TREATMENT OF WASTE ENGINE OIL USING OPTIMIZED ACID/CLAY REFINING METHOD

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ABSTRACT: This paper addresses the treatment of waste engine oils (WEO) by acid/clay refining method using glacial acetic acid. An optimization of the process parameters in terms of settling time, stirring speed and mixing temperature for treating the WEO was performed using Response Surface Methodology to improve the quality of treated lubricating oil. The quality of the treated WEO (Castrol brand) was evaluated in terms of viscosity index and flash point value. The treated fuel quality is found to have about 95% similarity to the fresh oil used as a standard at the settling time of 24 hours, temperature of 50 °C and mixing speed of 150 rpm. Analysis of variances (ANOVA) showed that settling time plays the most significant parameters of the process followed by the mixing temperature. Solid contaminants which were collected after the treatment were analyzed using SEM-EDS. They contained rough heteregenous shaped particles with elements such as carbon (97%), calcium (1.12%), zinc (0.74%), sulphur (0.73%) and phosphorus (0.29%). Then, four different brands of WEO (Liqui Moly, Castrol, Shell and Pennzoil) were treated at the optimized conditions to determine the feasibility of the method to treat any brands of WEO. It can be concluded that the optimized treatement method is suitable to treat most of WEO. The findings of this study provide the information on the best process condition for treating WEO as well as the solid contaminants present in it.

KEY WORDS: *Waste engine oil, Glacial acetic acid, Acid/clay refining method, Viscosity and Flash Point*

1. INTRODUCTION

Waste engine oil (WEO) is considered as chronic source of pollution worldwide which are mainly contributed by automotive traffic and industrial activities. Apart from hydrocarbons, WEO may contain heavy metals, solid contaminants and lubricant additives which regard WEO as a hazardous waste and may harm the environment. Due to high demand of engine oil especially from vehicle users, huge amount of WEO will be generated. These oils must be changed and expelled from the vehicle after a couple of thousand kilometers of driving i.e. every 3000 to 3500 km. Globally, it is reported that only 45% of WEO is being collected and the remaining 55% is thrown away to the environment [1]. Thus, WEO must be properly managed or treated to avoid damage to the soil, aquatic environments and atmosphere. Treated WEO can be reused for various applications such as industrial heater, concrete plasticizer and production of diesel-like fuel [2].

The effort to treat WEO will benefit the country environmentally and economically. Nowadays, many researchers have conducted research on how to recycle or treat the WEO. The treatment of WEO includes clay treatment, vacuum distillation, membrane technology and acid/clay refining methods. The conventional methods of treatment of WEO either require a high cost technology such as vacuum distillation or the use of toxic materials such as sulfuric acid [3-6]. Among these methods, the treatment of WEO using acid/clay refining process offers an affordable cost at moderate treatment process compared to other conventional methods. The treatment by glacial acetic acid provides mild condition and reacts lightly with base oils which are the added advantages compared to other acids (i.e. sulphuric acid).

So far acid/clay refining treatment using glacial acetic acid has only been conducted at constant parameters and no investigation on the solid contaminants that were extracted after the treatment has been done. Often the solid contaminants collected after the treatment is directly discarded with little information are known on the real composition of WEO. In addition, current method with acid/clay refining method is less efficient and has low yield of oil recovery [7]. Thus, in this study the treatment parameters such as settling time, stirring speed and temperature was optimized and both the treated WEO and solid contaminants properties were characterized. It is expected from this study that the optimum condition to efficiently treat the WEO can be identified and characteristic of the solid contaminants can be revealed for various other potential applications.

2. MATERIALS AND METHOD

2.1. Chemicals and Instrumentation

Shell, Liqui Moly, Penzoil and Castrol branded WEO were collected from a car workshop (BMB Automotive Centre, Selangor, Malaysia) were used in this study. Glacial acetic acid and kaolinite were purchased from Merck company. Instruments that were used in this study are hot plate stirrer, centrifuge, field emission scanning electron microscope (SEM) with energy-dispersive X-ray spectroscopy (EDS) and viscotester VT-06 RION.

