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Adsorption of Sodium, Magnesium, Calcium and Zinc from Recovered Base Oil of Used Lubricants Using Chitosan

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ABSTRACT

In this study, the adsorption processes using chitosan as adsorbent to refine base oil from used lubricants was explored. The recovered base oil characterizations showed that the content of metals such as sodium, magnesium, calcium and zinc are significant for further refining. The effects of operating parameters on these metals removal were then investigated. Two level factorial designs were conducted to examine the effect of chitosan dosage, temperature, contact time and chitosan grain size. The results of factorial design indicated that the most influential parameters affecting the metals removal were chitosan dosage and temperature. Therefore, further studies were focused on the determination of the effect of chitosan dosage and temperature on sodium, magnesium, calcium and zinc removals from the recovered base oil. It was found that the increase of chitosan dosage from 0.5 to 1.0 g decrease the metals removal percentage. Furthermore, the increase of temperature from 30 to 70°C and the increase of contact time from 2 to 10 min resulted in the decrease of metals removal.

Key words: Adsorption, recovered base oil, used lubricants, chitosan, factorial design

INTRODUCTION

It has been reported that about 13 billion gallons of used motor and other oils are generated annually (Ciora and Liu, 2000). According to Rincon *et al.* (2007), these waste oils are considered as hazardous waste because of their high content of pollutants (contaminated with oxidation and degradation products, water, fine particulates, metals and carbon oil additive products). Used lubricating oil generally contains approximately 0.5 wt% of ash residue after combustion (Fletcher and O'Blasny, 1981) and for reuse as fuel, this ash creates air pollution concerns. Nevertheless, the used oil still contains a large proportion of valuable base oil that can be used to formulate new lubricant, such as crank case fluid if the contaminants are properly removed, odour is eliminated and the colour is improved. Thus, the recovery and purification of this base oil not only give the economic contributions but also good for environment.

In recent years, there are several processes available for the recycling of used lubricating oil. The traditional method used was chemical cracking using acid treatment of the used oil to flocculate carbonaceous particles and other impurities from the used oil. However, Brownawell and Renard (1972) reported that this process generates acid sludge, creating environmental concerns and disposal problems. Other popular conventional method used was acid clay treatment (Hess, 1979), where base oil was separated from used lubricant using sulphuric acid as a separate medium. At the end of the process, the acidic sludge was disposed of while the base oil was treated with clay for colour and odour removal. Other alternative, distillation/clay treatment of the used lubricant oil has been suggested to separate ash and other contaminants from the oil (Hess, 1979). He reported that the high boiling point of the used oil at atmospheric pressure with the assistance of vacuum and high operating temperature was effectively evaporate oil and leave the contaminants and impurities as residue. Although, this process to be technically feasible, it is energy intense due to the phase change involve in evaporation. In addition, to deliver an acceptable quality of colour and smell, a polishing step is required.

Nowadays, solvent extraction technique is one of the cheapest and most efficient processes experienced in recycling used lubricating oils (Elbashir *et al*., 2002). This method was very attractive and effective alternative that can be commercialized for re-refining the base oil. Solvent extraction is a simple process where the used oil and solvent are mixed in appropriate proportions to assure complete miscibility of the base oil in the solvent. The extracting solvent should also reject the additives and carbonaceous impurities normally found in used oils. These impurities flocculate and settle under gravity action.

Solvent extraction system should have the capability of separating the maximum amount of sludge particles from used oil and at the same time losing the minimum amount of base oil in the sludge phase. At the end of this process, organic sludge with huge potential as burning fuels is produced. However, the successful of the re-refining process will be achieved if the recovered base oil meets the acceptance contents of contaminant. Thus, a need has arisen for an effective method of purifying the recovered base oil in order to achieve the desired product quality.

In this study, adsorption process using chitosan as adsorbent was explored to overcome such problems. Chitosan is a natural product derives from chitin after deacetylation process. This polysaccharide found in the outer skeleton of crustacean shells, shrimps, prawn and crabs and also obtained from fungal sources. From the studies carried by Guibal *et al*. (1994) and Yang and Zall (1984), the amine groups on the chitosan chain can serve as chelation sites for metals, thus chitosan become an effective adsorbent in the uptake of metals. The properties of chitosan products such as deacetylation, molecular weight, viscosity and solubility are the important characteristics of chitosan. Most of the studies were carried out as a function of parameter condition and selected metals adsorption such as plumbum (Ngah *et al*., 2002a, b), chromium (Ngah *et al*., 2002a, b) and cadmium (Cervera *et al*., 2003), copper (Da Silva and da Silva, 2004), zinc and mercury (Benavente, 2008) by using chitosan.

