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Residual oil and suspended solid removal using natural adsorbents chitosan, bentonite and activated carbon: A comparative study

A.L. Ahmad*, S. Sumathi, B.H. Hameed

School of Chemical Engineering, Engineering Campus, Universiti Sains Malaysia, 14300 Nibong Tebal,
Seberang Perai Selatan, Pulau Pinang, Malaysia

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Abstract

Palm oil mill effluent (POME), which contains about 4000 mg/l of residual oil, has been chosen to remove its residual oil using three types of adsorbents. Jar-test method has been used to identify the best adsorbent to remove the residual oil from POME. Chitosan was compared to activated carbon and bentonite as a potential residual oil remover. Chitosan showed the best removal compared to the other adsorbents for all the parameters studied. Chitosan could successfully remove 99% of residual oil and minimize the suspended solid content to a value of 25 mg/l from POME at a dosage of 0.5 g and employing a mixing time of 30 min, a mixing rate of 100 rpm, sedimentation for 30 min and a pH value of ranging from 4.0 to 5.0. For activated carbon and bentonite, the optimum dosages were 8.0 g and 10.0 g/l, respectively, 30 min of mixing time at 150 rpm, 80 and 60 min of settling time, respectively, and pH of 4.0–5.0 to obtain the same percentage of removal as performed by chitosan. Activated carbon and bentonite can only reduce the suspended solid values up to 35 and 70 mg/l, respectively, at the optimized conditions.

Keywords: Residual oil; Palm oil mill effluent; Chitosan; Bentonite; Activated carbon

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1. Introduction

Treatment and disposal of oily wastewater, such as palm oil mill effluent is presently one of the serious environmental problems contributors. Palm oil mill wastes have existed for years but their effects on environment are at present more noticeable. The oily waste has to be removed to prevent interfaces in water treatment units, avoid problems in the biological treatment stages, and comply with water-discharge requirements. The major difficulty in disposing the oily residuals are the emulsified oil droplets, which are sheltered from spontaneous coalescence into larger flocs, making oil separation by simple gravity a difficult and time consuming process [1]. Numerous methods have been used to remove residual oil from wastewater, such as adsorption, flocculation, electrocoagulation and flotation [2].

Palm oil mill effluent (POME) is a colloidal suspension, which contains 95–96% of water, 0.6–0.7% of oil and grease and 4–5% of total solids [3]. It is thick brownish in color liquid and discharged at temperature between 80 and 90 °C. It is fairly acidic with pH ranging from 4.0 to 5.0. Table 1 shows the refined characteristics of raw POME [3]. POME contains about 4000–6000 mg/l of oil and grease. The oil droplets of POME can be found in two phases. They are suspended in the supernatant as emulsions and also floating as oil droplets on the upper layer of the suspension. The residual oil droplets in POME was solvent extractable [4]. The maximum allowable limit set for oil and grease level is 50 mg/l.

Activated carbon, bentonite and chitosan are three types of natural adsorbents, which have been used in many applications, ranging from food and separation technology to wastewater treatment. Chitosan is a natural, modified carbohydrate biopolymer. It is a partially deacetylated derivate obtained by alkaline treatment of chitin [5]. Chitosan is recommended as a suitable resource material, because it has excellent properties, such as biodegradability, biocompability,

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* Corresponding author. Tel.: +60 4594 1012; fax: +60 4594 1013.
E-mail address: chlatif@eng.usm.my (A.L. Ahmad).

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Table 1
Characteristics of palm oil mill effluent (POME)

	Concentration (mg/l)
Parameter	
Oil and grease	4000–6000
Biochemical oxygen demand	25000
Chemical oxygen demand	50000
Total solids	40500
Suspended solids	18000
Total volatile solids	34000
Ammonicals nitrogen	35
Total nitrogen	750
Element	
Phosphorus	180
Potassium	2270
Calcium	439
Boron	7.6
Iron	46.5
Manganese	2.0
Copper	0.89
Magnesium	615
Zinc	2.3

adsorption property, flocculating ability, polyelectrolisity and its possibilities of regeneration in number of applications [5]. Bentonite is a type of peat consisting dominantly of smectite minerals. The important properties of bentonite include its ability to exchange cations, its swelling and hydration capacity, it could act as a binder, its permeability, viscosity and thixotropy [6]. Their sorption capabilities come from their high surface area and exchange capacities [7].