2.2 Experimental Set Up

The optimization experiment was carried out using Castrol brand WEO followed by comparison study at the optimized condition using three other different brands of WEO i.e Shell, Liqui Moly and Penzoil. For this study, WEO were collected from the mileage of around 150,000 – 200,000 km. Each WEO was collected into a 2 L container and shaken well for 15 min. After mixing, each oil was then divided into the smaller containers of 0.5 L. This is to ensure that the WEO were well-mixed before being further treated with glacial acetic acid.

2.3 Treatment of WEO by Acid/Clay Refining Treatment

Glacial acetic acid was mixed with WEO using a ratio of 1.0 mL glacial acetic acid to 9 mL WEO respectively. The purpose of this step was to extract the solid contaminants and other impurities from the WEO. This followed by stirring at the normal atmosphere pressure in a closed container at various temperatures for 1 hour. The mixture were left for several hours to allow settling of the solid contaminants at various settling time and further centrifuged for 30 min at 6000 rpm to separate the base oil from the contaminants. The effect of various settling times, stirring speeds and temperatures on the amount of solid contaminants produced during the treatment were investigated. The optimization study was analyzed using Design Expert software v6. Each run was conducted in duplicate.The separated base oil was further mixed with kaolinite at a ratio of 7.5 mL oil to 0.3 g of kaolinite by heating to a temperature of 110 °C for 15 mins. The kaolinite was used to absorb metals and any smell as a result of oxidation of some components in the oil. After that, the sample was centrifuged for 30 mins at 6000 rpm to obtain the treated engine oil.

2.4. Optimization of Acid/Clay Refining parameters for WEO Treatment

Process optimization of acid/clay refining method was carried out using Castrol oil brand WEO based on the three chosen parameters i.e. the settling time, stirring speed and temperature. The settling time was varied for 12, 24 and 36 hour whereas the stirring speed was set at 100, 150 and 200 rpm. The range of temperature was selected at 40 °C, 50 °C and 60 °C while the viscosity index and flash point values were selected as the response. The range of values used in this study was selected based on the findings from the literature. The exprimental design and optimization study were carried out using Design Expert software v6. Central Composite Design (CCD) of Response Surface Methodology (RSM) was used and resulted in a total of 20 experimental runs (Table 1). Each run was conducted in duplicate.

2.5 Analysis of Treated WEO Physiochemical Properties

2.5.1 Viscosity Index

Viscosity testing can indicate the presence of contamination in WEO [8] using viscotester. The value of viscosity index was obtained by comparing the kinematic viscosity value of engine oil at 40 °C and 100 °C [9]. Kinematic viscosity was calculated by dividing the dynamic viscosity of a fluid by its density based on ASTM D2770. The viscosity index for the fresh oil and untreated WEO were also measured as comparison.

2.5.2 Flash Point

The flash point of samples was analyzed using open cup flash point apparatus by ASTM D97 [10]. A beaker containing 5 mL of sample was placed on a bunsen burner which was fitted with a thermometer. A flame source was brought at intervals to determine the temperature at which a flash appears on the surface of the sample while the treated WEO in the beaker was heated. Flash point for fresh oil and untreated WEO were also measured as comparison.

2.6 Characterization of Solid Contaminants

After centrifugation step, the solid contaminants were collected and cleaned several times with ethanol to remove oil residues. They were then transferred into a petri dish and dried in an oven at 50 °C for 24 hours. The weight of the dried solid contents was measured, and the structure and compositions of the solid contaminants were determined using SEM-EDS.

3. RESULTS & DISCUSSION

3.1 Process Optimization

The best treatment conditions should be identified from the design of experiment so that the quality of the treated WEO is comparable to the fresh engine oil. As shown in Table 1, 20 experimental runs were performed at three level values for each selected parameters and each run was performed in duplicate. The responses were viscosity index and flash point (°C). It is found that the settling time of 24 hours, agitation speed of 150 rpm and treatment temperature of 50 °C (Run 1, Run 9 and 15) exhibited values of viscosity index and flash point that is close to the value of fresh engine oil's viscosity index and flash point (up to 95%). This is attributed to the possible conversion of contaminants by the acid and removal by kaolinite from the lube oil [5]. These comparable properties also indicate that glacial acetic acid has no reaction with the base oil. Meanwhile, Run 7 and Run 11 have the lowest value of viscosity index and flash point values compared to the fresh oil properties. This observation depicted that less efficient treatment of the WEO occurred at shorter setting time (12 hours).