The aim of this study was to investigate the effects of operating parameters on metals removal from the recovered base oil using adsorption process. This study is an extension from an earlier study by Jamil *et al*. (2010).

MATERIALS AND METHODS

Experimental material: Used lubricating oil was collected from a garage and mixed in a single container. It was stored in a close drum and the contents were homogenised prior to any testing. Used lubricant evaluated according to standard test of American Society for Testing and Materials (ASTM). ASTM D6595 was used to complete this metals content analysis. Chitosan was soluble in 1% diluted acetic acid with a 90% degree of deacetylation. N-hexane and 2-propanol was selected as the composite solvent. The 2-propanol was selected as the polar component due to its high solubility while n-hexane was selected as the non-polar component due to its economical cost and availability.

Based oil recovery procedure: Base oil was recovered from used lubricant by solvent extraction method using composite solvent. The increase of 2-propanol composition in the composite solvent of 2-propanol and n-hexane tends to force the polymer into rather extensive configuration which encourages more particles to be absorbed on its surface and form larger flocs (Gary, 1999). He found the ratio of 60% 2-propanol and 40% n-hexane to be the optimum ratio. Lim (2001) observed that the optimum ratio of that composite solvent to oil as 4:1. Foo *et al*. (2002) suggested that with the addition of 1.5 g potassium hydroxide (KOH) into the composite solvent, the sludge sedimentation rate will increase. In order to achieve the best conditions to extract the recovered base oil, all the suggestions from above mentioned research were followed.

The mixture was then subjected to intense agitation for 15 min at 300 rpm to allow good mixing between the oil and the solvent. After strong agitation, it is then left under room temperature for 24 h to allow extraction-flocculation. Consequently, the oil was introduced into simple filtration process to allow separation base oil from the oil sludge. The recovered base oil with darkish colour was collected and kept in a close drum of 20 L. In order to separate the solvent from the mixture, the content in the drum have to be homogenized before being it undergoes the evaporation step.

Solvent extraction process: After the mixture of the extracted recovered base oil and solvents were separated from the sludge, rotary evaporator was used to separate the solvents from the recovered base oil. The mixtures were placed inside the flask. A heating bath which equipped with temperature controller was used to provide heat for boiling process. A chiller which was used to provide cooling water for condenser. When the mixtures were boiled at the boiling point of the hexane and 2-propanol, the solvents were vaporized and condensed to form liquid droplets that were collected through a collection flask.

Metal removal by adsorption process: Adsorption experimental set-up was used to study the effect of selected parameters to the adsorption process, using the stirrer to allow good mixture between oil and chitosan and water bath equipped with temperature controller to provide heat for the temperature studied. The centrifugation system was used to centrifuge the sample. The samples were poured into the glass centrifuge tubes and introduced to the centrifuge support in order to segregate the mixture.

Parameter estimation: In the experiment, the effect of process parameters such as chitosan dosage, adsorption temperature and contact time to the adsorption processes were investigated. The recovered base oil was mixed with chitosan in a beaker. Different amounts (0.5, 0.75 and 1.0 g) of accurately weighted sieved chitosan was brought in contact with 50 mL of recovered base oil under continuous stirring (speed of agitation 200 rpm), at temperature 30, 50 and 70°C with various contact time (2-10 min). The temperature range was chosen according to a previous study where they considered that chitosan degrade above 100°C. The chitosan grain size was fixed at 0.5 mm mesh size. Afterwards, the samples were centrifuge for 10 min with 500 rpm speed. After the centrifugation step, the sample was decanting into a beaker. The reproducibility of the experimental data was analyzed by repeating each experimental runs for two times. Then, the samples were placed in a bottle for further determination of their metals content.

