hydroxyl and carboxyl (Licisko, 1993). Charge neutralization will destabilize the colloid and cause settling of the metal cations together with organic anions (Jekel, 1986).

Study by Solvolainen (1993) showed that the coagulation mechanism of the ferrous sulfate involves oxidation Fe (II) to Fe (III). This explains why the efficiency of the former is comparatively lower than Fe(III) (Figure 2). Except this oxidation, other reaction schemes for both salts are similar in nature.

The merit of using iron coagulants over aluminium is the production of tougher and denser flocs. Besides, they can operate at broader pH range and less sensitive to overdosage (Liang *et al.*, 1993).

The net charge per aluminium atom is +0.5 which implies that highly positively charged species are formed in solution and consequently it should in theory be a good coagulant (Parthasarathy and Buffle, 1985). Tipping *et al.* (1988) concluded that hydrolytic behavior of aluminium coagulants poses significant effect on the removal efficiency, flocs settling, characteristics of the sludge and the concentration of the residual aluminium escapes with the effluent. The use of alum normally increases the sulfate ions and total solids in the effluent creates a setback to the wastewater treatment in the broader sense (Stephenson *et al.*, 1996). It has been said that, PAC is much less affected by temperature than alum (Benschoten and Edzwald, 1990).

The addition of ferric chloride resulted in fine grey-brown flocs which tend to form large amorphous aggregates. Above the poorly settling floc, the translucent supernatant was very dark brown in color (Zuhairi, 2000). FeSO<sub>4</sub> coagulation led to an opaque tea-brown supernatant and light brown flocs some of which settled and others floated on the surface of the liquid.

The net charge of the colloidal particles is governed by the type of the colloidal system and the medium pH. The charge will change with a change in pH. At higher pH, the colloidal particles tend to be negatively charged while at lower values, the particles remained in cationic form. The charge balance can actually be associated with changes in H<sup>+</sup> and OH<sup>-</sup> ions to maintain the ion balance with water at different pHs.

Figures 3a, b and c depict changes in BOD, COD and SS removal efficiencies at different pH values. As can be seen, the optimum coagulation occurred at pH of between 4 to 5. This range coincides with the pH of a typical fresh POME. In other words, effective coagulation can take place without pH adjustment for fresh untreated POME.