Table 1: The average viscosity index and flash point of treated WEO based on RSM design

Run	**Factor 1: Settling Time [hour]**	**Factor 2: Stirring Speed [rpm]**	**Factor 3: Temperature [°C]**	**Response 1: Viscosity Index**	**Response 2: Flash Point [°C]**
Fresh oil				768.23	173.30
1	24.00	150.00	50.00	767.86	170.33
2	24.00	150.00	60.00	652.67	158.53
3	24.00	150.00	40.00	775.66	165.67
4	36.00	100.00	40.00	635.68	169.67
5	12.00	200.00	40.00	725.73	163.97
6	36.00	150.00	50.00	626.56	167.90
7	12.00	150.00	50.00	539.91	158.00
8	24.00	100.00	50.00	732.26	165.17
9	24.00	150.00	50.00	731.19	172.30
10	24.00	200.00	50.00	763.13	165.73
11	12.00	200.00	60.00	533.77	153.77
12	36.00	200.00	60.00	689.45	149.37
13	24.00	150.00	50.00	630.43	164.73
14	36.00	200.00	40.00	657.14	163.17
15	24.00	150.00	50.00	763.33	169.07
16	24.00	150.00	50.00	737.51	169.60
17	24.00	150.00	50.00	734.76	151.47
18	12.00	100.00	40.00	762.05	144.87
19	36.00	100.00	60.00	630.20	163.70
20	12.00	100.00	60.00	722.74	133.33

Viscosity index is an empirical number that is used to indicate the temperature dependence of oil's kinematic viscosity. The value can be determined by comparing the kinematic viscosity value of the oil at 40 °C and 100 °C. A high viscosity index signifies relatively low change of viscosity with the temperature, which also means good thermal stability and low temperature flow behaviors that provides better protection of the engine under vast temperature variations [2, 11]. A decrease in viscosity index value of WEO compared to fresh oil is due to contamination in the form of solid contaminants or dilution with light fuel.

Menwhile, the flash point test gives an indication of the presence of volatile compounds in oil and is the temperature to which the oil must be heated under specific conditions to give off sufficient vapor to form a flammable mixture with air [5]. The decrease in flash point for the WEO compared to fresh oil indicates that the oil has been contaminated with volatile products that resulted from oxidation of the engine oil [12] which indicates reduced quality of the oil.

The results were fitted into a second order quadratic model coded units (Eq. 1) in which the positive sign in front of the terms indicates synergistic effect while negative sign indicates antagonistic effect. Eq. 1 is used to determine the predicted response of flash point value within the range of experimental condition.

$$Y\ (Flash\ point) = 162.21 - 5.85A + 4.24B + 4.68C - 6.02A^2 + 8.03B^2 - 4.17C^2 + 5.02AB + 0.48AC - 1.70BC \qquad (1)$$

where A = settling time (hour), B = stirring speed (rpm) and C = temperature (°C)

The analysis of variance (ANOVA) is then carried out for the response in order to test the significant of the design as presented in Table 2. The model p-value (0.0457) indicates that the model terms are significant, as the probability > F is less than 0.05. In this case, A (settling time) and C (temperature) are significant model terms. The design suggested that settling time is the most significant variable with P-value of 0.0186 followed by the mixing temperature with P-value of 0.0486. Values greater than 0.1 indicate the model terms are not significant. The “Lack of Fit F-value” of 1.15 implies the lack of fit is not significant relative to the pure error. Based on [13], the design model was strongly recommended to have insignificant lack of fit in order to represent an adequate design model.

R-squared value obtained was 0.7370 which indicated the model had 73.70% of variation in response to the dependent variables. The negative predicted R-squared of -0.2754 implies that the overall mean is a better predictor of response than the current model.