Data calculation: The percentage of metals removal was calculated using the following equation:

$$\text{Metal removal (\%)} = \frac{c_o - c_i}{c_o} \times 100$$

where, c_o and c_i are the concentration of metal (ppm) before adsorption and after adsorption, respectively. Assumptions for this equation are as follows:

- Negligible volume changes
- Intraparticle transport mechanism predominantly surface diffusion

Parameter identification: The factorial design analysis was performed by a statistical method called "full factorial" (2k) designs. Four parameters were chosen to be investigated, namely temperature, contact time, chitosan grain sizes and chitosan dosage. Table 1 shows the parameters involved in the studies. The responses variables used were the percentage of metals removal. The metals involved were sodium (Na), magnesium (Mg), calcium (Ca) and zinc (Zn). For a two-level factorial design with four variables, sixteen runs (24 = 16) of experiment were conducted.

Table 1: Actual and coded values of parameters in 2^4 full factorial design for metals removal by adsorption

Level of variables	Low	High
Temperature (°C)		
Actual (x_1)	30	70
Coded (X_1)	-	+
Time (min)		
Actual (x_2)	10	60
Coded (X_2)	-	+
Grain size (mm)		
Actual (x_3)	0.25	1.0
Coded (X_3)	-	+
Chitosan dosage (g)		
Actual (x_4)	0.5	2.5
Coded (X_4)	-	+

RESULTS AND DISCUSSION

Recovered base oil characterizations: Adsorption efficiency of metals from recovered base oil is partly influenced by the conditions of the collected samples. Therefore, the characterization of its physical and chemical properties is important prior to experiment. The properties of waste oil sample and the recovered base oil that was prepared by solvent extraction method were shown in Table 2. As can be seen, the waste oil samples recorded higher density value than the new base oil, hence reflects the presence of contaminants such as fuel root or oxidised material in the waste oil. Moreover, the waste oil shows the increase in kinematic viscosity which indicates the presence of contamination in used lubricant oil. The waste oil also shows the increase of the oil viscosity that cause by the increase of oxidation and polymerization products that were dissolved and suspended in the oil (Awaja and Pavel, 2006). On the other hand, the waste oil only contents with a little value of water. So, it does not require water removal procedures. Other than that, the carbon residue and ash content of used lubricant oil were higher than the specification of the new base oil which evaluates the remaining solid in the waste oil.

The recovered base oil characterizations shown in Table 2 was produced after the waste oil samples have been treated by solvent extraction method for the sample preparation step. As can be seen, the proportions of metals content in the recovered base oil are still high. These include the content of sodium, magnesium, calcium and zinc which shows the value greater than 10 ppm. As stated by Hess (1979), in order to recycle the recovered base oil, the metals content must be in a very small quantity (below than 10 ppm) so that it is easier to blend with the other additives to produce new lubricating oil.

Parameter identification: The full factorial design analysis was carried out to identify the main and the interaction effects

Table 2: Properties of waste oil and recovered base oil

Properties	Waste oil	Recovered base oil (after extraction)
Physical properties		
Density at 15°C (kg L^{-1})	0.8955	0.8814
Kinematic viscosity at 25°C (cSt)	465.3	54.47
Water content (vol%)	0.10	0
Carbon residue (MCRT) (wt%)	1.45	0.340
Flash point (COC) (°C)	264	268
Ash content (wt%)	0.77	0.25
Chemical content (ppm)		
Sodium	58	22
Magnesium	72	14
Calcium	1008	227
Iron	28	5
Copper	3	3
Tin	<1	0
Zinc	380	157.7
Lead	11	1

