RE-REFINING OF BASE OIL FROM SPENT LUBRICANT: A COMPARATIVE STUDY BETWEEN LABORATORY AND PILOT SCALE STUDY

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To my utmost supporters;
Ayahanda Abdul Karim Sidam, Bonda Halidzah Talib,
my sisters Amalina Asyikin & Anati Asyran,
my brother Hezerul and
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ABSTRACT

A study on the performance of a pilot scale operation of re-refining used lubricant oil via solvent-extraction process route was conducted to investigate its performance in comparison to the laboratory scale operation. Two dependent variables, namely the Percentage of Sludge Removal (PSR) and Percentage of Oil Loss (POL), were determined and analyzed as the key parameters in measuring the performance of the extraction process, for laboratory as well as pilot scale operations. The performance of the solvent-extraction process in pilot scale was analyzed by varying the solvent-to-oil ratio (SOR) and the amount of potassium hydroxide (KOH) addition, and the results were compared with the data collected from the laboratory scale operation. In addition, the physical properties of spent lubricant and recovered base oil were analyzed and compared between the two scales of operation. In view of the practicality and commercial aspect of the project, the used solvent from previous extraction process was recycled for consecutive run. Thus, the composition of the recovered solvent must be identified to understand its effect on the performance of the solvent-extraction process. Results from the study showed that, in general, the PSR and POL for laboratory and pilot scales operation revealed similar trend of curves, but with expectedly lower performance from the pilot scale study. The main reason of the lower performance from pilot scale operation was due to the change of composite solvents' composition during the solvent-recovery process, which dragged the solvent/oil mixture into the two phase region of the ternary phase diagram. This results in the dispersion of sludge during extraction process. Another factor, which contributed to the difference in the performance of the laboratory and pilot scale study, was the consolidation process. The process simulation, which was carried out for the pilot scale study showed suitability to represent the pilot scale operation, where the data generated from the simulation was acceptable, with percentage error of $\pm 10\%$.

ABSTRAK

Kajian ini berkisar tentang pemulihan semula minyak pelincir terpakai melalui kaedah pengekstrakan pelarut dalam skala loji pandu, sebagai perbandingan terhadap operasi skala makmal. Dua pemboleh ubah bersandar, iaitu Peratus Penyingkiran Enapcemar (PSR) dan Peratus Kehilangan Minyak (POL) ditentukan dan dianalisis, sebagai parameter utama untuk mengukur dengan tepat proses pengekstrakan, dalam kedua-dua skala. Proses pengekstrakan pelarut dalam skala loji pandu dianalisis dengan mempelbagaikan parameter penting yang memberi kesan kepada proses tersebut iaitu nisbah pelarut kepada minyak (SOR) dan jumlah penambahan kalium hidroksida (KOH), dan keputusan yang diperoleh dibandingkan dengan data yang dikumpulkan melalui operasi skala makmal. Seterusnya, ciri-ciri fizikal minyak pelincir terpakai dan minyak dasar yang telah dipulihkan dianalisis dan dibandingkan antara kedua-dua skala operasi. Dari sudut praktikal dan komersial, pelarut yang telah digunakan dalam proses pengekstrakan yang sebelumnya digunakan semula dalam proses pengekstrakan yang seterusnya. Maka, komposisi pelarut yang diperoleh semula perlu dikenalpasti untuk memahami kesannya terhadap keupayaan proses pengekstrakan pelarut. Secara umumnya, PSR dan POL bagi operasi skala makmal dan loji pandu yang telah dijalankan, membentuk graf yang sama, tetapi keupayaan yang ditunjukkan oleh skala loji pandu adalah lebih rendah. Keadaan ini disebabkan perubahan komposisi pelarut komposit yang melalui proses perolehan semula pelarut, dimana komposisi larutan pelarut-minyak berada di dalam zon dua fasa bagi diagram tiga fasa. Selanjutnya, proses penyerakan enapcemar berlaku semasa proses pengekstrakan. Penyimpangan keputusan dalam skala makmal dan loji pandu juga disebabkan oleh proses pemampatan. Data yang dijanakan daripada simulasi proses bagi mewakili operasi skala loji pandu dapat diterima dengan peratusan ralat $\pm$ 10%.

