

REGENERATION OF CONTAMINATED LUBRICATING OIL BY SOLVENT EXTRACTION

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ABSTRACT: The regeneration of contaminated lubricating oil by solvent extraction method has been known as one of the inexpensive and efficient method. In this work the capability of three cheap and regeneratable solvents i.e. Methyl-ethyl-ketone (MEK), 1-butanol, 2-propanol has been tested for cleaning and regeneration of contaminated lubricating oil. Percentage oil losses were studied at varying solvent to oil ratios and extraction temperatures of 25°C (298K) and 50°C (323K). Percentage oil losses showed a significant decrease as the solvent to oil ratio and extraction temperatures were increased. The best results were obtained with Methyl-ethyl-ketone. The base oil samples regenerated with three solvents were tested for flash point, pour point, cloud point, kinematic viscosity and specific gravity and the results were compared with virgin lubricating oil. The base oil was regenerated from solvent-oil mixtures by treating each mixture under vacuum distillation.

Key Words: Contaminated lubricating oil, Solvent extraction, regenerating used oil, extraction temperature

INTRODUCTION

Lubricating oil having good properties plays a vital role in terms of reduction in heat transfer and friction. Lubricants employed automobile engines can be deteriorated by different factors, such as operating conditions, quality of fuel and ambient conditions [1]. In the absence of proper treatment, the contaminated oil would be wasted that pollutes the atmosphere [2]. The contaminated lubricating oil becomes ineffective for further applications because of the degradation and impurities introduced during the usage. Degradation encompasses alterations in the aimed properties of oil as a consequence of variation in the structure of lubricating oil that results due to several processes that occur at high temperatures i.e. cracking, isomerization and polymerization reactions. During degradation about 50% of the lubricating oil is consumed [3].

Lately, different methods have been suggested for reuse of contaminated lubricating oil among which solvent extraction has been proved as a competent process because it is widely considered as the cheapest method as the solvent can be recovered for reuse [4-6]. In solvent extraction method, contaminated oil and specific solvents are usually mixed in preferred amounts to make sure that the base oil gets highly miscible within the employed solvent. The additives and contaminants that contain carbon are usually present in contaminated oil needs to be discarded by the employed solvent. These contaminants settle down and flocculate due to gravity. The solvent is then separated by the process of vacuum distillation for reuse [7]. The recovered solvent still contains minimal amount of oil, which can be separated by re-distillation to get pure solvent. Solvent extraction method can remove 10-14% impurities of the contaminated oil. This method removes maximum amount of sludge however, little amount of base oil may also be lost in sludge [8].

The purpose of this study is to reduce the oil losses by extraction with a suitable solvent. Parameters such as the amount of solvent and oil, extraction conditions and quality tests are graphically studied to determine the best possible solvent and figure out the conditions for the optimum results. The process has been explained previously in literature is used to regenerate contaminated lubricating oil by solvent extraction method [9]. The choice of solvents plays a vital role in solvent extraction and minimizing percentage oil losses. Different solvents have been used for extraction

process [8, 9]. The process involves mixing procedures to ensure complete miscibility of the solvent in oil mixture. Solvent extracts the base oil, leaving low proportions of oil in the sludge phase. Extracted solvent-oil mixture is then passed through a process of vacuum distillation in order to recover the pure base oil. The base oil samples regenerated with three solvents were tested for different properties, i.e. flash point, pour point, cloud point, kinematic viscosity and specific gravity and the results were compared with virgin lubricating oil.

EXPERIMENTAL WORK

In this study, the retrieval of contaminated lubricating oil by solvent extraction is carried out by using only three solvents i.e. 2-propanol, 1-butanol and methyl-ethyl-ketone (MEK) as a solvent [8].

The process [10] employs the use of lubricating oil obtained from a vehicle running on a motor oil. The lubricating oil was first treated in a batch distillation unit at a temperature of 200°C to remove water contents and lower hydrocarbons. The oil was then filtered to remove large impurities. After the initial steps the lubricating oil was mixed with three solvents in different ratios. The different solvent to oil ratios (V_{sol}/V_{oil}) were varied at (0.8, 1, 1.2, 2, 3, 4 and 5). The homogenous mixtures were formed with the help of an electronic homogenizer operating at 1520 rpm for 25 minutes and at extraction temperatures of 25°C and 50°C in 500 ml beakers. Each mixture after mixing was transferred to the separating funnel and left for a period of 24 hours to settle under the action of gravity.