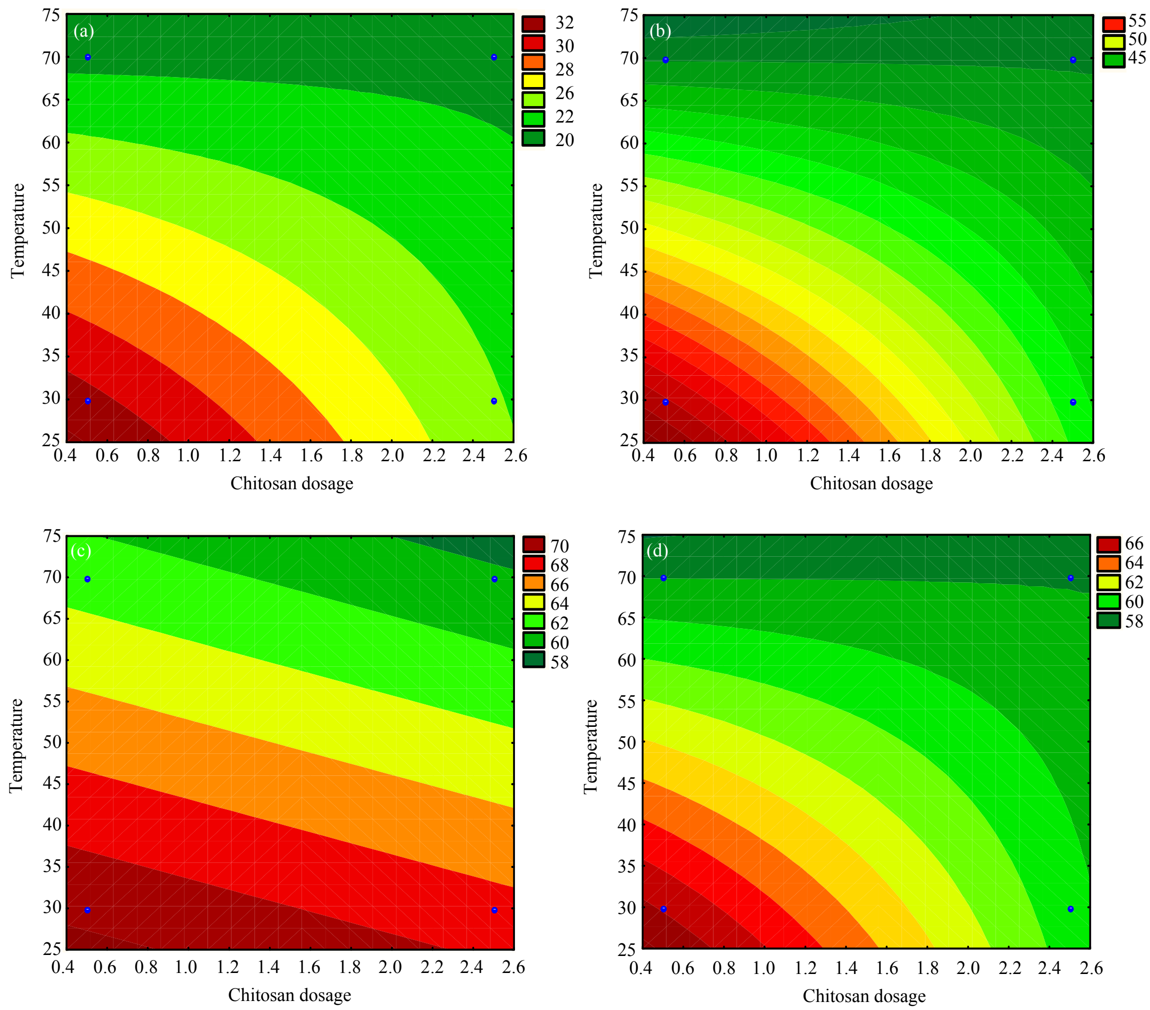

Fig. 1(a-d): Contour plot for, (a) Na removal, (b) Mg removal, (c) Ca removal and (d) Zn removal

of the experimental parameters on the four dependent variables, percentage of sodium, magnesium, calcium and zinc removal. A design matrix with the responses Y_1 (percentage of Na removal), Y_2 (percentage of Mg removal), Y_3 (percentage of Ca removal) and Y_4 (percentage of Zn removal) are illustrated in Table 3 with the coded units and results for percentage of metals removal analysis.

From Fig. 1a, as the temperature and chitosan dosage increase, the Na removal will decrease. As illustrated, the best range for temperature was from 25-40°C with chitosan dosage ranging from 0.5 to 1.5 g.

Besides that, as the chitosan grain size increase, the Na removal will decrease and the best range was from 0.25-0.5 mm. The contact time from 10-60 min seems like does not affect the metals removal.

