

COAGULATION-SEDIMENTATION-EXTRACTION PRETREATMENT METHODS FOR THE REMOVAL OF SUSPENDED SOLIDS AND RESIDUAL OIL FROM PALM OIL MILL EFFLUENT (POME)

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ABSTRACT: Suspended solids and residual oil removal in a liquid are relevant to numerous research areas and industry. The suspended solid cannot be removed completely by plain settling. Large and heavy particles can settle out readily, but smaller and lighter particles settle very slowly or in some cases do not settle at all. Because of this, it requires efficient physical-chemical pretreatment methods. Our current research is to study the pretreatment methods in the removal of suspended solids and residual oil content in POME. Preliminary analysis shows that POME contains 40,000 mg/L suspended solid and 4,000 mg/L oil and grease content that relatively very high compared to the maximum allowable limit by the Malaysian Department of Environment which are only 400 mg/L and 50 mg/L respectively. The methods chosen were coagulation-sedimentation method for suspended solids removal and solvent extraction for residual oil removal. Jar test apparatus was used as the standard procedure for bench-scale testing and alum was used as the coagulant. Parameters studied were alum dosage, mixing time, mixing speed, sedimentation time and pH. For removal of residual oil, six different organic solvents; n-hexane, n-heptane, benzene, petroleum ether, pentane and petroleum benzene were used. For every solvent the effect of solvent ratio, mixing time, mixing speed and pH were analyzed. The results show that the optimum conditions in removal of suspended solid from POME were at pH 4.11, sedimentation time of 100 minutes and 150 rpm mixing speed with 1.5 hr mixing time. N-hexane give the best performance in extracting residual oil from POME with solvent to POME ratio of 6:10. It was estimated about 0.54 grams of oil and grease can be extracted with optimum variables at pH 4, mixing speed of 200 rpm, and 20 minutes mixing time.

Key Words: palm oil mill effluent, coagulation, suspended solid, residual oil, solvent extraction.

1. INTRODUCTION

Palm oil mill effluent (POME) represented the highest by-products generated by palm oil mills, which is about 21 %. POME is a colloidal suspension of 95%-96% water, 0.6% - 0.7% oil and 4%-5% total solids including 2%-4% suspended solids originated from the mixture of

a sterilizer condensate, separator sludge and hydrocyclone wastewater ^[1]. The quantity of POME produced is about 60% for every tonne of fresh fruit bunches (FFB) processed. Thus, an average of 30 tonnes FFB/hr mill in this country will generate about 18 – 19.5 tonnes effluent/hr ^[2]. With over 300 palm oil mills in Malaysia, generation of large volumes of effluent could give significant impact to the environment since the characteristics of POME are extremely polluting with its high organic content. The characteristic of raw POME is shown in Table 1^[1]. The discharge of untreated POME into the river will undergo natural decomposition during which dissolved oxygen in the river water is rapidly depleted. This will certainly result in the decline or destruction of aquatic lives and deterioration of natural ecosystem. Therefore POME requires a sound and efficient management system in utilization, treatment and disposal.

Table 1 Characteristics of palm oil mill effluent.^[1]

Parameter	Conc.*	Element	Conc.*
pH	4.7	Phosphorus	180
Oil and grease	4,000	Potassium	2,270
Biochemical oxygen demand	25,000	Magnesium	615
Chemical oxygen demand	50,000	Calcium	439
Total solids	40,500	Boron	7.6
Suspended solids	18,000	Iron	46.5
Total volatile solids	34,000	Manganese	2.0
Ammoniacal nitrogen	35	Copper	0.89
Total nitrogen	750	Zinc	2.3

*All parameters in mg/L except pH.

Looking at the seriousness of environmental impact by palm oil industry, the government of Malaysia has enacted the Malaysian Environmental Quality Act, 1974 and Environmental Quality (Prescribed Premises)(Crude Palm Oil) Regulations 1977. The Malaysian Department of Environment, DOE, has set the discharge standards for POME as shown in Table 2 ^[3]. Therefore, the palm oil industry faces the challenge of balancing the environmental protection, its economic viability and sustainable development. There is an urgent need to

find a way to preserve the environment of our country while keeping her economy healthy.