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c	Cohesive energy density
ΔH	Heat of vaporization
T	Temperature
V_m	Molar volume
R	Gas constant
δ	Solubility parameter
$C_x - OH$	Several type of alcohol
K^+	Ion of Kalium/Potassium
H^+	Ion of Hydrogen
z_o	Initial total depth of suspension
KOH	Potassium hydroxide
PSR	Percentage of sludge removal
POL	Percentage of oil loss
W_{dry}	Mass dry sludge
W_{oil}	Mass of used oil
W_{wet}	Mass of wet sludge

CHAPTER I

INTRODUCTION

1.1 Background

Used lubricant oil is one of the abundant liquid wastes in the country that needs to be further treated. Currently, this liquid waste has been disposed of to the environment and creates a lot of problems. Used oil is dumped on the ground, down the sewers, or sent to landfills, where it finally either seeps into ground or floats on water surface. Used oil that manages to migrate into soil or ground may come across the underground water sources. When this happens, one-gallon dosage of this used oil will contaminate one million gallon of drinking water. Meanwhile, only one ppm of used oil concentration that manages to float on the water surfaces can be very harmful to marine species. Used oil forms a thin black film on water surfaces and this film blocks the sunlight penetration. As a result, aquatic plant will not be able to photosynthesize and therefore reduces the oxygen supply to marine life. Without oxygen, marine life cannot survive and finally interrupt the natural food chain where human being is the prior victim.

Meanwhile, many companies spend billions of dollars worldwide on exploring, recovering and refining crude oil to obtain a quality lube oil stock. In addition, these companies then spend million dollars on research and development, and marketing of retail product. Then, after utilizing the oil in cars, trucks and machinery, the oil is basically thrown away. This can be considered as waste of precious and non-renewable resource, where the thrown away used oil will finally destroy human life.

Due to these concerns, there is an opportunity if the hazardous of used lubricant oil can be avoided. As mentioned by Lim (2000), the molecular structure of the base oil, which is the main ingredient of lubricant oil, remains at its initial condition; even it has been used for numbers of time. The major problem is the presence of the additives compound and impurities that increase in the used lubricant oil, after duration of usage. This is common condition in lubricant oil when it is being used.

The remaining molecular structure of base oil encourages researchers (Hess, 1979) to carry out an investigation to obtain the base oil from used lubricant oil. Synthetic base oil can be produced in the laboratory, but due to environmental concern and operation cost, the researchers spend their energy and mind towards finding a suitable method to re-refine the base oil from used lubricant oil. The most popular conventional method is the acid/clay treatment. However this method creates another environmental problem due to the production of acidic sludge. The replacement of sulphuric acid consumption with hydrogenation process to re-refine the base oil somehow creates another problem when it requires high temperature to operate optimally. Implementation of high temperature, however, destructs the base oil molecular structure, which can only remain until 320^0C.

The introduction of the solvent extraction method eliminates the usage of high temperature or hydrogenation process in re-refining the base oil from used lubricant oil. This new method retained the structure of the base oil through the whole process. Theoretically, the presence of polar solvent segregates the particles from liquid phase, while slightly/non-polar solvent stabilizes the polymeric molecules (e.g. polyolefins) and other additives. The stable dispersion, which occurs in the presence of this two extracting solvent, is destabilized in the presence of flocculating agent. It is then removed in the form of sludge and may be used as a raw material to the ink industry. The best performance of solvent extraction method is high sludge removal and minimum of oil loss.

In this following research, used lubricant oil is treated under solvent-extraction process, filtration process and followed by the solvent-recovery process. Oil that manages to undergo this treatment is called the treated oil where the impurities were physically removed from the base oil, but the oil appearance, which was affected by its chemical properties (Lim.2000), can be improved by polishing process or adsorption to remove the colour and the odour for acceptance requirement.

This research will be focusing on the solvent-extraction and solvent-recovery process, on laboratory and pilot scales study. The initial stage of this study is on the laboratory scale experimental work that produces the basis in up-scaling the re-refining of used lubricant oil. In laboratory scale, the parameters of interest are the potassium hydroxide (KOH) addition and the solvent-to-oil ratio (SOR). Percentage of sludge removal (PSR), percentage of oil loss (POL) and physical properties of the treated oil, are the measurement of the effectiveness of solvent-extraction process. During solvent-recovery process, solvents are separated from the oil, where the mixture is introduced into the rotary evaporator. The chromatograms of the recovered solvent are obtained as the measurement of the capability of solvent-recovery process as well as the physical properties of the oil after the treatment.