Activated carbon has been one of the most popular and widely used adsorbent in wastewater treatment applications throughout the world. Furthermore, activated carbon requires complexing agents to improve its removal performance for inorganic matters [7]. Despite of its prolific use, activated carbon is still assumed as an expensive material and not much work has been done in order to adsorb residual oil.

This paper is to present the removal performance of chitosan compared to commercial adsorbents, i.e., bentonite and activated carbon in removing the oily residual in POME and to investigate, which adsorbent projected the highest removal efficiency. In view of the fact that, no work has been done in the literature regarding the removal of residual oil from POME by natural adsorbents particularly with chitosan, bentonite and activated carbon. Even chitosans' performance compared to the commercial adsorbents, i.e., bentonite and activated carbon have not been explored. Furthermore, not many studies have been done with real effluent, whereby these studies were done using homemade synthetic effluent. Therefore, this research can be listed as a novel study. The optimum dosage and pH of adsorbent needed to achieve maximum removal of residual oil was determined. Best contact time, sedimentation time and mixing rate for each adsorbent were optimized. Suspended solid removal was also analyzed, because some of the residual oil in POME is suspended in the solids. Therefore, by removing the suspended solid from POME, we could indirectly remove the residual oil.

2. Material and methods

2.1. Experimental materials

Samples of POME were collected from local palm oil mill, at a temperature ranging from 80 to 90 °C. Samples may vary day to day depending on the discharge limit of the factory, climate and condition of the palm-oil processing. Before the samples were dispensed into the batch system, the samples were cooled to room temperature and let to sediment to reduce the total solid. Portions of this suspension were withdrawn and analyzed for their initial residual oil content, suspended solid and pH properties.

Chitosan was supplied by Hunza Pharmaceutical Sdn. Bhd. Malaysia in off-white fine powder with mesh size less than 120. Activated carbon and bentonite were supplied by Quicklab Sdn. Bhd with a mesh size less than 100.

Distillated water was used to dilute hydrochloric acid solution (Merck, Germany) and dissolve sodium hydroxide pellets (Merck, Germany) to obtain solutions of 5 M. These solutions were then used for pH adjustment during the treatment process; *n*-hexane (Merck, Germany) was used as the oil extraction in the oil and grease analysis.

2.2. Experimental procedure

POME was sedimentated for 1 h. After sedimentation, the collected supernatant was analyzed for its residual oil content. A conventional jar apparatus (Stuart Science Flocculator model (SW1)) was used to coagulate POME with the adsorbents. The apparatus could accommodate six beakers. The beakers were filled with 1 l of POME for each test run and stirred simultaneously at a defined speed with six-spindle of steel paddles. After adding the adsorbents into the suspension, the beakers were rapidly mixed at various mixing time and speed for different doses of adsorbents. The clarified and sedimentated supernatant was then analyzed for its residual oil content.

The samples were analyzed with different dosages of chitosan 0.08–0.8 g/l, activated carbon and bentonite 2–12 g/l. Parameters, such as effect of sedimentation time 5–80 min, effect of contact time 5–60 min and rate of mixing 20–200 rpm were also analyzed. pH adjustments from 3 to 6, were done to obtain the best pH condition to remove residual oil and suspended solid from POME. The reproducibility of the experimental data was analyzed by repeating each experimental runs for three times. The variance of the averaged data was within ±5%.