Table 2 Parameter limits for watercourse discharge of POME [3]

Biochemical oxygen demand (BOD) (mg/L)	100
Suspended solids (mg/L)	400
Oil and grease (mg/L)	50
Ammoniacal nitrogen (mg/L)	150
Total nitrogen (mg/L)	200
pH	5-9

BOD – sample incubated for 3 days at 30 °C

Several innovative treatment technologies have been developed and applied to palm oil mills to treat POME, and conventional biological treatments of anaerobic or facultative digestion are the most commonly used [4]. However, these biological treatment systems need proper maintenance and monitoring as the processes rely solely on microorganisms to break down the pollutants. The microorganisms are very sensitive to changes in the environment and thus great care has to be taken to ensure that a conducive environment is maintained for the microorganisms to thrive in. Besides, it also generates vast amount of biogas. This biogas contains methane, carbon dioxide and trace amount of hydrogen sulphide where these gases are corrosive and odorous.

Besides the biological treatment, evaporation process has been suggested to treat POME [1]. By using POME containing 3-4 % total solids as feed, about 85 % of the water in the POME can be recovered as distillate. Unfortunately energy requirement is a major constrain in this process.

Another physical treatment method, air-floatation systems was tested on a pilot plant scale. In this system, tiny, bubbles of air or oxygen are released in the effluent. In rising the surface of the effluent, this bubbles carry suspended solids to the surface where they are easily removed. However it was found to be very inefficient and unreliable [5].

Full scale tests were carried out using centrifuge process. The suspended solids are separated from the liquid or concentrated by means of centrifuges but the system found to be unsatisfactory due to high wear rates and erratic operation [5].

Treatment of Palm Oil Mill Effluent (POME) requires a sound and efficient system in facing the current challenge in compliance to the discharge standard regulation, environmental protection and economic viability. Biological treatment systems are widely applied for treating POME however there are still some mills that fail to comply with the regulatory standard set by The Malaysian Department of Environment (DOE). Therefore an alternative POME treatment system based on membrane separation technology is being studied and a promising result was observed. The proposed treatment consists of two stages of treatment; series of pretreatment processes (i.e. Coagulation-sedimentation-solvent extraction) and membrane separation processes.

1.1 Coagulation

Coagulation process can alter the suspended particles so that they can adhere to each other. During coagulation, a positive metal ion is added to water to reduce the surface charge to the point where the particles are not repelled from each other. Chemicals (coagulant) are added to the water to bring the nonsettling particles together into larger, heavier masses of solids called floc [6]. Technically, coagulation applies for the removal of colloidal particles or suspended solids.

The most commonly used coagulants are [7]:

- Alum (aluminium sulfate), $Al_2(SO_4)_3 \cdot 14H_2O$ is the most common coagulant and is often used in conjunction with cationic polymers.
- Polyaluminium chloride, $Al(OH)_x(Cl)_y$, is efficient in some waters requiring less pH adjustment and producing less sludge.
- Ferric chloride, $FeCl_3$, may be more effective than alum in some application.
- Ferric sulfate, $Fe_2(SO_4)_3$, is effective in some waters and more economical in some locations.
- Cationic polymers can be used alone as the primary coagulant or in conjunction with aluminum or iron coagulants.

Eilbeck and Mattock [8] presented a list of common coagulants in treating wastewater from various industries such as food, textiles, pulp and paper. They found that aluminum sulphate was the most suitable coagulant. While a study carried out by Lin et al. [9] found that coagulation using polyaluminum chloride (PAC) and polymer (coagulation aid) was found to be effective as a pretreatment for partial removal of suspended solids, color and chemical oxygen demand of many industrial wastewaters.

1.2 Solvent extraction

Solvent extraction method lately is the most powerful separation and purification tool in the process industries. It is used commercially in hydrometallurgy (including nuclear industry) and widely used within the chemical industry including organic chemical, petrochemical and pharmaceuticals.