Figure 1b depicts the contour plots for Mg removal. As can be seen, as the temperature and chitosan dosage increase, the Mg removal will decrease. The best temperature range was

Table 3: Factorial design result

Run	X_1	X_2	X_3	X_4	Y_1	Y_2	Y_3	Y_4
1	-	-	-	-	22.22	57.14	66.67	61.18
2	+	-	-	-	22.22	42.86	77.78	60.00
3	-	+	-	-	44.44	42.86	77.78	57.65
4	+	+	-	-	33.33	42.86	66.67	55.29
5	-	-	+	-	66.67	71.43	88.89	70.59
6	+	-	+	-	44.44	57.14	77.78	60.00
7	-	+	+	-	0.00	42.86	44.44	58.82
8	+	+	+	-	11.11	57.14	66.67	71.76
9	-	-	-	+	22.22	42.86	55.56	65.88
10	+	-	-	+	11.11	42.86	44.44	63.53
11	-	+	-	+	11.11	42.86	55.56	51.76
12	+	+	-	+	22.22	28.57	44.44	54.12
13	-	-	+	+	11.11	57.14	66.67	65.88
14	+	-	+	+	11.11	42.86	66.67	54.12
15	-	+	+	+	22.22	42.86	66.67	63.53
16	+	+	+	+	11.11	42.86	55.56	50.59

ranging between 30-45°C and chitosan dosage between 0.5-1.5 g for Mg removal. The increase of chitosan grain size does not affect the Mg removal. For the contact time, the best range was between 30-60 min.

The contour plots for Ca removal were present in Fig. 1c. From the plot, the increase of temperature and chitosan dosage will decrease the Ca removal. The best range for temperature was from 30-45°C with chitosan dosage from 0.5-2.2 g. While as the chitosan grain size and contact time increase, the Ca removal will decrease. The best range for chitosan grain size was ranging from 0.2-0.5 mm with contact time from 10-60 min.

Figure 1d gives the contour plots for Zn removal. As can be seen, the increase of temperature and chitosan dosage will decrease the Zn removal. The best temperature range was from 30-45°C with chitosan dosage ranging from 0.5-1.5 g. Besides that, the increase of chitosan grain size will decrease the Zn removal. The best range for contact time was ranging from 50-60 min.

Parameters screening: From the factorial design analysis, since the increase of chitosan dosage and temperature will decrease the metals removal, it was decided to choose chitosan dosage ranging from 0.5-1.0 g and temperature ranging from 30-70°C as the range of parameters to be studied. On the other hand, because of the difficulties to find the best range for contact time and the best chitosan grain size to be used, another set of simple screening experiments has been conducted. Figure 2 presented the results from the contact time parameter screening.

The screening effect of contact time was conducted at T = 300°C with 0.5 g chitosan dosage using 0.50 mm chitosan grain size. From Fig. 2a, it can be seen that from 10-20 min contact time, the metals removal start to decrease and it remains constant until 60 min and the equilibrium

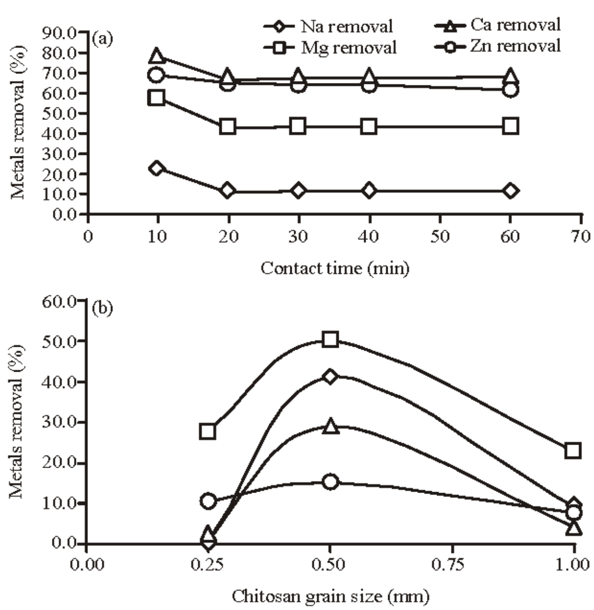

Fig. 2(a-b): Effect of (a) Contact time and (b) Chitosan grain size to the metals removal

does not improve the metals removal by chitosan. Because of this situation, the contact time range between 2-10 min was selected for the subsequent experiments.

In other set of parameter screening experiment, effect of chitosan grain size ranging from 0.25-1.00 mm on metals removal were investigated at T = 30°C with 0.5 g chitosan in 10 min contact time. The results are plotted in Fig. 2b.

