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Production of High Temperature Grease from Waste Lubricant Sludge and Silicone Oil

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Abstract: This research was carried out to investigate the effects of different ratios of waste lubricant sludge to fumed silica and mixing time on characteristics of high temperature grease produced from waste lubricant sludge and silicone oil. The ratios used were 100:0, 90:10, 80:20, 70:30, 60:40, 50:50 and 40:60. In terms of mixing time, it was varied at 1, 2, 3, 4 and 5 h. In this experiment, the grease was prepared by using heating and mixing technique. The grease produced was analysed according to the American Society for Testing and Materials (ASTM) and National Lubricating Grease Institute (NLGI) standard via penetration test, dropping point test, copper corrosion test, Fourier Transform Infra Red (FTIR) and Atomic Absorption Spectroscopy (AAS). FTIR analysis was carried out to study the functional groups present in the grease. AAS analysis was also carried out to determine the concentration of a specific metal element in the grease. The results show that the grease produced with a minimum ratio of fumed silica to sludge (10:90) was high temperature grease with a dropping point of 272.5°C. Further analysis show that the grease produced is high temperature grease with a worked penetration of 270-290 which makes the grease fall into NLGI number of two. It also has a low tendency to corrode copper and very little metal element was present in the grease. It can be concluded that the ratio of fumed silica to sludge and the mixing time of the grease produced significant effect on worked penetration and dropping point of the grease.

Key words: High temperature grease, lubricant sludge, waste to wealth, work penetration, dropping point

INTRODUCTION

Greases are one of the oldest forms of lubricating material and in the early days, greases are made from environmentally friendly and biodegradable materials such as mutton fat, beef fat and lime. Ancient Egyptians about 1400 BC made crude greases to lubricate the wheels of their chariots (Rudnick, 2003). Grease is a solid to semi-solid lubricant consisting of a thickening agent dispersed in a liquid lubricant such as mineral oil (petroleum oil), synthetic oil (silicone oil) or vegetable oil. The most important factors affecting the properties and characteristics of a grease are the amount and type of thickener used, the viscosity and physical characteristics of the oil and the additives (Sharma *et al*., 1983). Different types and combinations of base fluids with the thickener and additives and the way in which the grease is made will produce greases with different lubricating properties. For example, low viscosity oil is chosen as the base oil in situations where light loads, fast speeds and low temperatures are applied whereas, high viscosity base oils are for different conditions. The thickener will determine grease properties such as water resistance, high-temperature limit, resistance to permanent structural breakdown, "stay-put" properties and cost (Sharma *et al*., 1983; Cann, 2007).

Currently, many researches were conducted to explore alternative sources for grease production due to the increasing of crude oil prices in the world market and the depletion of the source. To date, there is no specific research on the production of grease using lubricant sludge as thickener in its formulation (Dresel, 1994; Couronne *et al*., 2000; Martin-Alfonso *et al*., 2007; Moreno *et al*., 2008; Sanchez *et al*., 2011). Lubricant sludge is a waste generated during solvent extraction process of used lubricant oil. This process is usually performed to recover a significant amount of base oil from the used oil. In this study, waste lubricant sludge was recovered and reuse as thickener in grease formulations. The effects of different ratios of waste lubricant sludge to fumed silica and mixing time on characteristics of high temperature grease produced were also investigated. The successful of this research will not only reduce the disposal cost of waste lubricant but will also produce grease at a low cost.

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MATERIALS AND METHODS

Materials: Silicone oil supplied by R and M Chemicals was selected as base oil for grease formulation. Hexane 96% purchased from Merck, isopropyl alcohol 99.7% and potassium hydroxide 85% supplied by R and M Chemicals were used as solvent during separation of sludge from used lubricant. The used lubricant sludge was collected from motorcycle workshop at Gambang. Calcium chloride 80%, ammonium fluoride 98% and ethanol 96% supplied by R and M Chemicals were used for preparation of calcium fluoride. Fume silica (SIGMA-Aldrich) and molybdenum disulphate (98.5%, ACROS Organics) were used as thickener and additive, respectively.