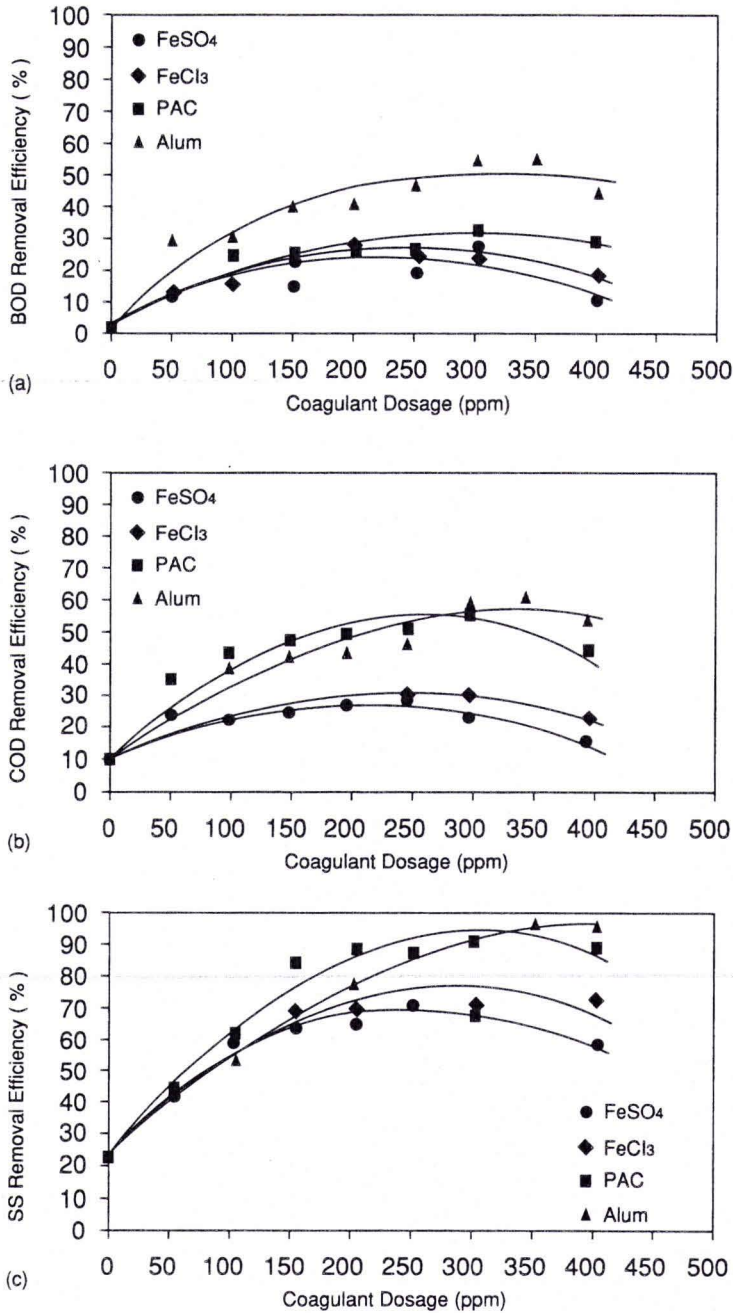
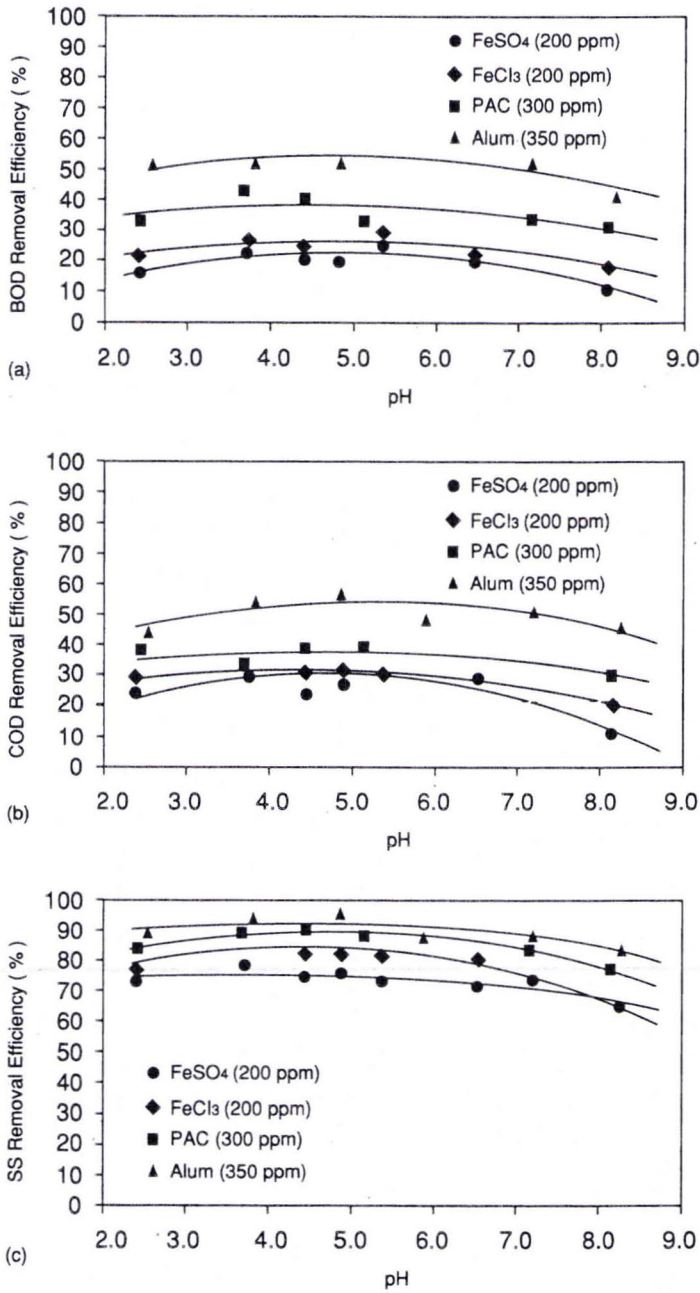