Table 2: Analysis of Variances (ANOVA) for model regression

Source	Sum of Squares	Df	Mean Square	F value	p-value Prob > F
Model	1218.30	9	137.37	3.11	0.0457
A – Settling Time	342.22	1	342.22	7.87	0.0186
B – Stirring Speed	179.78	1	179.78	4.14	0.0694
C - Temperature	219.02	1	219.02	5.04	0.0486
A^2	99.75	1	99.75	2.29	0.1608
B^2	177.20	1	177.20	4.08	0.0711
C^2	47.88	1	47.88	1.10	0.3186
AB	202.00	1	202.00	4.65	0.0565
AC	1.81	1	1.81	0.042	0.8426
BC	23.12	1	23.12	0.53	0.4825
Residual	434.68	10	43.47		
Lack of Fit	232.28	5	46.46	1.15	0.4418
Pure Error	202.39	5	40.48		
Cor Total	1652.98	19			

Fig. 1A and Fig. 1B show the viscosity index and flash point value for each run which is extracted from Table 1. From Fig. 1A, the condition for the highest flash point took place at 50 °C with the stirring speeds of 200 rpm and 100 rpm, respectivey for 24 hours of settling period. This result suggested that the mixing speed has no significant influence on the chosen treatment parameters which is supported by ANOVA results. It is reported that increasing the temperature above 50 °C will result in poorer quality of oil [14] which is also observed in this study as presented in Table 1. Similarly, Fig. 1B suggested that at 24 hours of settling time, the highest flash point value can be obtained at 50 °C t at the stirring speed of 150 rpm.

Based on the optimized process conditions (settling time of 24 hours, temperature of 50 °C and mixing speed of 150 rpm), comparison of four different brands of WEO (Castrol engine oil, Shell engine oil, Liqui Moly engine oil and Pennzoil oil) were conducted. The properties of WEO before and after the treatment using acetic acid/clay method were studied.

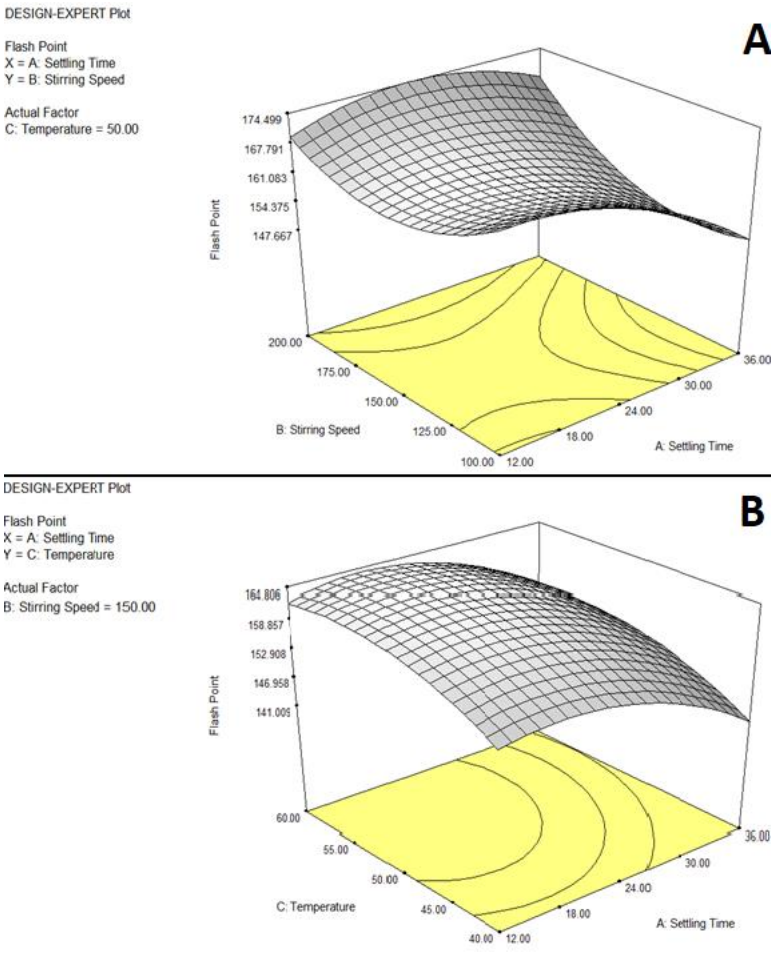

Fig. 1. 3D surface of design model based on settling time and stirring speed (A) and 3D surface design model based on temperature and settling time (B).