The principle is illustrated in a separatory funnel which contains two layers of liquids. One that is generally water (S_{aq}) and the other, generally, an organic solvent (S_{org}). Normally the organic solvent has a lower density than water, but the opposite situation is also possible. A solute A, which is initially dissolved in only one of the two liquids, eventually distributes between the two phases. When this distribution reaches equilibrium, the solute is at concentration $[A]_{aq}$ in the aqueous layer and at concentration $[A]_{org}$ in the organic layer. The distribution ratio of the solute [10], D is defined as

$$D = [A]_{org} / [A]_{aq} \quad (1)$$

“the total analytical concentration of the substance in the organic phase to its total analytical concentration in the aqueous phase, usually measured at equilibrium” [11], irrespective of whether the organic phase is the lighter or heavier one.

Process efficiency will depend on the equilibrium distribution of the constituents between the wastewater and the extracting solvent and the kinetics of mass transfer between the two liquid phases. To increase efficiency, the solvent-to-waste ratio can be increased, or multiple extraction stages can be employed.

Solvent extraction has assumed importance in virtually all vegetable oil recovery plant. It recovers up to 98 percent of cottonseed oil. The soybean, which has low oil content, but whose physical structure is particularly suited to solvent extraction has been responsible for this development and solvent hexane is used for this purpose [12]. While in pharmaceutical industry, solvent extraction method is extensively used for purification and concentration of penicillin by utilizing n-butyl acetate or amyl acetate as solvent [10].

2. MATERIAL AND METHODS

2.1 Experimental materials

POME was collected from Felcra Nasaruddin Palm Oil Mill, Ipoh, Perak and cooled in a refrigerator from the temperature of 90°C to 0°C. Technical grade alum [Al₂(SO₄)₃ · xH₂O (x=13-16 H₂O)] obtained from Merck, Germany, was used for the preparation of the alum solution. Six technical grade solvents obtained from Quicklab Sdn. Bhd. (Perak); n-hexane, benzene, pentane, petroleum benzene, petroleum ether, and n-heptane were used as solvents in solvent extraction process. In the present study, benzene has been chosen only to study its performance for comparison with other solvent, although benzene is known to be toxic and carcinogen.

2.2 Experimental Procedure

2.1.1. Removal of Suspended Solid using Coagulation and Sedimentation Process

The jar test was performed using six beakers filled with 250 ml of POME and diluted with 750 ml of distilled water. 1 grams of aluminium sulphate (alum) equal to 1000 mg/L was added in each beaker. Each beaker was mixed and flocculated uniformly using rapid mixing rate of 150 rpm for 10 minutes and slow mixing rate of 25 rpm for 20 minutes. The mixture was left to settle for 30 minutes. The parameters varied were alum dosage (1,000 – 7,000 mg/L), mixing speed (0 – 300 rpm), mixing time (0 – 3 hours), pH (4 – 9) and sedimentation time (0 – 420 minutes).

2.1.2. Removal of Residual Oil using Solvent Extraction

The experiments were conducted to evaluate the performance of six types of organic solvents, namely n-hexane, benzene, petroleum benzene, petroleum ether, pentane and n-heptane in extracting residual oil from POME. It was performed using three beakers filled with 300 ml of POME. Then, each beaker was filled with 60 ml of solvent and covered with an aluminum foil to

prevent solvent evaporation and mixed in a flocculator for 5 minutes at the speed of 50 rpm. The mixture was left for another 5 minutes to let it separated into two phases. These two phases were then separated into two different beakers to be analyzed.

In solvent extraction process, several parameters were varied. The variables were ratio of solvent to POME (2:10 – 10:10), mixing rate (50 – 250 rpm), mixing time (0 – 25 minutes) and pH of the sample (4 – 9).

3. ANALYTICAL METHOD

After sedimentation, 5 ml of the supernatant sample was pipetted and diluted to 25 ml with distilled water. The samples were analyzed for turbidity using a 2100P Turbidity meter (Hach. Company, Colo, U.S.A.). This value was comparable to the concentration of suspended solids in sample.