After 24 hours the mixture of solvent and oil was separated from the sludge. The sludge being heavy was concentrated at the bottom of the funnel. The sludge was removed from the bottom and collected in a separate beaker where it was weighed as m_s and termed as wet sludge. The wet sludge was heated for a duration of 15 min at 100°C to evaporate any dissolved solvents. The dry sludge was left behind the weight of which was reported as m_d . The percentage oil losses were then calculated using the formula:

$$\text{Percent oil losses (\%)} = \frac{m_s - m_d}{m_{oil}} \times 100$$

The base oil was regenerated from solvent-oil mixtures by treating each mixture under vacuum distillation [7]. The vacuum distillation was carried out at a reduced pressure of 700 mmHg and a temperature of 200°C for a period of 120 minutes to separate solvent which is more volatile component in solvent-oil mixtures. The process recovers 85-90% pure lubricating oil from the mixture.

Quality tests

Flash point

The apparatus used for flash point was the Pensky-Marten closed cup apparatus. The flash points were determined by heating the oil in closed cup apparatus. The temperature at which the fumes of oil catches fire momentarily was termed as the fire point

Cloud point

The base oil was introduced into a metallic pot surrounded by ice. The temperature at which oil sample begins to form wax was noted down as the cloud point.

Pour point

Pour point was calculated by placing the lubricating oil in a pot surrounded by ice. The temperature at which oil ceases to flow was noted. This temperature is the pour point of the lubricating oil.

Specific gravity

25 ml specific gravity bottle and weighing balances were taken to measure specific gravity. Empty specific gravity bottle was weighed. Specific gravity bottle was filled with oil and then weighed again. The difference gives the mass of the oil. The mass of the oil was divided with its volume to find the density of the oil. Specific gravity of oil was found by:

$$\text{Specific gravity} = \frac{\text{Density of oil}}{\text{Density of water}}$$

Kinematic viscosity

The kinematic viscosity of the samples was calculated through the Redwood Viscometer II. The time taken by the oil to pass through a small orifice and getting collected in a 50 ml beaker was noted and the viscosity was calculated by using correlations.

RESULTS AND DISCUSSION

Fig. 1, 2 and 3 explains the impact on percent oil losses by different solvent to oil ratio of 2-propanol & oil, 1-butanol & oil and methyl-ethyl-ketone & oil at extraction temperature *i.e.* 25°C and their comparison is plotted in Fig. 4. Result shows that the increase in solvent to oil ratio decreases the percentage oil losses. Increase in the ratio provides more solvent to ensure that more oil gets dissolved into the solvent thus reducing the amount of oil left in the sludge.

Percentage oil losses decreased considerably when the ratio of solvent to oil was increased from 1 to 3. Further increase in the ratio either increases or marks no further change in

percentage oil losses. Increase in the oil losses were observed at ratios of 4 and 5 with certain solvents at both the temperatures under consideration. This phenomenon indicates that increasing the ratio, decreases the recovery of oil as the mutual solubility of the mixture increases. Also, carrying out the process with large amounts of solvent is not economical and increases the operating cost of the process. All the solvents used showed the same results with increase in the solvent to oil ratio, however, methyl-ethyl-ketone provided minimum percent oil losses at 25°C as presented in Fig. 4.

Fig. 5, 6 and 7 explains the impact on percent oil losses and their dependence on the solvent to oil ratio of 2-propanol & oil, 1-butanol & oil and methyl ethyl-ketone & oil at extraction temperature *i.e.* 50°C and their comparison is plotted in Fig. 8. Result shows that percentage oil losses further reduced at higher extraction temperature of 50°C thus more contaminant could be removed. The result can be attributed to the fact that at higher extraction temperature, miscibility of oil in the solvent increases thus reducing its amount in the sludge removed and also higher extraction temperature favors in high sludge removal [10]. Fig. 8 shows that methyl-ethyl-ketone provided better results on comparing with other solvents because of minimum percent oil losses.