Table 3 shows the comparison of viscosity index and flash point values of Castrol, Shell, Liqui Moly and Pennzoil WEO before and after treatment with the acid/clay method. The data shows that untreated WEO has lower viscosity index and flash point values compared to their respective fresh lube oils. The decreasing of viscosity index value can cause several effects such as excessive wear in the components, increased sensitivity of oil to particle contamination, increase friction between components [15]. However, after the treatment of WEO using glacial acetic acid at the optimized condition, the value of viscosity index and flash point value has increased to almost similar to the fresh oil ones. The increasing of both values proved the low destructive effect glacial acetic acid on them [2].

Table 3. Comparison of WEO (different brands) properties before and after treatment using the acidic refining method using acetic acid

Oil Type	Viscosity Index	Flash Point ℃
Castrol Oil (Semi-synthetic)		
Fresh Oil	768.23	173.3
Used Engine Oil[a]	641.50	139.7
Treated Oil	767.86	170.3
Shell Oil (Semi-synthetic)		
Fresh Oil	711.80	188.6
Used Engine Oil[a]	637.00	147.6
Treated Oil	636.05	172.4
Liqui Moly Oil (Fully-synthetic)		
Fresh Oil	779.36	189.7
Used Engine Oil[a]	631.93	155.3
Treated Oil	774.18	175.8
Pennzoil Oil (Mineral)		
Fresh Oil	721.48	193.9
Used Engine Oil[a]	586.07	161.5
Treated Oil	697.98	177.5

As expected, WEO treated at the optimized condition of the glacial acetic acid treatment method has achieved almost 90% similarities in terms of flash point value as the fresh engine oils. Compared to the previous finding reported by Hamawand et al. [2] and Udonne et al. [5], this study has shown to have improved treatment efficiency of the acid/clay method using glacial acetic acid in terms of viscosity index. In contrast, the viscosity index of Shell oil shows that the treated oil is lower than WEO which indicate unsuitability of the method for the treatment of the Shell oil. Similarly, from Table 3, the flash point of all treated WEO is much lower than the value of fresh oil.

3.1. Characterization of Solid Contaminants

3.1.1 Weight of Solid Contaminants

The weight of solid contaminants represents the amount of solid contaminants being removed after the completion of WEO (Castrol brand) treatment with acid/clay method. Based on the literature, it is expected that the settling time of 24 hours will give almost complete removal of contaminants from the WEO [16].

Based on Fig. 2, the highest average weight of solid contaminants (2.17 g) were collected at 24 hours settling time compared to when the WEO was treated at a shorter settling time at the constant tempertaure and mixing speed. However, when the settling time was prolonged to 36 hours, the average amount of solid contaminants started to decline to 1.52 g and this observation explains further increasing the settling time will hamper the separation of solid contaminants from the lube oil. In addition, acetic acid solution helps to break down or separate the solid compounds and metals from the base oil in the WEO [17] by releasing and precipitating the insoluble particles. Meanwhile, the average yield of solid contaminants for 150 rpm mixing speed was higher (2.23 g) than that for 100 rpm and 200 rpm at 50 °C and 24 hours of settling time as shown in Fig. 3. It is reported that mixing can significantly affects the amount of extracted solid contaminants from treated oil [18]. Increasing the stirring speed up to 200 rpm not only consumes more energy but also results in low amount of solid contaminants produced than that of 150 rpm.

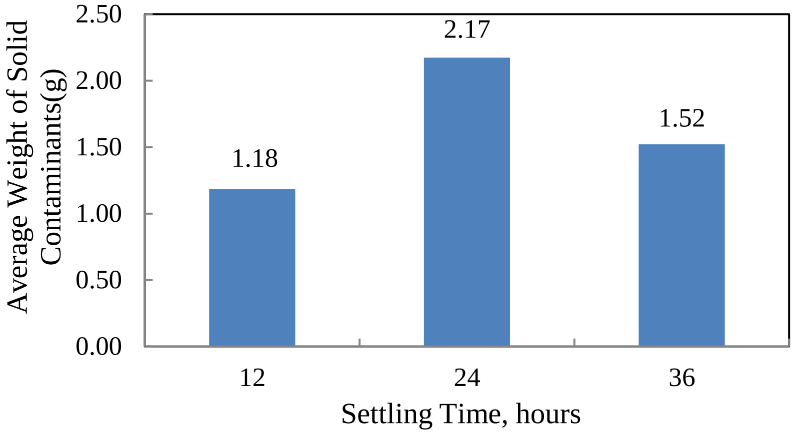

Fig. 2. Average weight of solid contaminants in the treated WEO for various settling times at 150 rpm and 50 °C.