2.3. Residual-oil analysis

The residual-oil content was measured using the oil and grease method recommended by APHA Standard Method of Examination of Water and Wastewater [21], with *n*-hexane being used as the oil-extraction solvent. The oil and grease content in the suspension was determined for each sample of

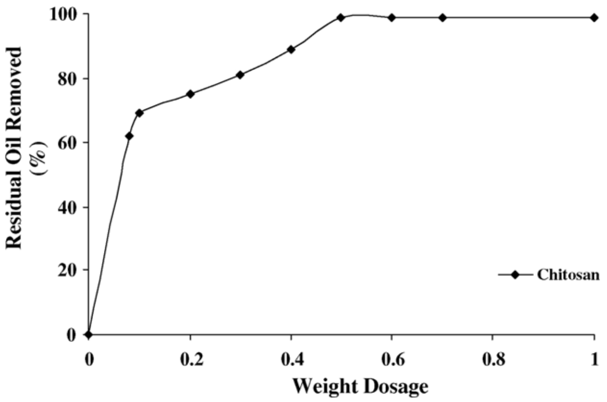

Fig. 1. Percentage of residual oil removed vs. dosage of chitosan.

POME both before and after experiment. Three replicates of each test run were undertaken with the mean value obtained for residual-oil content being calculated from the replicates.

3. Results and discussion

3.1. Effect of adsorbent dosage

Effect of adsorbent dosage of different adsorbents compared to chitosan was analyzed for mixing time of 60 min, a mixing rate of 100 rpm, sedimentation time of 60 min and with its original pH, i.e., 4.5. Figs. 1 and 2 show the removal percentage of residual oil from POME using chitosan, bentonite and activated carbon. From Fig. 1, it was observed that chitosan needed an adsorbent dosage of 0.5 g to achieve the highest percentage of removal, i.e., 99%; whereas in Fig. 2, activated carbon and bentonite needed 8 and 12 g, respectively, to achieve 99% removal. It can be concluded that chitosan was far better than the commercial adsorbents to remove the residual oil. The adsorbent dosages needed by the other commercial adsorbents were very much higher compared to chitosan.

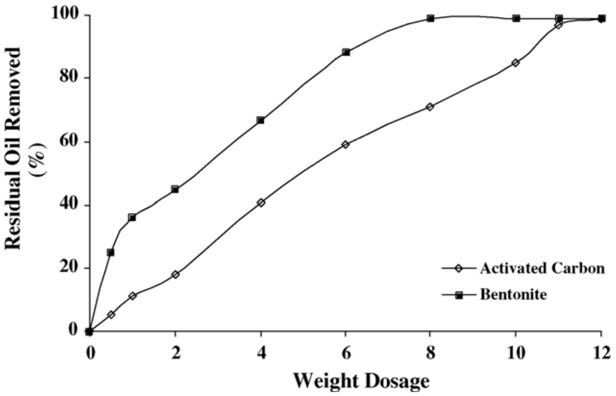

Fig. 2. Percentage of residual oil removed vs. dosage of activated carbon and bentonite.

Chitosan adsorbs the emulsified residual oil in the aqueous suspension of POME. Furthermore, chitosan adsorbs the residual oil in the suspended solid and this mechanism indirectly coagulates the suspended solid of POME. This proves why chitosan showed the highest percentage of residual-oil removal. Chitosan has amine functional groups, which are very attracted to anionic ions; therefore, it could easily bind and bridge [8] into flocs. The overall charge of chitosan is positive; whereas for residual oil is negative, therefore, attractions between the charges enhance the agglomeration process this mechanism is called as charge neutralization and has been study by Jill et al. [9]. Hence, chitosan not only acts as an adsorbent but at the same time as a coagulant to agglomerate the residual oil. Therefore, it can be concluded that chitosan not only adsorbs the residual oil in POME but successfully coagulates the suspended solid, which contains residual oil.

However, this is not the case for bentonite and activated carbon. This can be clearly proven by the results. It was found that, activated carbon needs about 12 g to remove 2 g/l of residual oil from POME. This fact shows that activated carbon is a poor choice of adsorbent and not economically to remove residual oil from POME compared to chitosan. Normally, activated carbon is used for separation technology, i.e., color and organic compounds removal in waste and water treatments [10,11], and so far, there is no research done on the removal of residual oil; so, from this research, it can be proved and concluded that activated carbon is a bad choice of adsorbent to remove residual oil.

Bentonite showed almost a similar trend of poor residual-oil adsorption compared to chitosan. A research on oil removal using attapulgite a type of clay material was done by Qiu et al. [12], but this process was aided with commercial coagulant (alum and polyacrylamide) to enhance the removal of oil. This finding proves that natural adsorbents are poor for residual-oil adsorption.