The yield can be enhanced by increasing stages of extraction or using a counter-current extraction process but it also increases the overall cost of the process [11]. However, the temperature related to the desired results of extraction can be determined by considering the factors like maximum removal of sludge, least percent oil losses and least cost of the process. Also, the solvent used plays a vital role in the selection of the suitable extraction temperature [8].

Each graph shows the results obtained by experimentation at different conditions of temperature and ratios. The results are presented with the error bands showing experimental uncertainty of $\pm 5\%$.

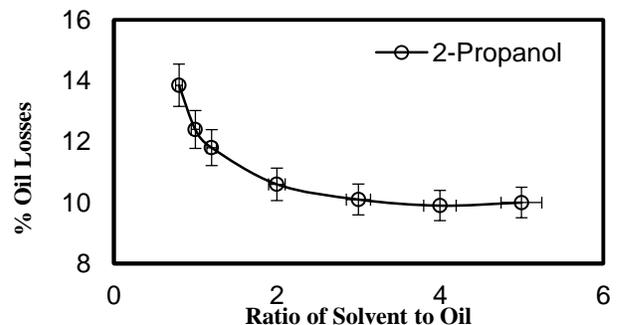


Fig. 1 Effect of percentage oil losses on ratio of 2-propanol to oil at 25°C. Error band shows experimental uncertainty $\pm 5\%$.

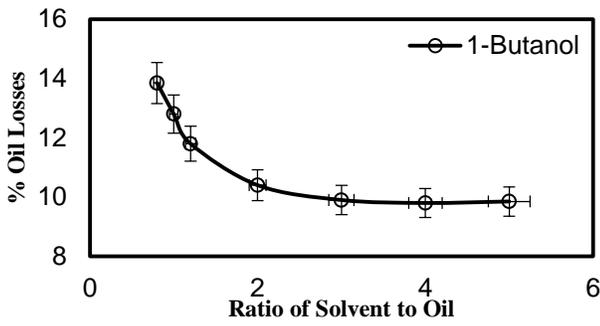


Fig. 2 Effect of percentage oil losses on ratio of 1-butanol to oil at 25°C. Error band shows experimental uncertainty $\pm 5\%$.

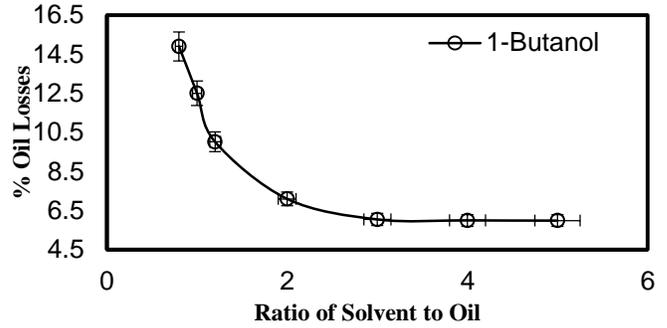


Fig. 6 Effect of percentage oil losses on ratio of 1-butanol to oil at 50°C. Error band shows experimental uncertainty $\pm 5\%$.

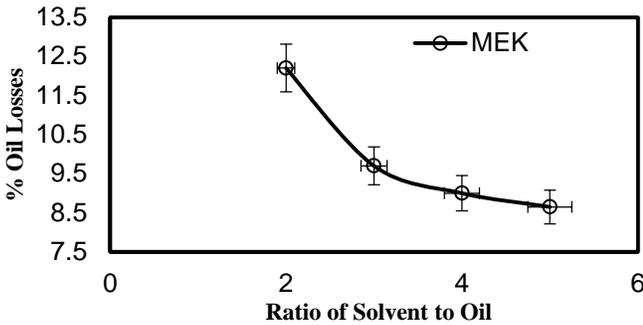


Fig. 3 Effect of percentage oil losses on ratio of Methyl ethyl ketone to oil at 25°C. Error band shows experimental uncertainty $\pm 5\%$.