Figure 2. Effect of coagulant dosage on a) BOD, b) COD and c) SS removal efficiencies. (Conditions : pH=4.5, Coagulant aid dosage = 1.0 ppm)



Chemical Coagulation of Palm Oil Mill Effluent (POME)



**Figure 3.** Effect of pH on a) BOD, b) COD and c) SS removal efficiencies at optimum coagulant dosages and 1.0 ppm coagulant aid

The removal efficiency is markedly reduced when pH is further raised beyond 6 for all the coagulants studied. This reduction was due to the higher concentration of OH<sup>-</sup> ion that will compete with organic molecules for adsorption sites. In addition to that, settling of metal hydroxides is unavoidable at high pH (Stephenson *et al.*, 1996). Ching *et al.* (1994) in their study concluded that at high pH, the charge of the coagulating species will become less positive and as a result, less attracted to anionic organic compounds. Besides, the solubility of the metal coagulant-precipitate solids was found to be strongly dependent on pH and precipitate which was formed under acidic conditions (Stephenson *et al.*, 1996).

The addition of coagulant aids is normally done to improve coagulation and to speed up the settling of the resulting flocs. High floc settling velocity is very much needed as it can be translated into smaller settling tank required for the operation in wastewater treatment (Simethurst, 1979; Negulescu, 1985). The coagulant aids however, are not likely to contribute positively in the coagulation process (Sastri, 1996).

Sinsabaugh *et al.* (1986) suggested that solutes are removed through two distinct mechanisms during metal salt-enhanced coagulation and precipitation. At low pH, the anionic organic molecules react directly to form insoluble complexes. Adsorption of organics onto the pre-formed metal hydroxide flocs followed by precipitation is the more dominant mechanism at higher pH values.

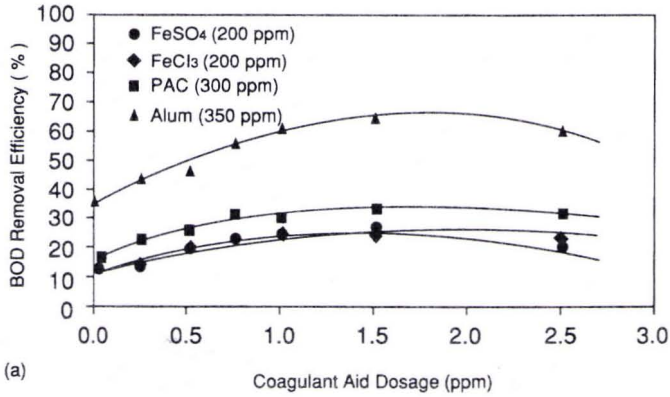
In some liquid of high buffer capacity like the POME, adjustment of pH to the optimum value is in fact, not feasible. Here, the flocculation pH is very often almost neutral, where the predominant formation of insoluble hydroxide is expected inducing the 'sweep flocculation' due to the enmeshment of particulate matter by the voluminous hydroxide precipitate.

Figures 4a, b and c illustrate the effect of different dosages of the coagulant aid on BOD, COD and SS removal efficiencies. As depicted in the figure, the optimum dosage occurred at about 1.0 ppm and further addition reduces the efficiency of the process. This result correlates to the conclusion made by Tan (1997) in his study on coagulation process of wastewater from textile industries.

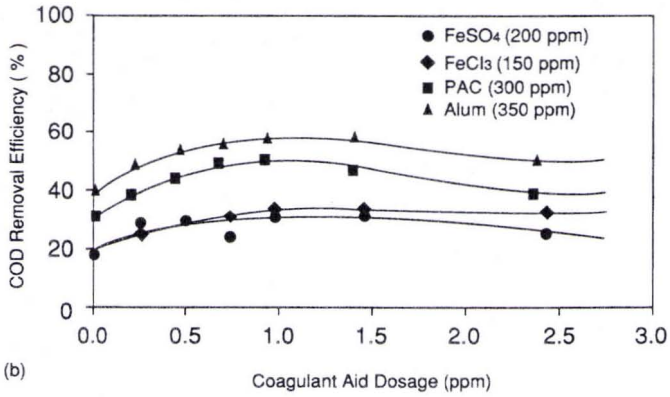
Reduction in removal efficiency at higher dosage of coagulant is due to its electronegative property. Anionic polyelectrolyte used as coagulant aid in this study actually replaced the anionic groups on POME colloidal particles and permitted hydrogen bonding between the colloid and the polymer. Over dosages however, can cause re-stabilization of the particles and consequently hinder the formation of flocs (Eckenfelder, 1989).