To further prove the removal of suspended solid because of residual-oil adsorption a study of suspended solid removal was also done. Determination of suspended solid concentration (in mg/l) gravimetrically gives a linear relationship with turbidity in NTU. Table 2 shows the performance of chitosan, activated carbon and bentonite as suspended solid remover. Most of the residual oil in POME was suspended in the suspended solid. Therefore, by removing the suspended solid, it could directly enhance the removal of residual oil. The

Table 2
List of adsorbent dosage needed to remove 99% residual oil and the minimum reading of suspended solid achieved

Adsorbent	Adsorbent dosage (g) needed to achieve 99% of residual-oil adsorption	Minimum reading of suspended solid (mg/l) after residual-oil removal at optimum adsorbent dosage
Chitosan	0.5	25
Bentonite	8	70
Activated carbon	12	33

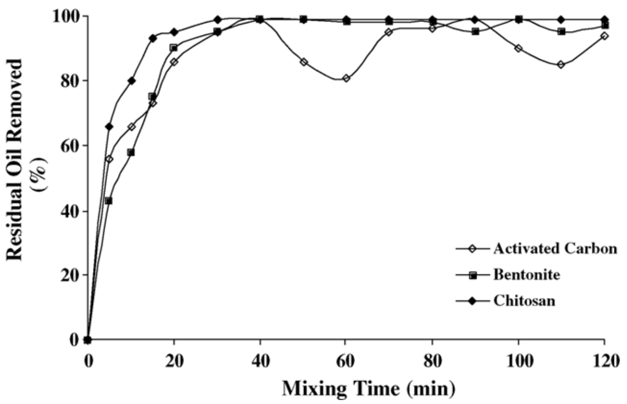

Fig. 3. Percentage of residual oil removed vs. mixing time for chitosan, activated carbon and bentonite.

initial value of suspended solid in POME before treatment was 10,000 mg/l and from the tabulated data, it shows that chitosan was the best in removing the suspended solid with a final value of 25 mg/l. This proves that chitosan was a good adsorbent as well as a coagulant compared to activated carbon and bentonite. Adsorbents adsorb compounds in wastewater, and they do not possess the function to coagulate. Therefore, the removal of suspended solid by commercial adsorbents was very poor compared to chitosan. Chitosan proves to be the best adsorbent with good removal of suspended solid compared to the other adsorbents, because it does two-in-one process, i.e., coagulation and adsorption.

3.2. *Effect of mixing time*

The effect of mixing time for 120 min on residual-oil adsorption and removal of suspended solid were analyzed using the optimized adsorbent dosage from the previous Section 3.1, mixing rate of 100 rpm, pH 4.5, and sedimentation time of 60 min. The results are demonstrated in Figs. 3 and 4. Chitosan needs 30 min to adsorb maximum amount of residual oil compared to other adsorbents; whereas for activated carbon and bentonite is 30 and 40 min, respectively. It is noticed that as the time was prolonged from 5 to 20 min, the removal was increasing. This is because the breakage of the oil droplets are enhanced thus reduces the diameter of the oil droplets (emulsification) causes more interfacial area for the adsorption to happen [13].

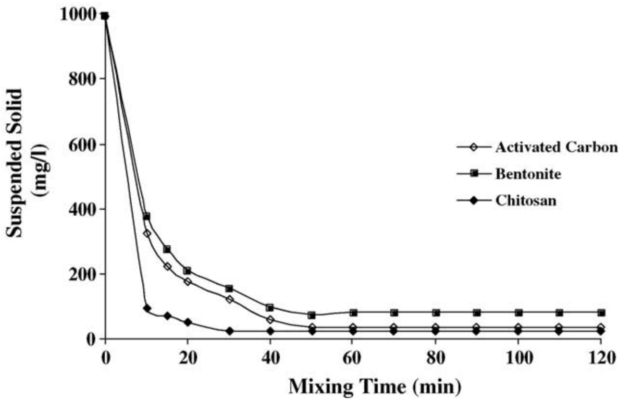

Fig. 4. Suspended solid removal vs. mixing time for chitosan, activated carbon and bentonite.