Sludge separation from used lubricant: About 0.576 mL of hexane, 864 mL of isopropyl alcohol and 2.6 g of potassium hydroxide were added into a beaker containing 360 mL of used motor oil. The solution was stirred for 1 h to ensure complete mixing. Then, the beaker was covered with an aluminium foil and set aside for one day, for settling filtration system to remove solvent from the sludge. The collected sludge was stored in a plastic container until used.

Preparation of calcium fluoride: Calcium fluoride was prepared using method described by Wang *et al*. (2007).

Grease formulation using different ratios of sludge to fumed silica: Eleven sample of grease were prepared using different ratios of sludge to fume silica. Twenty five grams of silicone oil were heated at temperature of ~100°C. Fumed silica and sludge were added according to the ratio as listed in Table 1. The mixture was continuously heated and stirred at temperature of ~100°C for 1 h. After that, molybdenum disulfide and calcium fluoride were added with the weight of 0.2 and 2%, respectively. The stirring and heating was continued at temperature of ~100°C for 1 h. Then, the grease was cooled to room temperature and store prior to analysis.

Table 1: Ratio of fumed silica to sludge in grease formulation

	Fumed silica		Sludge	
Sample	Wt. (%)	Wt. (g)	Wt. (%)	Wt. (g)
1	0	0.0	0	25.0
2	10	2.5	10	22.5
3	20	5.0	20	20.0
4	30	7.5	30	17.5
5	40	10.0	40	15.0
6	50	12.5	50	12.5
7	60	15.0	60	10.0
8	70	17.5	70	7.5
9	80	20.0	80	5.0
10	90	22.5	90	2.5
11	100	25.0	0	0.0

Wt.: Weight

Grease formulation using different mixing time: The same procedure as mentioned in "Grease formulation using different ratios of sludge to fumed silica" was repeated where, the best ratio of sludge to fume silica were used. However, the mixing time was varied from 1-5 h.

Analysis: The grease produced using different ratios of sludge to fume silica were tested for dropping point in order to determine the best ratio. The grease produced using different mixing time were tested for dropping point (ASTM D-566), work penetration (ASTM D-217), copper strip (ASTM D-4048), metal content using AAS and organic content using FTIR.

RESULTS AND DISCUSSION

Effects of fumed silica to sludge ratio on grease characteristics: Table 2 shows the physical appearance and dropping point for grease 1-7 grease. The colour is varying from shiny black to greyish brown. The colour of grease is affected by the amount of fume silica and oil sludge used. Since waste lubricant sludge and molybdenum disulfide were used, the greases obtained were in dark colour. In terms of dropping point it was varied according to amount of ratio used. In grease sample 1-7, the composition of fumed silica increases while the composition of sludge decreases. In this work, the dropping point for grease 1 and 7 were not tested as the physical condition of both of these greases was unsuitable for the testing.

According to Ding and Wunder (2010), thickener property, combined with the non melting characteristic of the fumed silica will contribute significant effect to the dropping point of the grease. Fumed silica is a commercial grease thickener with a dropping point more than 1500°C. This means that, the higher amount of fumed silica in the grease, the higher the dropping point. However, event though by replacing the fume silica with the oil sludge, the dropping point the grease produced is still considered high and suitable for high temperature application. The exact dropping for grease 5 and 6 were unable to measure due to equipment constrain.

Table 2: Grease's physical appearance analysis and dropping point

Sample	Description	Appearance	Dropping point (°C)
Grease 1	Semi fluid	Shiny, black liquid-like	-
Grease 2	Very soft	Shiny, black	272.5
Grease 3	Soft	Shiny, black brown	288.0
Grease 4	Semi firm	Not shiny, like hardened mud, dark brown	293.0
Grease 5	Firm	Not shiny, like hardened mud, dark brown	>300.0
Grease 6	Very firm	Lumpy, brittle	>300.0
Grease 7	Sandy	White, greyish brown like sand	-

Table 3: Grease's physical appearance

Sample	Mixing time (h)	Food analogy	Appearance
Grease A	1	Peanut Butter	Shiny, dark brown-black
Grease B	2	Peanut Butter	Shiny, dark brown-black
Grease C	3	Peanut Butter	Shiny, dark brown-black
Grease D	4	Peanut Butter	Shiny, dark brown-black
Grease E	5	Peanut Butter	Shiny, dark brown-black