Laboratory scale data are used as a basic pile in up-scaling into pilot plant scale operation. Pilot scale is operated under atmospheric pressure in batch process. The rig is designed for 120L liquid capacity operated at a time, constructed with stainless steel material. Instead of rotary evaporator, the oil/solvent mixture is subjected to the solvent-recovery system comprising the vertical jacketed heater, cyclone separator and condenser. The effectiveness of pilot scale solvent-extraction and solvent-recovery processes are measured similarly to that of the laboratory scale operations.

In this chapter, the discussion is focused on the background of the research as well as the objective of the study and the research scopes. This chapter is followed by a literature review associated to the topic of interest, including the mechanisms involved during the processes, such as extraction, sedimentation and consolidation. Chapter three discusses the methodology used to obtain the required results, where the method to determine the physical properties of the oil, experimental on laboratory and pilot scales, and simulation method are explained. The results are then discussed in chapter four, where the section are basically divided into three parts, comprising the laboratory scale study on the solvent-extraction and solvent-recovery processes as well as the physical properties of the oil, pilot scale study on similar topic to the laboratory scale, and finally the comparison between the results obtain from laboratory scale and pilot scale. Finally, this thesis is completed with conclusions and recommendations of this research in chapter five.

1.2 Objective

The objective of this study is to upscale the previous laboratory work (Lim, 2000) into pilot plant scale of re-refining used lubricant oil by monitoring solvent-extraction and solvent-recovery process.

1.3 Research scopes

In order to achieve the objective stated above this research is conducted with the following research scope;

1) Determination of the physical properties of the oil before and after the solvent-extraction and solvent-recovery treatment, in both laboratory and pilot scales operation.

   The physical properties of interest are the density (kg/m^3), flash point (^0C), kinematic viscosity (cSt), carbon residue content (wt%), ash content (wt%) and water content (wt%). These properties are monitored to study the effectiveness of the solvent-extraction and solvent-recovery processes on the oil that undergoes the above treatment. From previous study (Lim, 2000), the oil should have the reduced value of the density, carbon residue content, ash content and water content, while the viscosity of the oil is increasing as well as its flash point. Reduction and increment values are due to the removal of impurities and dirt during solvent-extraction process, and volatile compounds at solvent-recovery process. *American Society for Testing and Materials* (ASTM) standard methods are used to determine the physical properties of the oil.

2) Laboratory scale experimental and pilot scale study on solvent-extraction and solvent-recovery processes.

Solvent-extraction process is monitored by the Percentage of Sludge Removal (PSR), Percentage of Oil Loss (POL), and the physical properties of the oil. Parameters of interest are the addition of potassium hydroxide (KOH), which is varied in the range of 0g KOH/L Isopropanol to 6g KOH/L Isopropanol, and solvent-to-oil ratio (SOR), which is also varied between the range of 2:1 and 6:1. Solvent-to-solvent (isopropanol to n-hexane) ratio is fixed to 3:2, which is the optimum mixture of basic and polar compound in solvent-extraction process. Laboratory and pilot scales experiment are carried out with above parameter, similarly. In pilot scale experimental work, the process upsets will be monitored as well as some unpredictable conditions, which have not been considered during the laboratory scale testing.

During solvent-recovery process, the operation is carried out at 90-120^{0}C of the heating medium. Solvents are separated from the mixture, leaving the pre-treated oil. Physical properties of the pre-treated oil are determined by ASTM standard method and the recovered solvent composition is detected using gas chromatography to measure the quantity n-hexane and isopropanol in composite solvents. The chromatogram results are used to maintain the best fraction of composite solvent to be re-used in following solvent-extraction process.

3) Simulating a model of pilot plant scale of re-refining base oil from spent lubricant oil using a process simulator.

Based on the experimental work during laboratory and pilot scales, the data are used to simulate a process model that represents the pilot scale experimental process. The process model is used to predict experimental data in wider range since the difficulties, such as time-consuming operation, material tasks, and tedious work, occur during pilot scale experimental work.