The BOD, COD and SS removal efficiencies of each of the coagulant used for each of the POME supernatant concentrations (characterized in Table 2) are as shown in Figures 5, 6 and 7. As can be clearly observed, the removal efficiency dropped when higher strength

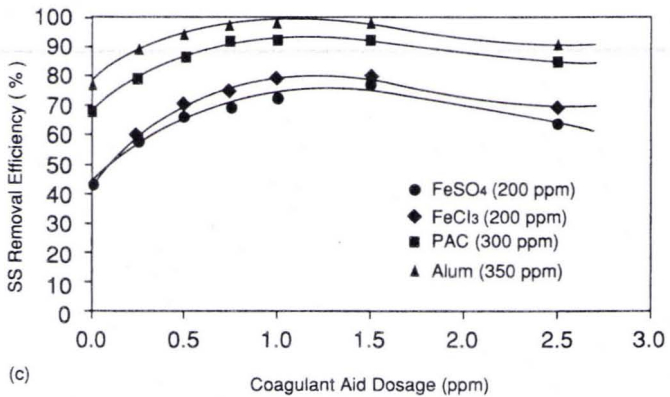
Chemical Coagulation of Palm Oil Mill Effluent (POME)



(a)

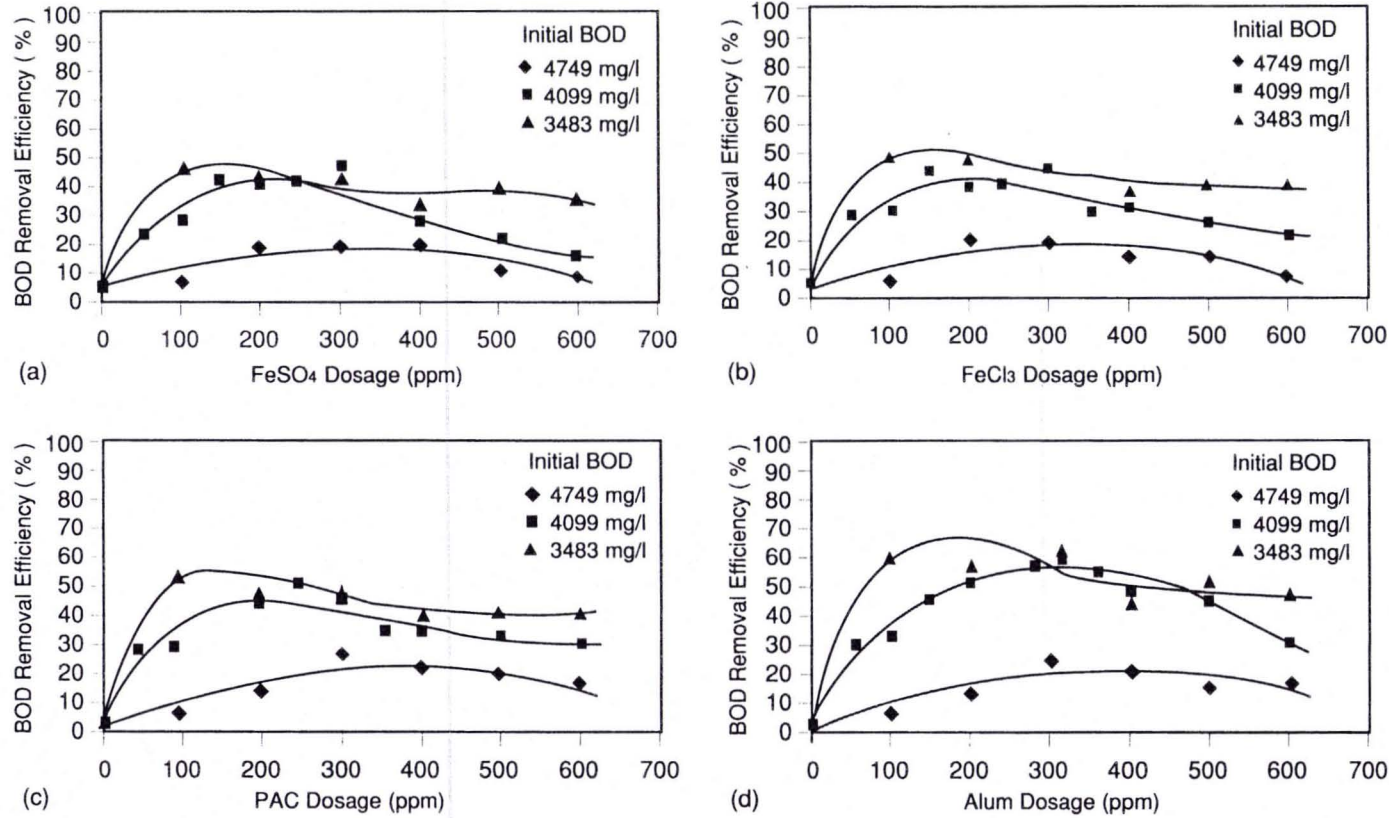


(b)