Fig. 3 shows that not much difference was observed in terms of removal percentage of residual oil when compared with other adsorbent. This is because the graph was plot using the optimum adsorbent dosages of each and every adsorbent, respectively. Normally, at optimum usage of adsorbent, the removal of residual oil will be the most favorable. However, the optimum weight dosage of chitosan required is relatively less compared to other adsorbents as discussed in Section 3.1.

In Fig. 3, it is observed that after 40–120 min of mixing, residual oil was introduced again into the suspension for activated carbon and bentonite. For activated carbon, it was very obvious but for bentonite it was lesser. This phenomenon is due to the high and intense agitation, which may cause the adsorbents to break-up and introduce the residual oil again into the system [14]. Chitosan was very intact even after 110 min of mixing.

This shows that only bentonite and chitosan shows a good adsorption of residual oil even after prolonged mixing time. The adsorption of residual oil by activated carbon is merely favorable on the surface of the adsorbent and the bonding between the residual oil and adsorbent was very weak. Furthermore, aggregation of activated carbon with residual oil happened after mixing of 60–120 min. This can be observed to have occurred. Therefore, the residual oil tends to break easily and introduced again into the suspension. For chitosan and bentonite, it was suggested that not just an adsorption on the surface but strong bonding of residual oil onto their surface as well diffusion of residual oil into their particles.

The suspended solid removal was displayed in Fig. 4. It can be seen that chitosan was the best-suspended solid remover compared to the commercial adsorbent. The lowest value of suspended solid achieved by chitosan is 25 mg/l; whereas by the activated carbon and bentonite, these are 35 and 70 mg/l, respectively. The removal of suspended solid or turbid using bentonite is very poor compared to chitosan and the minimum removal value was still high comparatively. Activated carbon easily dispersed in the suspension at prolonged mixing. The suspended solid attached to these adsorbents starts to break and disperse again into the suspension. This is due to the high-speed mixing, which indirectly enhances the breakage of flocs as well as attached suspended solids.

According to Demirci et al. [15], all types of bentonite does not give complete clarification due to the fact that while the pollutants are adsorbed on the surface and removed from the suspension, the adsorbent itself forms a colloidal suspension and causes somewhat turbidity or suspended particle. This was true and could be observed during the experimental lab work, whereby when the adsorbents were added into POME, the suspension become more colloidal.

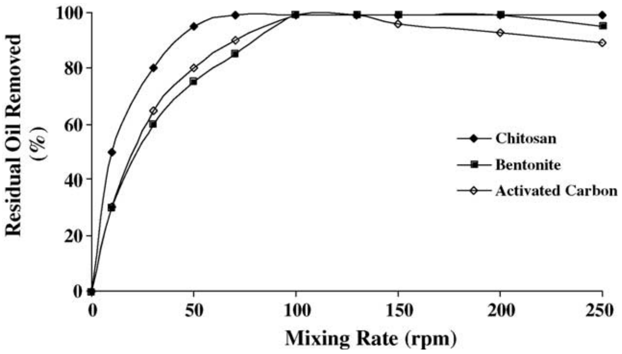

Fig. 5. Percentage of residual oil removed vs. mixing rate at 60 min of mixing time for chitosan, activated carbon and bentonite.

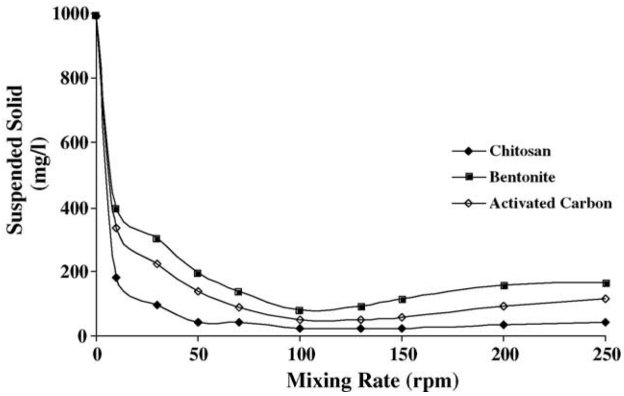

Fig. 7. Suspended solid removal vs. mixing rate of chitosan, activated carbon and bentonite.