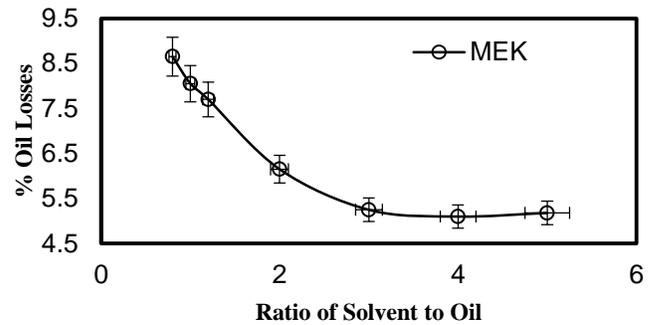


Fig. 7 Effect of percentage oil losses on ratio of Methyl ethyl ketone to oil at 50°C. Error band shows experimental uncertainty $\pm 5\%$.

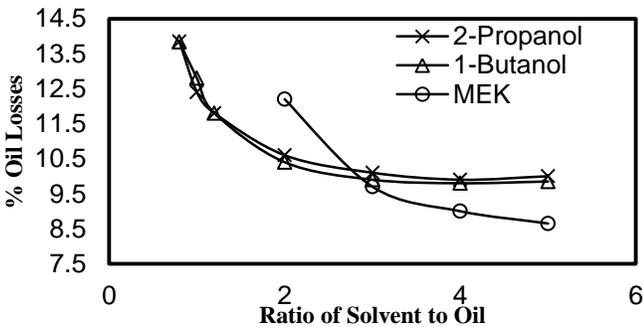


Fig. 4 Comparison of the effect of solvent to oil ratios on percentage oil losses for three solvents of extraction at 25°C.

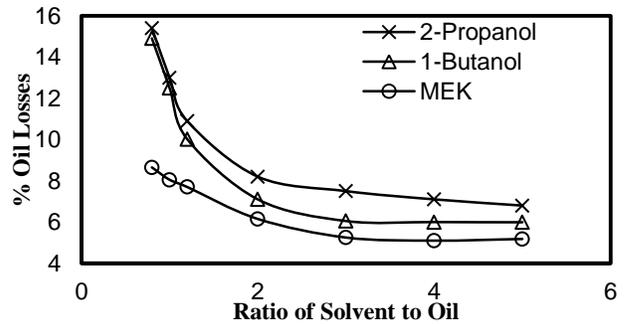


Fig. 8 Comparison of the effect of solvent to oil ratios on percentage oil losses for three solvents of extraction at 50°C.

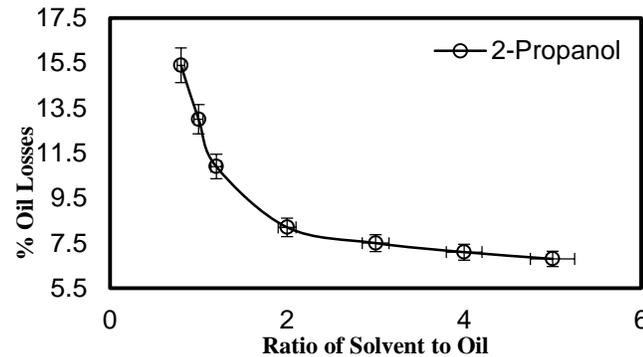


Fig. 5 Effect of percentage oil losses on ratio of 2-propanol to oil at 50°C. Error band shows experimental uncertainty $\pm 5\%$.

Quality Tests

Table 1. Results of the quality tests

	1 Virgin lube oil	2 Contaminated lube oil	3 MEK recovered lube oil
Flash point (°C)	120	186	174
Cloud point (°C)	-29	-7	-9
Pour point (°C)	-33	-9	-11
Specific gravity	0.975	0.960	0.938
Kinematic viscosity at 37°C (centistoke)	71.62	82.20	76.4

Table 1. *contd.*

	4 1-butanol recovered lube oil	5 2-propanol recovered lube oil
Flash point (°C)	171	170
Cloud point (°C)	-11	-14
Pour point (°C)	-13	-16
Specific gravity	0.900	0.872
Kinematic viscosity at 37°C (centistoke)	75.9	76.1

Results of quality tests are presented in Table 1 and are plotted in Fig. 9, 10, 11, 12 and 13 respectively. The properties of lubricating oil recovered by methyl-ethyl-ketone were found closer to the virgin lubricating oil as its flash point was highest among other recovered oils and was closer to that of virgin lubricating oil as shown in Fig. 9. The results of other lubricating oils were also significantly closer to the virgin lubricating oil. All the three recovered lubricating oils had cloud and pour point lower than the virgin lubricating oil with methyl-ethyl-ketone providing more accurate results on comparison as shown in Fig. 10 and 11.