Table 4: Dropping point of grease

Sample	Mixing time (h)	Dropping point (°C)
Grease A	1	237.5
Grease B	2	>300.0
Grease C	3	>300.0
Grease D	4	>300.0
Grease E	5	>300.0

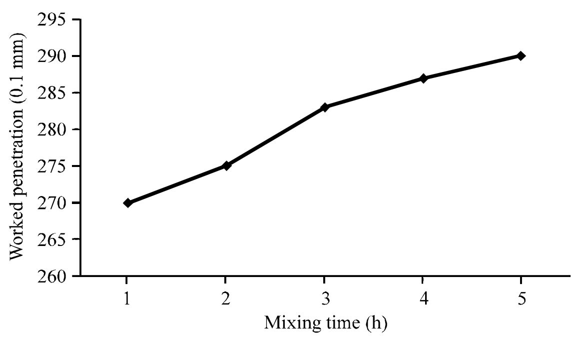

Fig. 1: Worked penetration versus mixing time

Effect of mixing time on grease characteristics

Physical appearance analysis: Table 3 tabulate the physical appearance of the grease. It can be seen that the mixing time does not affect the physical appearance of the grease. Grease A, B, C, D and E has the same appearance which is shiny, dark-brown-black grease. This result is inline with those reported by Abul Bari *et al.* (2008).

Dropping point: Table 4 shows the dropping point of the grease. Grease A has the lowest dropping point which is 237.5°C. The dropping point of the other greases is more than 300°C. The dropping point for grease A should be more than 272.5°C as this was the temperature obtained in the first parameter for greases with 10:90 ratios between fumed silica and sludge and a mixing time of 1 h. However, in this study, results show that the grease A with the same ratio and mixing time as in the first parameter has a dropping point difference of 35°C. The difference in the dropping point may have been caused by human error such as the sudden rise in the thermal oil bath temperature for the dropping point test or the uneven mixing during the production of grease.

Penetration test: Figure 1 shows that the worked penetration of the grease increases with the mixing time. This is because the longer the grease is heated the lower the consistency of the grease. Thus, this will cause the worked penetration of the grease to increase with the mixing point. This finding is inline with those reported by Abul Bari *et al.* (2008). Base on the worked penetration, all the grease formulated can be classified into NLGI number of two. The consistency of the grease is fall into common grease and suitable for highly loaded gearing (Rudnick, 2003; Landsdown, 2004).

Table 5: Copper strip classification

Sample	ASTM rating	Result
Grease A	1b	Pass
Grease B	1b	Pass
Grease C	1b	Pass
Grease D	1b	Pass
Grease E	1b	Pass

Copper strip corrosion test: The copper strip corrosion tests performed in this research indicate that the tendency of the grease samples to corrode under specific static conditions was low as shown in Table 5. According to the ASTM Copper Strip Classification, the copper strip immersed in greases A, B, C, D and E is only slightly tarnished and can be classified under ASTM rating which is a passing ASTM rating. This means that the greases produced in this study can be used in its suitable application as the test indicates that the presence of certain corrosive substances which may corrode equipment, such as acidic compounds or sulphur compounds is not present or is in a very low concentration.

Fourier transform infrared: In order to identify chemicals that are either organic or inorganic in the grease, Fourier Transform Infrared Spectroscopy was used in the present study. It can be utilized to quantities some components of an unknown mixture. The FTIR spectrum for grease A, B, C, D and E is almost the same. This is because; the chemicals used in each of the grease are the same, only the amount varies. Therefore, only one FTIR spectra will be discussed which is FTIR spectra of grease A shown in Fig. 2.

The absorption peak at 2972.02 cm^{-1} can be assigned to the symmetric stretching vibration of the CH_3 groups of the grease. The absorption peak due to silica is located at 1152.48 cm^{-1} while the absorption peak at 3764.07 cm^{-1} can be assigned to isopropanol and potassium hydroxide. The peak from the range of 400-500 cm^{-1} is due to the presence of halogens in the grease. This halogen can be attributed to the fluoride in the additive calcium fluoride (Socrates, 1994). These characteristic absorptions proof the presence of hexane, isopropanol, potassium hydroxide, silicone oil, molybdenum disulfide and calcium fluoride.