3.3. Effect of mixing speed

The effect of mixing speed was analyzed using two different mixing times at various mixing speed for all the adsorbents with optimized adsorbent dosages, pH of 4.5 and sedimentation time of 60 min. Figs. 5 and 6 show the trend for effect of mixing speed. It was observed that mixing speed and mixing time were interrelated. At 70 rpm with 30 min of mixing, the percentage of residual-oil removal for chitosan was more than 80%, but at the same speed, if the mixing time was prolonged to 60 min, the percentage increased to 99%. The same type of trend could be observed for activated carbon and bentonite. Chitosan shows the fastest reaction compared to the other adsorbents. Whereby, it can yield higher percentage of removal at a slower mixing speed. This tendency can be clearly seen in Figs. 5 and 6. At 50 rpm for 60 min, the removal was 80% and for 30 min was 60%.

Activated carbon and bentonite shows almost the similar trend but yield was much less compared to chitosan, that is, 70 and 65% for 60 min, and 60 and 50% for 1/2 h, respectively. It was also observed that when the mixing speed was increased to more than 150 rpm, the percentage of residual oil decreased massively for activated carbon. This is because the adsorbed oil broke and dispersed again in the sample. For bentonite, it was only a little and can be neglected.

Fig. 7 shows suspended solid removal using different mixing speeds for 40 min of mixing time. Mixing speed at 20–100 rpm shows a dramatic reduction in suspended solid. However, the suspended solid value increased again, when the mixing speed was increased. This is due to the powder of the adsorbents. Restabilization of suspended solid will happen, when the mixing speed is very fast. This phenomenon can be clearly seen for readings of suspended solid for mixing speed more 150 rpm. The lowest suspended solid readings or the maximum removals of suspended solid that can be achieved by the adsorbents are: chitosan, 25 mg/l; bentonite, 70 mg/l; activated carbon, 35 mg/l. Chitosan shows the most stringent changes, which proves that chitosan is a good binder and coagulant.

3.4. Effect of sedimentation time

Sedimentation of adsorbents after residual-oil adsorption in terms of suspended solid is illustrated in Fig. 8 and was analyzed using the optimized adsorbent dosage at pH 4.5, mixing rate of 100 rpm and mixing time of 30 min.

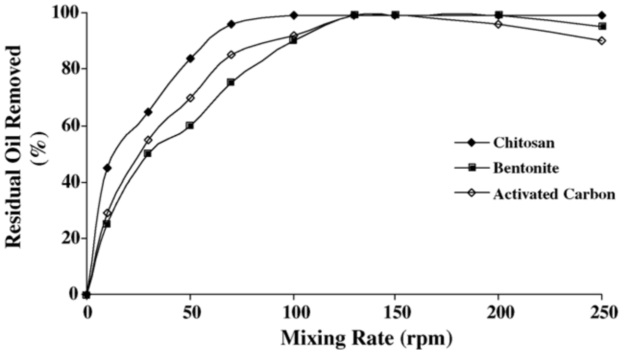

Fig. 6. Percentage of residual oil removed vs. mixing rate at 30 min of mixing time for chitosan, activated carbon and bentonite.

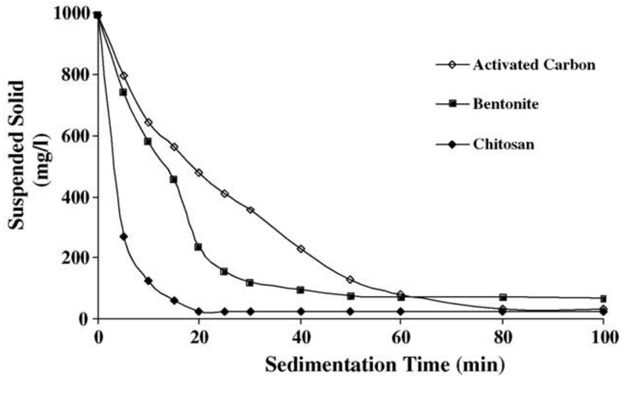

Fig. 8. Suspended solid removal vs. sedimentation time of chitosan, activated carbon and bentonite.