As can be seen, the metals removal increases with the increases of chitosan grain size from 0.25-0.5 mm. However, as the chitosan grain size increase from 0.5-1.0 mm, the metals removal percentages were decreased. This trend showed that 0.5 mm chitosan grain size was the most preferable size to be used in the subsequent experiment. Karthikeyan *et al.* (2004) stated that the maximum uptake by the smallest grain size is a function of the surface area of the chitosan where the adsorption site per unit mass increase with the decreasing grain size. In a smaller particle size, chitosan are available to bind metals because of the opening adsorption sites on the chitosan chain.

Results from factorial design showed that the most influential parameters affecting the metals removal were chitosan dosage and temperature while the least important parameters were contact time and chitosan grain size. After considering the result of parameters screening, the chitosan dosage, temperature and contact time were selected as the main parameters while chitosan grain size was omitted in the subsequent experiment.

Effects of operating parameter on metal removal: In this experiment, the determination of metals removal performance was conducted at the selected range of chitosan dosage, temperature and contact time with fixed chitosan grain size. The effects of selected operating parameters are discussed below.

Effect of chitosan dosage: Figure 3 shows the effects of chitosan dosage as a function of contact time to the metals removal from recovered base oil. This effect was examined by conducting adsorption experiments using fixed chitosan grain size, 0.50 mm at 30°C for three series of chitosan dosage at 0.5, 0.75 and 1.00 g As can be seen from Fig. 3, the percentage of sodium, magnesium, calcium and zinc removals decrease with the increase of adsorbent dosage. This observation could be explained by the theory that in the adsorption process, the increasing of the adsorbent mass in the solution result in the increase of the amount of active sites on the surface of the adsorbent, giving greater chance of the metals ion to be adsorbed. However, when the adsorbent mass used in the solution is too large, the metals removal decrease; probably due to the problem of mass transfer or mobility of the metals ion into the surface of the adsorbent. A similar phenomenon was observed for the adsorption of Zn onto chitosan from aqueous solution. For the adsorption process at this condition, the percentage of metals removal reaches the highest value when 0.5 g chitosan was used, resulting in 55% Na removal at 2 min, 61% Mg removal at 4 min, 51% Ca removal at 8 min

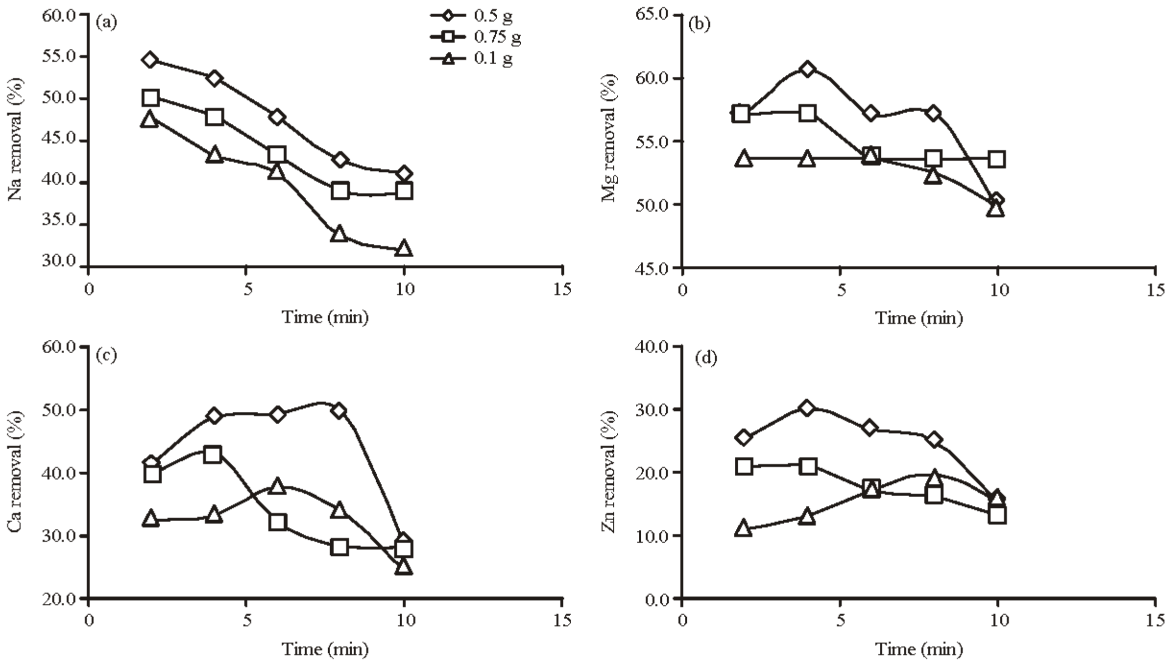

Fig. 3(a-d): Effect of chitosan dosage on metals removal (a) Sodium, (b) Magnesium, (c) Calcium and (d) Zinc (T = 30°C, grain size = 0.50 mm)