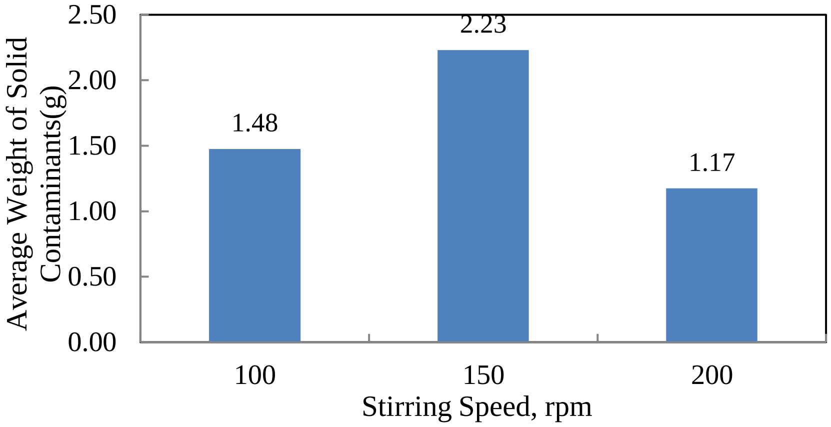

Fig. 3. Average weight of solid contaminants in the treated WEO for various mixing speeds at 50 °C and 24 hours of settling time.

In addition, it can be seen in Fig. 4 that the average weight of solid contaminants increased with the increasing temperature (between 40 °C and 50 °C), but it is further decreasing at the higher temperature (60 °C). It shows that temperature has an effect on the extraction process. This may be due to the high heating mixture of acetic acid and WEO which results in evaporation of acetic acid thus hampers the treatment process and reduced the extraction of the solid contaminants from 2.19 g to 1.39 g. Moreover, by further increasing the temperature, the refined oil yield will result in poor quality of oil [7] which can be observed from the data presented in Table 1.

It can be deduced that the amount of solid contaminants collected after the WEO treatment is related to the improvement of the physical properties of the treated oil. Nevertheless, the results indicated that removal of solid contaminants does not directly represent improved quality of the treated oil when compared to the result obtained in Table 1. This could be explained due to the removal of additive from the engine oil [19].

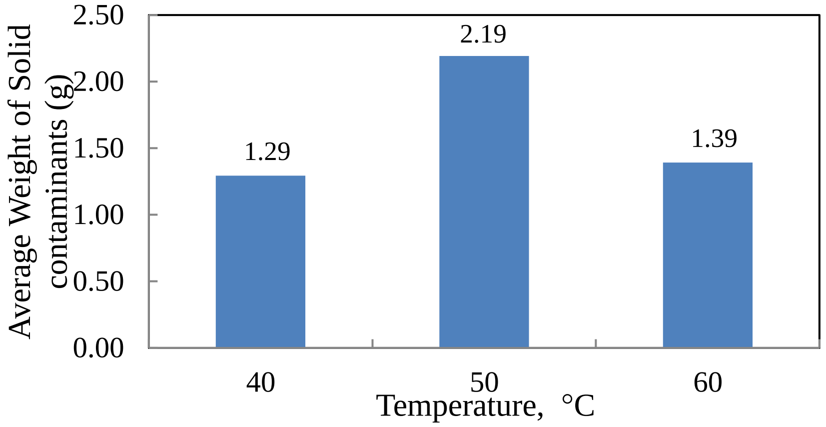

Fig. 4. Average weight of solid contaminants in the treated WEO for various temperatures at 24 hours of settling time and stirred at 150 rpm.

3.1.2 Structure of the Solid Contaminants

The solid contaminats that were collected after the WEO tretament is black in color. The morphological property of solid sample at the optimized treatment condition was further observed under SEM, as shown in Fig. 5 at two different magnifications. SEM observation shows heterogeneous shaped of solid particles with sharp edges as can be observed from Fig. 5A. The size of the particles measured using ImageJ software ranging from 0.6369 mm to 1.186 mm. Under the magnified views, the particles' surfaces are observed to be highly roughed and coarse as shown in Fig. 5B.