The sedimentation process of chitosan-adsorbed residual oil was the fastest compared to the other adsorbents. The adsorbed residual oil and suspended solid has been settled by 30 min with chitosan, whereas activated carbon and bentonite needed almost 80 and 60 min, respectively, to settle. Fig. 8 shows that the minimum value of suspended solid could be achieved by chitosan after coagulation at 30 min, i.e., 25 mg/l. Activated carbon and bentonite need longer time to settle, because they do not form flocs to settle easier. Almost all the residual oil adsorbed by the commercial adsorbents has been settled after 80 min of sedimentation.

3.5. Effect of pH

pH study was conducted at optimum adsorbent dosages, mixing time of 30 min, mixing rate of 100 rpm and sedimentation time of 30 min. The study on the effect of pH was essential to determine the optimum pH condition of the treatment system. It was imperative to determine the optimum pH, because pH not only affects the surface charge of the adsorbents but also the degree of ionization and adsorption of residual oil during reaction. In order to destabilize an emulsion in POME, it was necessary to provide adjustments that will affect the oil/grease–water interfacial film appropriately. Hence, emulsion breaking was usually brought about by changing the samples pH value or addition of inorganic coagulants [16]. Therefore, to examine the effect of pH on the residual-oil removal percentage, the pH of POME was varied from 2.0 to 7.0. The study of pH effect was concentrated from the acidic regent to neutral, because the pH of POME is acidic in the range of 4.0–5.0.

Fig. 9 shows the effect of pH on the removal of residual oil from POME suspension with the addition of the adsorbents. The plots in Fig. 9 are results obtained under optimum conditions, viz. optimum adsorbent dosage of chitosan powder (0.5 g/l), bentonite (8 g/l) and activated carbon (12 g/l), continuous mixing for 40 min with a mixing rate of 100 rpm, and the treated sample was sedimented for 60 min. Fig. 9 shows that, the removal of residual oil with chitosan was the best, i.e., 99% removal within the range of original pH of POME, i.e., pH 4.0–5.0.

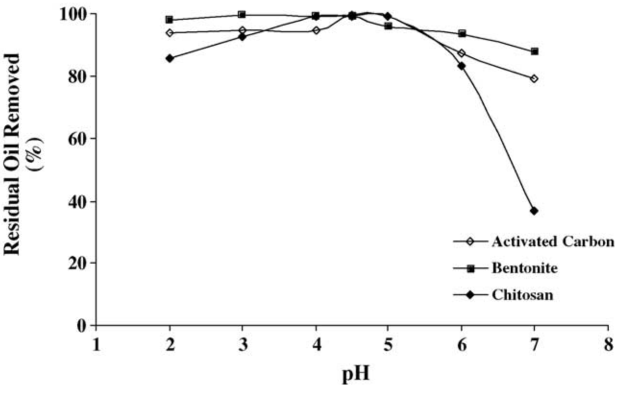

Fig. 9. Percentage of residual oil removed using chitosan, activated carbon and bentonite vs. different pH of POME.

Normally, the original pH value of POME was about pH 4.5 and from Fig. 9, it shows that at this pH value, the removal was very satisfying and achieved 99% of removal at this pH for all the adsorbents. This encouraging observation leads to a conclusion that pH adjustment of POME would be unnecessary under real-process treatment conditions for removing residual oil using chitosan, bentonite and activated carbon. This might be the best point, and at this acidic condition, the isoelectric point between residual oil and the adsorbents has been achieved and indirectly enhances the adsorption of residual oil in POME. At these pH values, the concentration of H^+ ion and the negative charge density are relatively very small compared to the situation under more acidic or higher alkaline conditions.