and 30% Zn removal at 4 min. In view of this, 0.5 g of chitosan was fixed as the dose along with 0.5 mm grain size for further experiments.

Effect of adsorption temperature: The results for the effect of adsorption temperature are plotted in Fig. 4. The experiment was carried out at 30, 50 and 70°C using 0.5 g chitosan with 0.5 mm grain size. It is observed that the increase of temperature from 30-70°C did not enhance the removal significantly. From the plotted result, at 70°C only minimum metals removal was achieved. In theory, the adsorption temperature has two major effects on adsorption. According to Bernardin (1995), at higher temperature the rate of adsorption is usually increased due to the increased rate of diffusion of adsorbate molecule through the solution to the adsorbent. Moreover, since the solubility and adsorption are inversely related, as temperature affects the extent of adsorption. As mentioned before, increasing the solubility results the smaller extent of adsorption. In a previous study the effect of temperature on copper removal from diesel oil was investigated. They found that as the temperature increase from 25-75°C, copper removals also increase but further increase of temperature to 100°C, chitosan started to degrade. Jideowno *et al.* (2007) reported the same phenomena in the investigation on the effect of temperature to the adsorption of lead, cadmium and nickel using chitosan. They emphasized that at the higher temperature, the kinetic energy of the system will rise up and the substrate-metal ion complex molecules will energized. This phenomena result to the instability of the molecules. All these might be responsible for the reduction of metals removal. Since at 30°C the metals removal shows the highest value, the rest of experiment were run at this temperature.

Effect of contact time: Figure 5 illustrates the effect of contact time from 2-10 min on metals removal that were conducted at T = 30°C with 0.5 g chitosan and 0.5 mm chitosan grain size. As can be seen, sodium removal starts to decrease after 2 min. Meanwhile, magnesium and zinc removal increase until 4 min and start to decrease after 4 min and calcium removal increases until 8 min and decrease after that. This initial stage of the magnesium, zinc and calcium curves may be related to the transfer of metal ions from the bulk of solution to the boundary film of the adsorbent and later to its surface. The decreases of the sodium removal after 2 min, magnesium and zinc removals after 4 min and calcium removal after 8 min may be due to desorption process where the metals adsorbed on the chitosan surface start to release into the recovered base oil. According to adsorption isotherm, removal of heavy metals should increase for longer contact time with the adsorbent. However, the results obtained have contradicted with the theory. According to Bernardin (1995), to better estimate the effect of contact time on a particular system, a single adsorbent dosage should be contacted with a sample of solution simultaneously with isotherm condition. The measurement of the concentration change over time in this system will show the effect of time on adsorption process. The