In order to determine the oil concentration in the treated sample, the extracted POME was transferred to a separating funnel. The POME container was rinsed with 30 ml of petroleum ether and solvent washings were added to the separating funnel. The mixture was shaken vigorously for 2 minutes and the layer was left for further separation for 5 minutes. The aqueous layer was drained into a sample container. The extraction was repeated with another two portion of 30 ml petroleum ether. The solvent layer was drained through a funnel containing a filter paper and 2 g anhydrous sodium sulphate, both of which have been solvent-rinsed, into a clean conical flask. The solvent was then distilled off using the rotary evaporator. The drying was completed in the oven at 103 °C for 5 to 10 minutes. The flask was cooled in a desiccator for about 30 min and weighed. The drying and cooling steps were repeated until the weight becomes constant to calculate the concentration of residual oil extracted. The experiment was repeated 3 times and the results were reproducible within an error of ± 5%.

For determining the concentration of residual oil after the adsorption process, a calibration curve was made. This calibration correlates the concentration of residual oil with its absorbance. The concentration of residual oil was obtained from the solvent extraction process while its absorbance was measured by using a spectrophotometer (Cecil 100 series) at a wavelength of 444 nm. The overall mass balance was verified by considering the removal efficiency at various stages in the process.

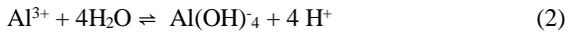
4. RESULTS AND DISCUSSION

4.1 Coagulation

The figures presented below show the results of batch experimental procedure for five samples that were represented as A, B, C, D and E respectively.

a) Effect of alum dosage

The effect of alum dosage on the coagulation process is given in Fig. 1. Alum dosage was varied from 0 to 7,000 mg/L. while other parameters were kept constant at pH 4, 150 rpm for mixing rate, 10 minutes of mixing time and 30 minutes for sedimentation time. Figure 2 shows a significant reduction in turbidity value with the increase in alum dosage until it reached the concentration of 4,000 mg/L. The turbidity reduced from 14,080 NTU to 1,615 NTU. The trend shows the increased in Alum dosage leads to the reduction in turbidity. This phenomenon was explained by the chemical reaction during coagulation process ^[13]



Whereby the turbidity of the sample were reduced due to the formation of the aluminium hydroxide flocs that settle to the bottom of the beaker.

However, after reaching 4,000 mg/L point, the turbidity value almost reached steady state even with the increased of alum dosage. This result shows that the optimum coagulation will occur when the concentration of the alum is 4,000 mg/L. At this dosage, the zeta potential is zero and this is defined as the isoelectric point. Zeta potential is used since it can represent the stability of the particle in the waste. At isoelectric point, almost all the particles charge have been neutralized by the high valence cations and this induce coagulation and precipitation.