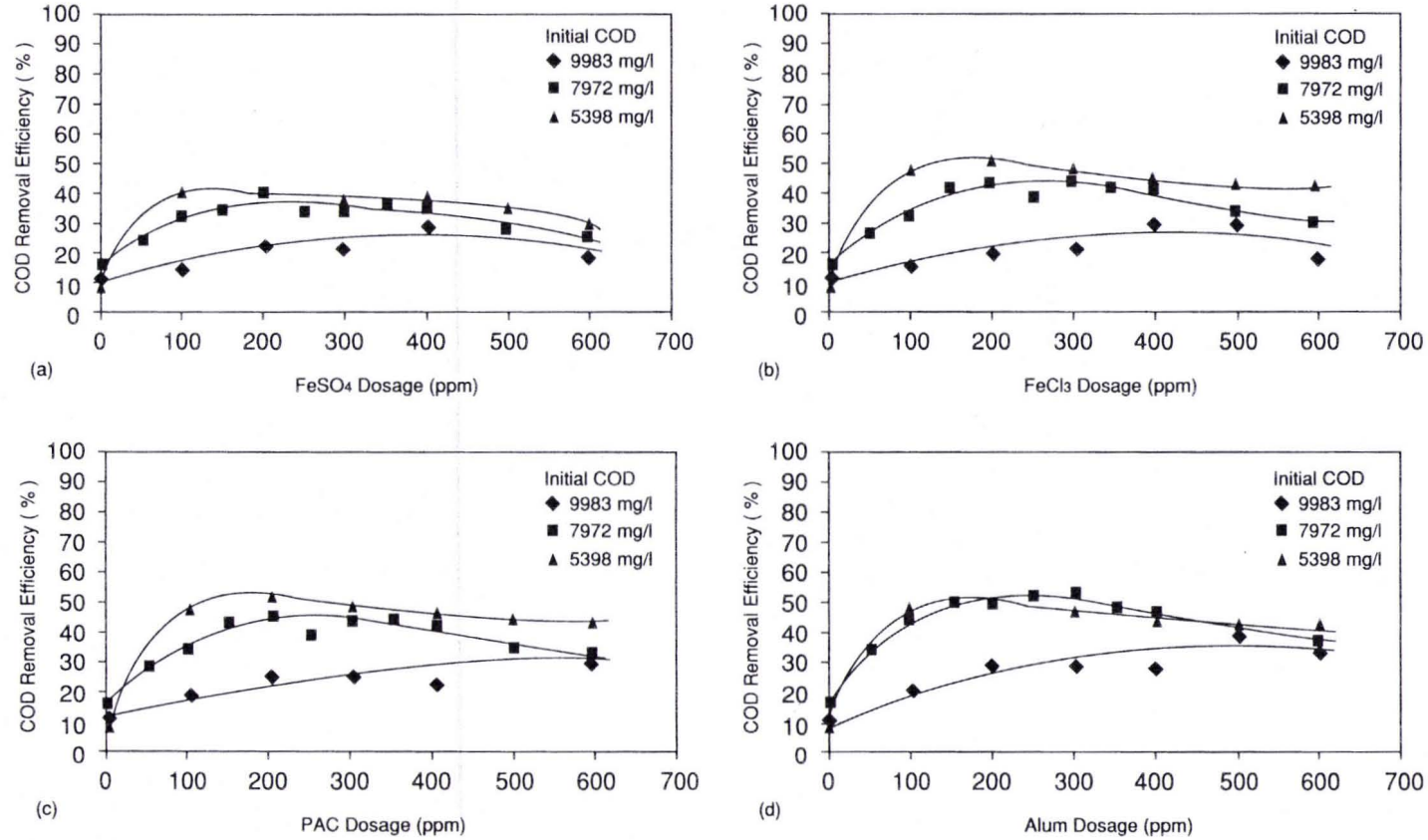


(c)

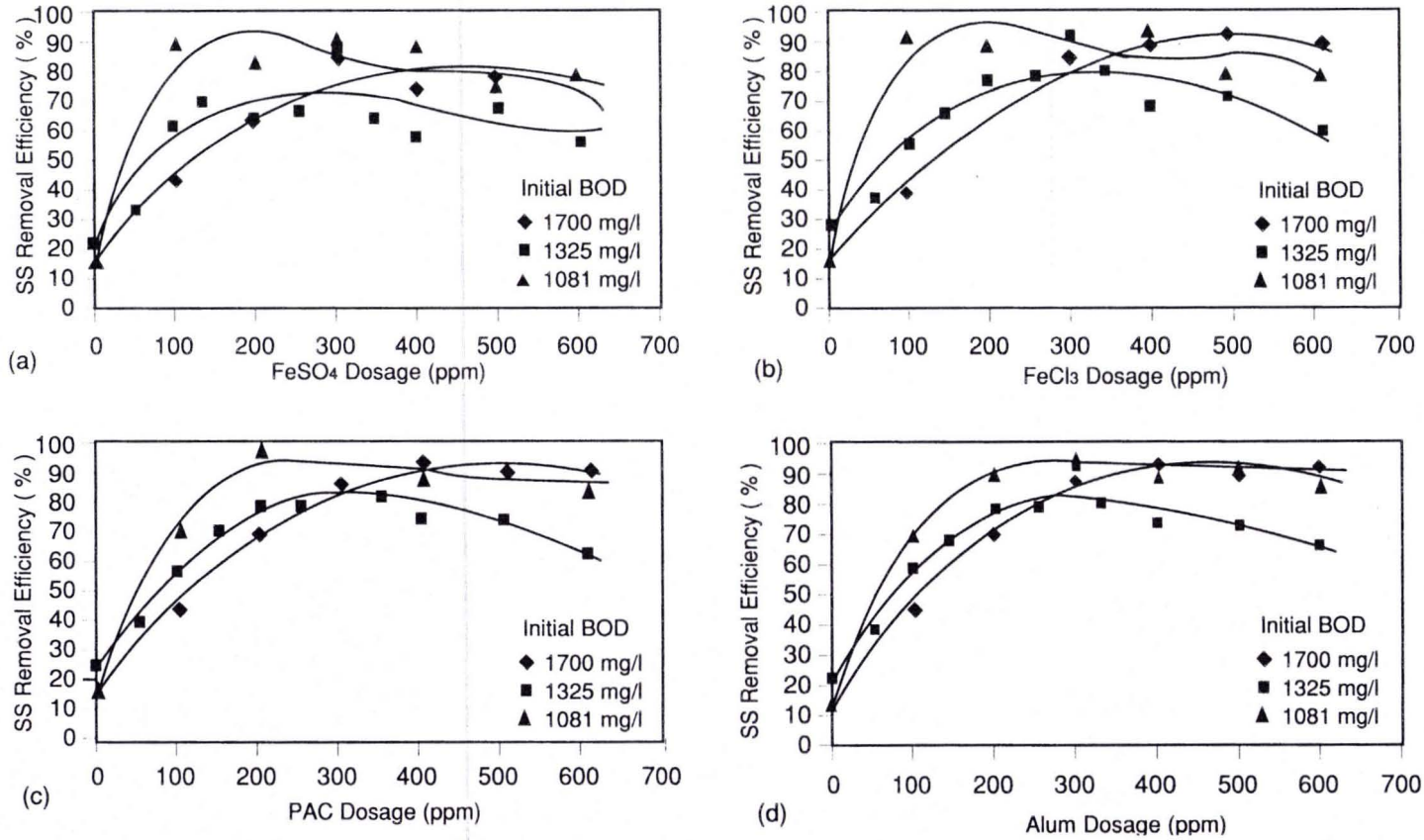
Figure 4. Effect of coagulant dosage on a) BOD, b) COD and c) SS removal efficiencies using different coagulants at their optimum dosages and constant pH=4.5