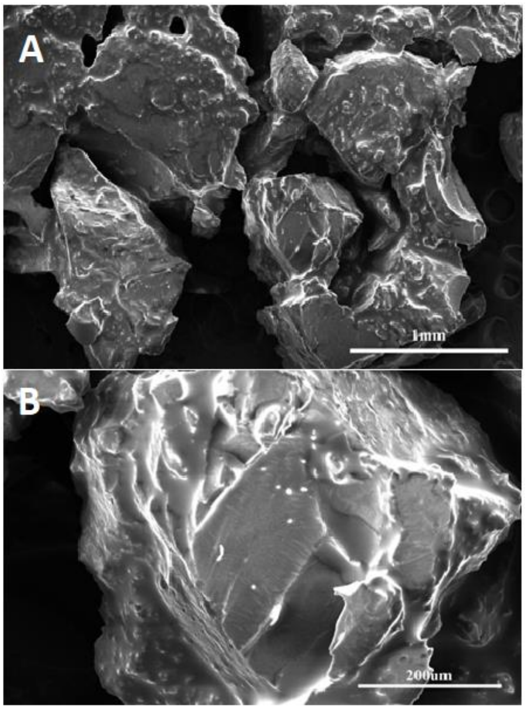

Fig. 5. SEM image of solid contaminants at two different magnifications 130x (A) and 500x (B).

3.1.3 Composition of the Solid Contaminants

The composition of solid contaminants was identified using Energy Dispersive X-Ray Spectroscopy (EDS). Table 3 shows the EDS analysis for solid particles present in the treated WEO which are carbon, zinc, sulphur and phosphorus. The result was confirmed by the higher intensity of positive signals in the mappings of EDS analysis for carbon particles in the solid contaminants. Moreover, the signals for carbon in the samples were homogeneously distributed throughout the inspected area. Table 3 provides the percentage content of the elements. Carbon has the highest percentage content in the solid contaminants with 97.12% followed by calcium (Ca) (1.12%), zinc (Zn) (0.74%), sulphur (S) (0.73%) and phosphorus (P) (0.29%). Carbon residue is a byproduct of thermal decomposition and oxidation of hydrocarbon molecules in lube oil at high temperature [2]. Carbon deposits can harden on intake valves, piston heads, cylinder walls and injectors leading to decreased MPG, hesitation, detonation (knock) pre-ignition, stalling, loss of power and excess emissions [20].

Table 3. The weight percentage of solid contaminants by EDS

Element	Wt%	Wt% Sigma
C	97.12	0.11
P	0.29	0.04
S	0.73	0.04
Ca	1.12	0.04
Zn	0.74	0.08
Total	100.00	

Phosphorus and zinc act as an anti wears or anti-oxidant additive which act as a film to surround metal parts, helping to keep them separated. Meanwhile, sulphur is an extreme pressure additive found along with phosphorus. Extreme pressure agents bond to metal surfaces, keeping them from touching even at high pressure [21]. Similarly, calcium is a detergent-type additive which provides some alkalinity to help neutralize acids formed from diesel fuel combustion [22]. Result shown in Table 3 indicates that some additives were also removed during the treatment which could reduce the physical properties of the treated engine oil in term of flash point value [19]. The engine block is made of aluminum, iron and lead [5]. None of aluminium, iron and lead elements which are the components of the engine block [5] were presence in the analysis. Thus, this demonstrates that no metal wear occur during the combustion of the engine where the WEO were collected.

4. CONCLUSION

This study focuses on improving the WEO treatment method by optimizing the acetic acid/clay refining process parameters. Based on this study, the comparable properties of treated oil to that fresh engine oil was achieved at the settling time of 24 hours, temperature of 50°C and mixing speed of 150 rpm.The values of viscosity index and flash point were found nearly comparable to the value of fresh engine oil's viscosity index and flash point (up to 95% similarity). These comparable properties indicate that glacial acetic acid has no reaction with the base oil and the same condition is suitable to be used to treat various brands of WEO. The highest amount of solid contaminants collected at the optimized treatment condition was 2.23 g consists of carbon (97%), calcium (1.12%), zinc (0.74%), sulphur (0.73%) and phosphorus (0.29%). This study provides an improvement of process efficiency to scale up the treatment of WEO using acidic refining method as well as to open up ideas for further application of the solid contaminants.

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