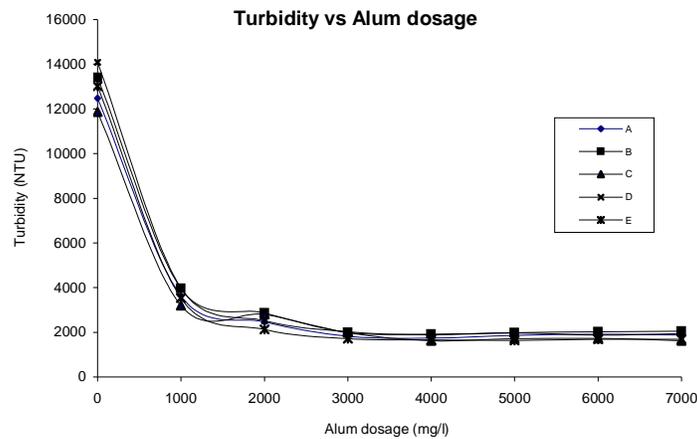


Fig. 1: The effect of Alum dosage on suspended solid removal from POME

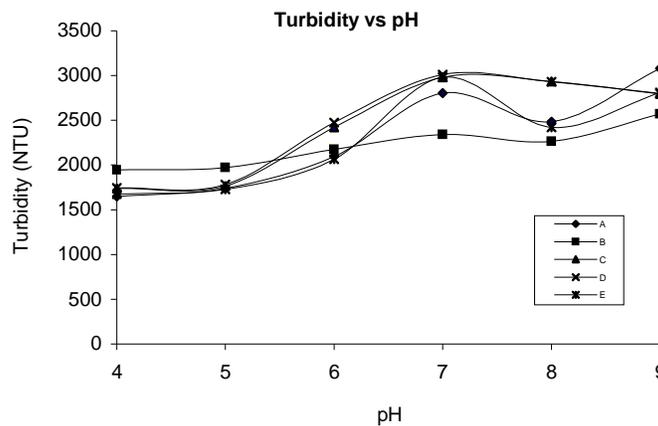


Fig. 2: The effect of pH on suspended solid removal from POME

b) Effect of pH

Fig. 2 shows the effect of pH on flocculation process for the sample of POME. The value of pH were varied from pH 4 to pH 9 with constant parameters of Alum dosage by 4,000 mg/L, 150 rpm mixing rate, 10 minutes mixing time and 30 minutes sedimentation time. It was observed that the best supernatant quality (low turbidity value) was obtained in acidic range, pH 4 to pH 6, while in neutral and alkaline range the turbidity value increased. This explained that in acidic range, particles in POME sample form an unstable suspension and coagulate among them. Besides, the changes in pH value also give the effect of electric charges to the particles, whereby it influenced the particle stability [14].

The concentration of H⁺ ions and the density for negative ions on the surface of particles is low. Thus particles become unstable and attraction forces in between particles keep increasing which lead to formation of bigger coagulant.

While in alkaline range, the quality of the supernatant is poor since the particles form a stable suspensions and coagulation process is not very effective. In addition higher molecular weight of coagulant prevents the process for destabilized scattered particles.