**Figure 5.** Effect of wastewater concentration on BOD removal efficiencies for treatment using a) FeSO<sub>4</sub>, b) FeCl<sub>3</sub>, c) PAC and d) Alum (Conditions : Coagulant pH = 4.5, coagulant aid dosage = 1.0 ppm)



**Figure 6.** Effect of wastewater concentrations on COD removal efficiencies for treatment using a)  $\text{FeSO}_4$ , b)  $\text{FeCl}_3$ , c) PAC and d) Alum (Conditions : Coagulant pH = 4.5, coagulant aid dosage = 1.0 ppm)



**Figure 7.** Effect of wastewater concentrations on SS removal efficiencies for treatment using a)  $\text{FeSO}_4$ , b)  $\text{FeCl}_3$ , c) PAC and d) Alum (Conditions : Coagulant pH = 4.5, coagulant aid dosage = 1.0 ppm)

of the wastewater was used. For example, for alum, maximum removals of about 58%, 48% and 93% were demonstrated for BOD, COD and SS respectively when concentration 3 was used. When higher strength of concentration 1 was used, the corresponding efficiencies were 25%, 30% and 83% respectively.

Figures 5, 6 and 7 also show that optimum coagulant dosages increased with increase in wastewater concentration. PAC for instance, demonstrated maximum BOD removal efficiency at about 100 ppm when the supernatant with BOD concentration of 4749 mg/l (concentration 1) was used (Figure 5c). When the supernatant with BOD concentration of 3483 mg/l was used, maximum removal occurred at a dosage of about 350 ppm. A similar trend was also observed in COD (Figure 6c) and SS (Figure 7c) removal efficiencies.

The results of this study support almost similar work done by Stephenson and his co-workers (1996). They found that the performance of the coagulation process was governed by type and dosage of the coagulant as well as the concentration of the wastewater used. The effect of dilution however, is not straightforward as it can change the form of the soluble organics (Ching *et al.*, 1994). Due to limited solubility, the organics might be present in the form of colloidal suspension before dilution.

It is generally accepted that every metal salt will form a unique non-soluble hydroxide in water. As such, there is no reason to conclude that there is a consistent relationship between coagulant dosage and wastewater concentration (Stephenson *et al.*, 1996). In this study, the emphasis was given on the general interaction between the two as what had been suggested by Rebhun and Lurie (1993).

Humic acids are among the most recalcitrant organics in wastewater treatment. Their presence normally requires the treatment system to operate at high retention time, sometimes, at unsatisfactory efficiency. Metal hydrolysis products derived from ferum and aluminium can act as polycations that participate in charge neutralization with humic substances to form insoluble humates or fulvates (Kuo *et al.*, 1988). It was reported that coagulation of humic substances produced a colloidal sol which could be effectively removed by centrifugation or filtration (Beulker and Jekel, 1993).

Since POME has long been known for its organic burden to the treatment, the removal efficiency resulting from chemical coagulation is greater than what can be expected from biological treatment. This study has proved that a significant portion of recalcitrant compounds can be removed from the solution. Therefore, the biodegradability of the supernatant is expected to be greatly enhanced.

## CONCLUSION

Chemical coagulation is the fastest way to reduce the organic load of the POME to an acceptable and economical level to be treated using conventional treatment systems. Up to 60% removal of the BOD and COD content and 90% of the SS content seems to be within reach with proper selection of coagulant and its optimum dosage. The final effluent, in the absence of settleable solids as well as colloidal and some soluble solids removed during coagulation process is expected to be much more amenable to biological treatment process as compared to fresh POME.

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