It was noticed that at strong acidic pH values (i.e., pH 3.0 and below) the adsorption capacity of bentonite and activated carbon was almost the same but for chitosan, it was lower. This is due to the increase of protons and this phenomenon allows the protonation of chitosan's amine groups to give ($-NH_3^+$ groups) [17]. This diminished the ability of the amine group of chitosan to attack the carbonyl group of residual oil and led to a reduction in the electrostatic attractions between the residual-oil molecules and adsorption site of chitosan. In acidic medium, all the free (NH_2^-) ions are neutralized by (H^+) ions to form (NH_3), which could not further adsorb the carbonyl ions of residual oil. Thus, protonation does not improve the ability of chitosan in adsorbing residual oil. According to Schmuhl et al. [18], chitosan is unstable at pH 2; hence, the removal of residual oil was deteriorating at this pH and this can be experimentally shown in Fig. 9. Furthermore in acidic condition, a strong competition existed between residual ions and protons for sorption sites, therefore, the sorption efficiency decreased.

The results also verified that adsorption of residual oil on bentonite and activated carbon was significant at strong acid regent. The pH of the solutions affects the surface charge of these adsorbents and their degree of ionization making it to be a better residual-oil adsorbent at this condition [19]. Susan et al. [19] proves that removal of anions with clay material like bentonite is more effective at low pH. Sandya and Tonni [7] explained that the electrostatic interactions of activated carbon in the acidic regent favor the adsorption capacity of any positively charged adsorbate. It was observed that at pH 6.0–7.0, the removal of residual oil was very poor for all the adsorbents. This is because at this pH condition, chitosan loses its cationic nature Guibal [20], whereas activated carbon and bentonite were very unstable. Particularly for bentonite, it was an extreme case, because bentonites' negative charges are very unstable at these alkaline conditions Sandya and Tonni [7]. Furthermore at this pH, the adsorption process itself is very unstable due to the characteristics of POME, which have changed drastically with the change of pH.

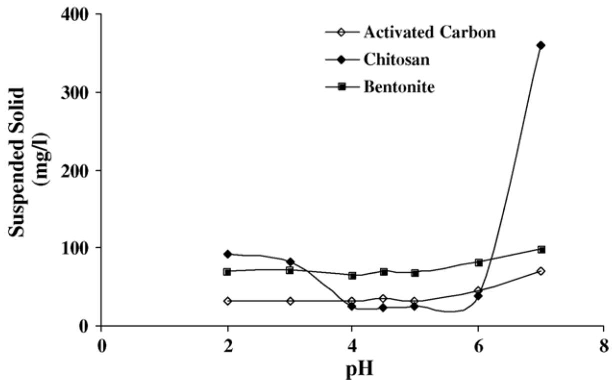

Fig. 10. Suspended solid removal using chitosan, activated carbon and bentonite vs. pH.

Fig. 10 shows the results of suspended solid removal using chitosan, bentonite and activated carbon. In Fig. 10, it appears that at pH values of 4.0, 4.5 and 5.0, the removal of suspended solid was the maximum, and these pH values contribute to the most favorable removal for all the adsorbents. Furthermore, the supernatant was visually very clear. The results indicate that the chitosan, activated carbon and bentonite underwent a transition in a narrow pH ranging near pH 4.5 for POME.

4. Conclusion

This investigation has looked into the removal of residual oil and suspended solid from palm oil mill effluent using adsorbent. Chitosan, activated carbon and bentonite were used to remove the residual oil, and a comparative study between the adsorbents was done. The destabilization of oil-in-water is successfully performed by the application of adsorbent, which shows a synergistic enhancement for the effective adsorption of residual oil. Chitosan shows the best performance as an adsorbent to adsorb residual oil from POME compared to the other adsorbents. The application of adsorbent in a coagulation treatment system resulted in higher than 99% reduction of residual-oil content in the supernatant and suspended solid of POME. The probable reasons for such behavior are discussed.

The following optimum parameters were found necessary for the reduction of the residual-oil content from an influent with a concentration of 2.0 to <10 mg/l and suspended solid content from 10,000 to 25 mg/l: chitosan as the best adsorbent with a dosage of 0.5 g; POME suspension pH value of 4.0–5.0; mixing rate, 100 rpm; mixing time, 30 min; sedimentation time, 30 min. In order to justify the usage of chitosan compared to bentonite and activated carbon in industrial scale other factors, such as the price difference, storage and recycling potential have to be thoroughly investigated.

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