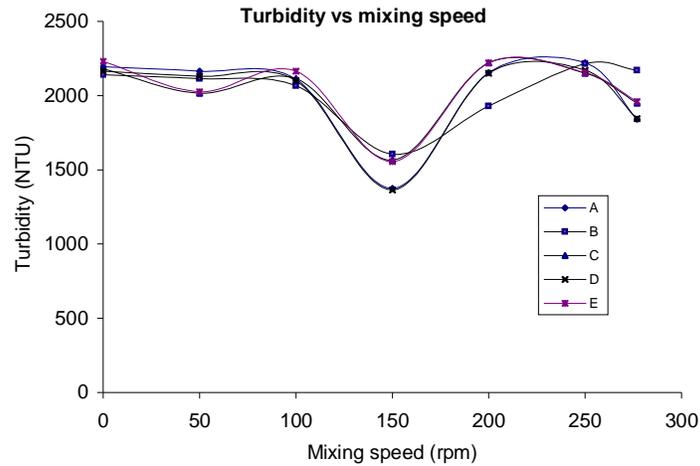


Fig. 3: The effect of mixing speed on suspended solid removal from POME

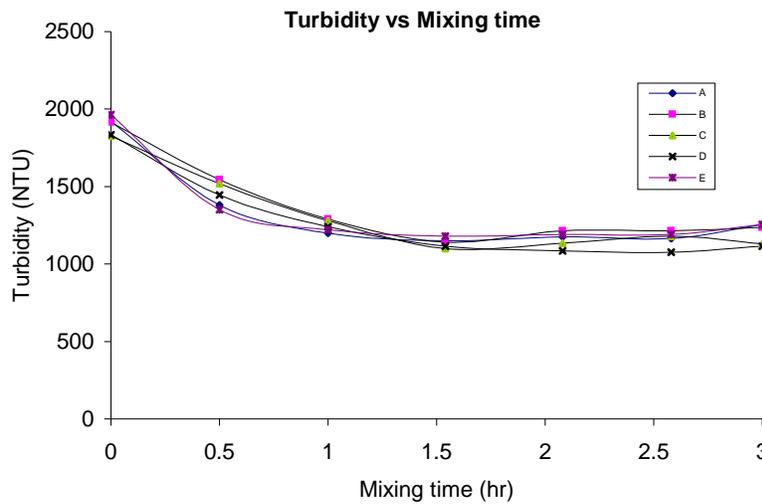


Fig. 4: The effect of mixing time on suspended solid

c) Effect of mixing speed

The effect of mixing speed on coagulation process is shown in Fig. 3. In this experiment the mixing speed were varied from 0 to 300 rpm. Other parameters were kept constant at pH 4, 4,000 mg/L of Alum dosage, 10 minutes mixing time and 30 minutes of sedimentation time. It is obviously seen from the figure that slow mixing speed, range 0 to 100 rpm, is not critical for treating POME. However once the mixing speed is increased to 150 rpm, a dramatically reduction is shown in turbidity value. This is explained that within this mixing rate it provides a suitable condition for coagulation process. However, the turbidity value increases again when the mixing speed is increased. This phenomenon demonstrates that, too high mixing speed will break-up the flocs produced and give poor results on turbidity value. Therefore from the results the optimum mixing speed is 150 rpm.

d) Effect of mixing time

The effect of mixing time to the coagulation process is shown in Fig. 4. This experiment was carried out under the constant parameters at pH 4, Alum dosage of 4000 mg/L, 150 rpm of mixing speed and 30 minutes sedimentation time. The mixing time is varied from 0 to 3 hours. Theoretically, mixing process give a chance to the particles contained in the POME to coagulate to each other. From the graph, the reduction in turbidity started once the mixing time is increase from 0 to 1.5 hours and this represent the condition where flocs are produced and settled to the bottom of the beaker. The turbidity value slightly increased after mixing time exceeded 1.5 hours. This shows that the reaction of coagulant agent and the particles are almost completed at 1.5 hours.

e) Effect of sedimentation time

The effect of sedimentation time is given in Fig. 5. After the addition of 4,000 mg/L of alum dosage to the POME and proceed with mixing speed of 150 rpm for 1.5 hours of mixing time at pH 4, particles will attract to each other and flocs will be performed. From the graph it can be seen clearly that there is a drastic reduction in turbidity value once the sample is left for 0 to 100 minutes of sedimentation time. However the value become constant once it exceeds 100 minutes of sedimentation time. This result explained that almost all flocs produced after the coagulation and mixing process have settled to the bottom of the sludge layer after 100 minutes. The settling process is mainly affected by the gravity where heavier flocs will settle faster than dispersed particles.

4.2 Solvent Extraction

All the figures presented show only the average value of repeating the experiment for five times of all the solvent.

a) Effect of solvent-POME ratio

It is clear from Fig. 6, that as the amount of solvent increases, the percentage of oil extracted also increases. But, after a certain point, the increment in the percentage of oil extracted becomes constant. Even though more oil can be extracted continuously until the solvent-palm oil mill effluent ratio reaches 10:10, but the amount of oil extracted after the ratio of 4:10, are comparable for almost all the solvents. The straight line portion of the curve in the graph show that all the fatty acids content in POME has been extracted to the solvent.