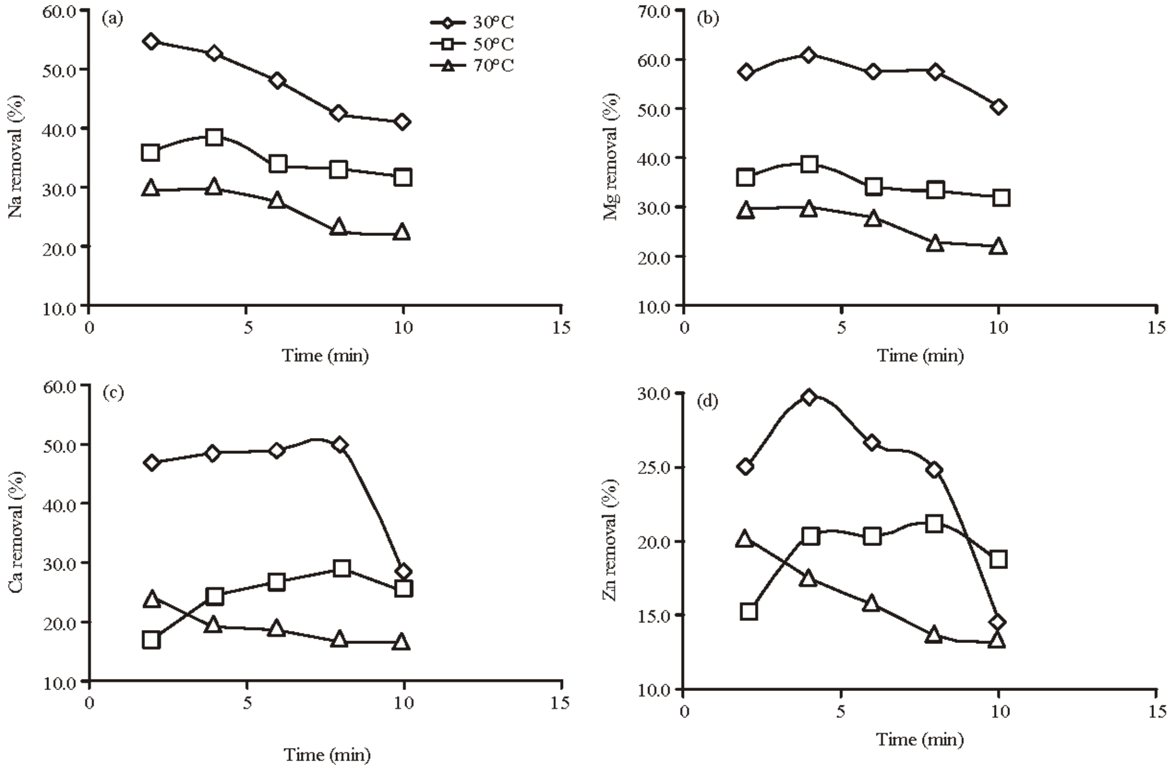

Fig. 4(a-d): Effect of temperature on metals removal (a) Sodium, (b) Magnesium, (c) Calcium and (d) Zinc (m = 0.5 g, grain size = 0.50 mm)

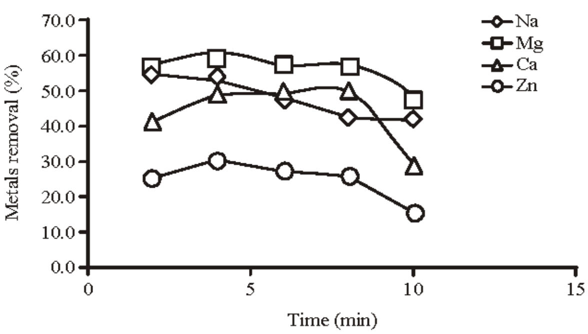

Fig. 5: Effect of contact time (2-10 min) on metals removal

difference between expected and obtained results may be due to weaknesses in experimental design.

It was observed that the increase of chitosan dosage from 0.5-1.0 g decreased the metals removal percentage. While for the temperature and contact time effect, the increase of temperature from 3-70°C and the increase of contact time from 2-10 min also results metals removal percentage.

CONCLUSION

This research was motivated by problems related to used oil treatments by traditional and distillation/clay method, such as generating acidic sludge, fouling of heating and distillation equipment as well as smell and quality of the recovered base oil. Although significant amount of contaminants removal was achieved with the solvent extraction, the recovered base oil is still in darkish color with stink odor and only minimum heavy metals removal was achieved. In this study, adsorption processes using chitosan as adsorbent was explored to overcome the problem.

Factorial design analysis indicated that chitosan dosage and temperature were the most influential parameters affecting metals removal from the recovered base oil. The least parameters affecting the metals removal were contact time and chitosan grain size. Besides that, the metals removal process depended on the interaction effect of temperature and chitosan dosage. Results from factorial design were used as the basis for the subsequent experiments. The characterization of recovered base oil reported that sodium, magnesium, calcium and zinc were the metals that needed to be removed from the recovered base oil. The target of the metals removal study is to reduce until the metals content below that 10 ppm.

In the subsequent experiment, the results reported that the increases of chitosan dosage from 0.5 to 1.0 g decreased the metals removal percentage. While for the temperature and contact time effect, the increase of temperature from 30 to 70°C and the increase of contact time from 2 to 10 min resulted in the decrease of metals removal.

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