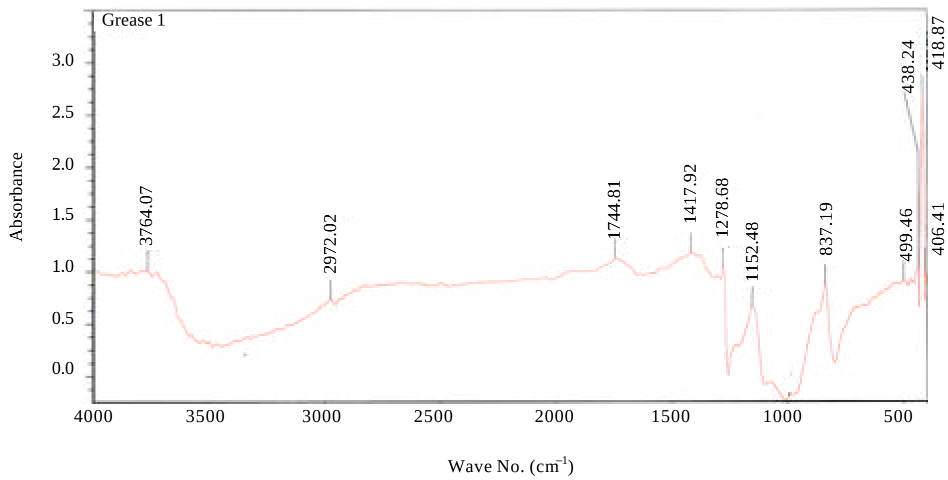

Fig. 2: FTIR spectra of grease A

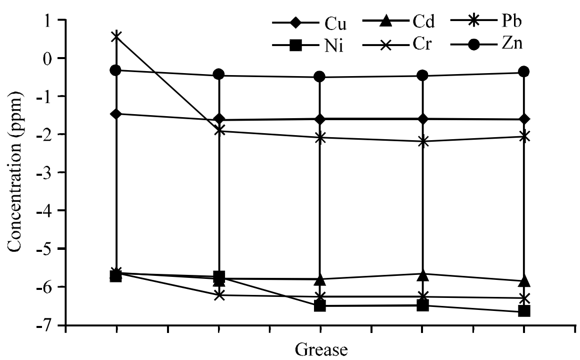

Fig. 3: Concentration of metal element, versus grease

Atomic absorption spectroscopy: Figure 3 shows the concentration for the type of metal element present in the grease sample. The metal elements tested in these analyses were copper, nickel, cadmium, chromium, lead and zinc. Most of the values obtained were of negative value. This is because, the concentration of the standard solutions prepared for the calibration curve were at from range 0.5-100 ppm. Thus, any concentration of metal element lower than the standard solution concentration cannot be read accurately. From the graph, we can see that the presence of the copper, nickel, cadmium, chromium, iron and zinc is very low in grease sample A, B, C, D and E.

CONCLUSION

The effects of ratio of fume silica to oil sludge and mixing time to characteristics of grease was studied. The best ratio of 10:90 (fumed silica: sludge) for grease formulation with a dropping point of 272.5°C and shiny black colour was selected. Five hour mixing time show the highest worked penetration value which are 290. The grease produced is classified as type 2 and suitable for high temperature application which is up to 50°C less than the dropping point value. It is proven that oil sludge could be turned into wealth by reuse it as thickener in grease formulation and thus reducing the pollution problem and at the same time reducing the cost of the grease produced.

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ABBREVIATIONS

AAS	=	Atomic absorption spectroscopy
ASTM	=	American Society for Testing and Materials
FTIR	=	Fourier transform Infrared
NLGI	=	National Lubricating Grease Institute
Cu	=	Copper
Cd	=	Cadmium
Cr	=	Chromium
Ni	=	Nickel
Pb	=	Plumbum
Zn	=	Zinc

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