removal from POME

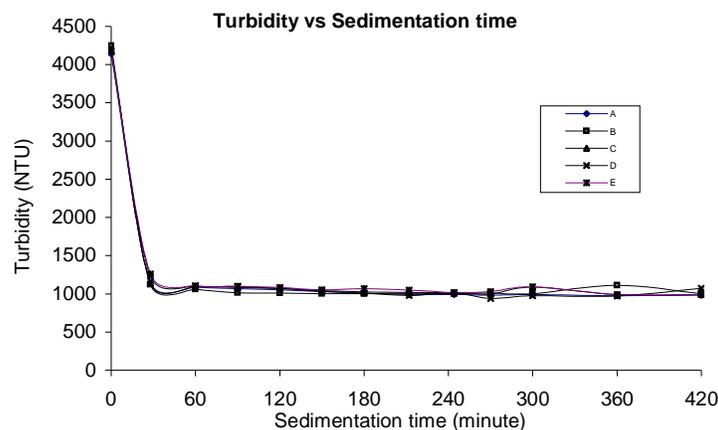


Fig. 5: The effect of sedimentation time on suspended solid removal from POME

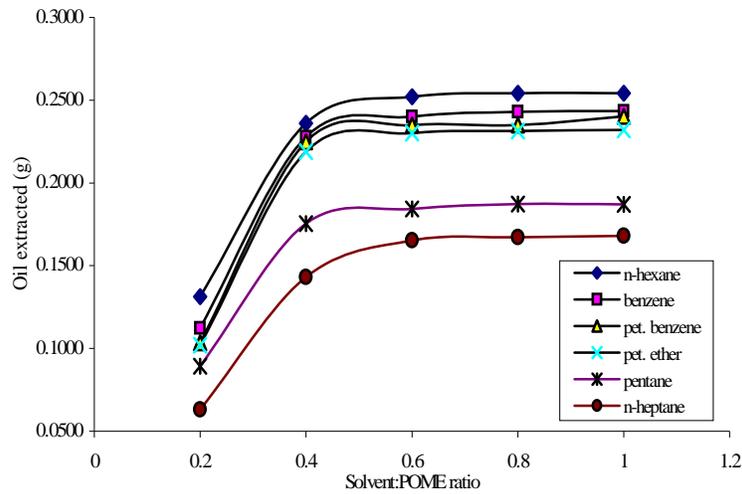


Fig. 6: The effect of solvent-POME ratio on extraction of residual oil from POME

b) Effect of mixing speed

As shown in Fig. 7, the rate of mass transfer increases with speed of mixing. Therefore increasing the mixing speed will increase the value of oil extracted from the POME. The maximum extraction occurs at the speed of 200 rpm for all the solvents. As the speed of mixing increases, the chances for the molecules to meet one another becomes more and this makes the rate of extraction increases. However, after mixing speed of 200 rpm, the oil extracted becomes constant. This is because almost all fatty acids have been extracted from

POME. Increasing the speed after this stage will not result any further extraction of oil.

c) Effect of mixing time

The oil extraction time of different solvents is shown in Fig. 8. Result on mixing time gives almost the same trend with the mixing speed. The oil extracted increased rapidly initially and reaching a constant value after 20 minutes. This is because, increasing the mixing time may not result further extraction of oil once equilibrium has been attained. Therefore, it is obviously seen from the figure that the equilibrium can be achieved after 20 minutes of running the experiment.

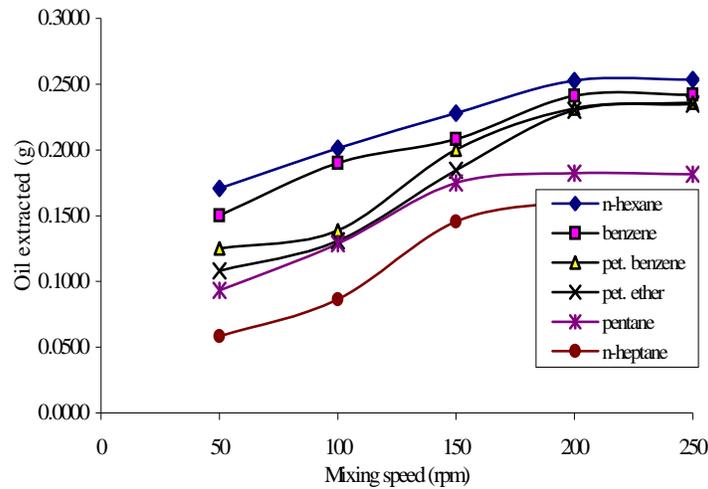


Fig. 7: The effect of mixing speed on extraction of residual oil from POME

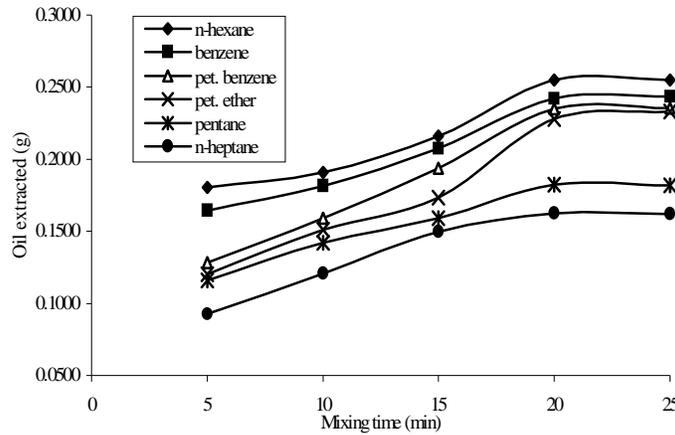


Fig. 8: The effect of mixing time on extraction of residual oil from POME

d) Effect of pH

For all the solvent used, the reading of oil extracted were low at $pH \leq 7$ but reach maximum at pH 9 as shown in Fig. 9. However it does not mean that the residual oil extraction is effective at pH 9 because, during studying the effect of pH for solvent extraction, NaOH is being added to POME to increase the pH, however this activity will lead to the saponification process which means, the residual oil react or undergo hydrolysis with sodium hydroxide to produce glycerol and a fatty acid salt called soap which is more soluble in

water rather than organic solvent. Therefore, while determining the oil concentration in treated sample using petroleum ether, the weight of oil found to be less at pH 9 since the oil was hydrolyzed inside the POME and not being extracted to the solvent. Thus utilizing the original pH of POME for residual oil extraction is preferred rather than adjusting the pH using strong acid or alkali to avoid hydrolysis reaction.

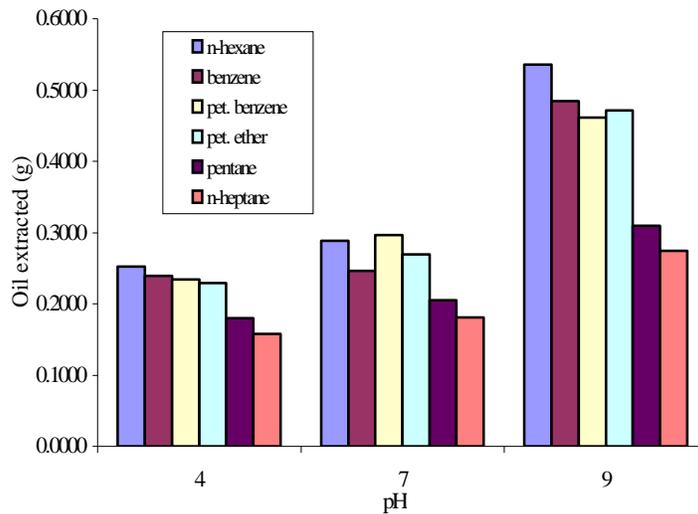


Fig. 9: The effect of pH on extraction of residual oil from POME

5. CONCLUSIONS

From the analysis result, it is clearly demonstrate that various parameters used in this experiment contribute their effect to the coagulation process. The optimum condition for suspended solids removal using a jar test process can be obtained at alum dosage of 4,000 mg/L at pH 4, mixing rate of 150 rpm and optimum mixing time of 1.5 hours. For sedimentation the optimum settling time is 100 minutes. At this optimum condition, the NTU reading was reduced from 14,080 NTU to 984 NTU whereby comparing to the calibration curve, the actual suspended solids content has been successfully reduced to 268 mg/L which is lower than the allowable limit by Malaysian Department of Environment (DOE) that is 400 mg/L.

In solvent extraction, the optimum value of the solvent to POME ratio is 6:10 and mixing speed of 200 rpm for 20 minutes at original pH of POME, pH 4, was obtained with the oil concentration reduced from 1089 mg/L to 54 mg/L.

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