It was found that the recovered lubricating oil samples had close specific gravities. However, methyl-ethyl-ketone showed higher specific gravity as compared to other solvents and also it was found closer to specific gravity of virgin lubricating oil as shown in Fig. 12. 2-propanol and methyl-ethyl-ketone showed values of kinematic viscosity closer to virgin lubricating oil but none of the recovered base oils matched the kinematic viscosity of virgin lubricating oil as shown in Fig. 13.

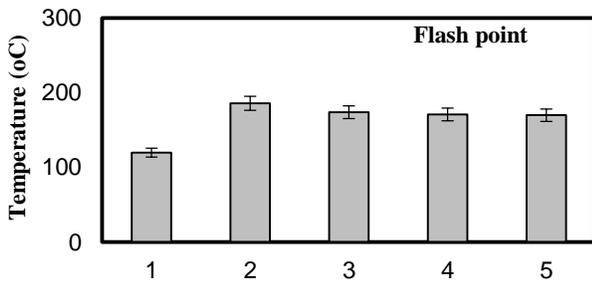


Fig. 9 Comparison of flash point of different lubricating oils *i.e.* 1. Contaminated lube oil 2. Virgin lube oil 3. MEK recovered oil, 4. 1-butanol recovered lube oil 5. 2-propanol recovered lube oil. Error band shows experimental uncertainty $\pm 5\%$.

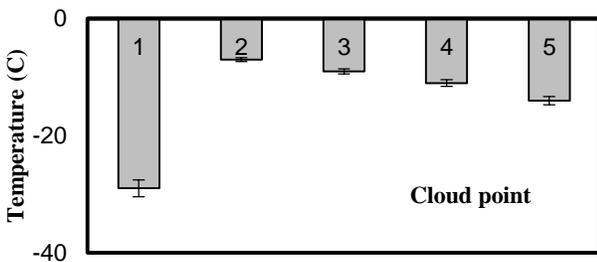


Fig. 10 Comparison of cloud point of different lubricating oils *i.e.* 1. Contaminated lube oil 2. Virgin lube oil 3. MEK recovered oil, 4. 1-butanol recovered lube oil 5. 2-propanol recovered lube oil. Error band shows experimental uncertainty $\pm 5\%$.

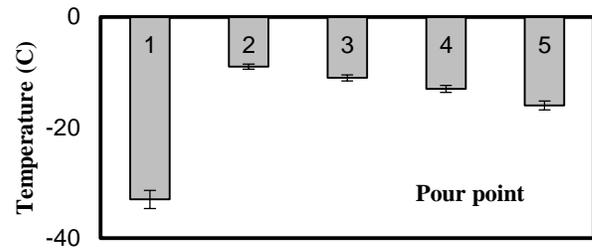


Fig. 11 Comparison of pour point of different lubricating oils *i.e.* 1. Contaminated lube oil 2. Virgin lube oil 3. MEK recovered oil, 4. 1-butanol recovered lube oil 5. 2-propanol recovered lube oil. Error band shows experimental uncertainty $\pm 5\%$.

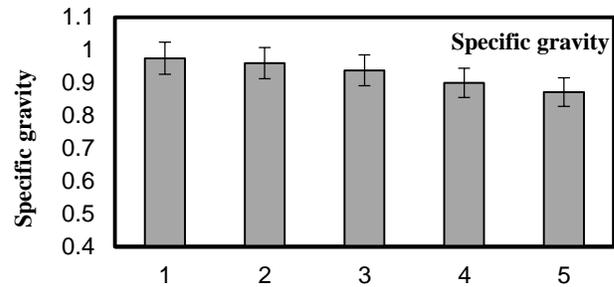


Fig. 12 Comparison of specific gravity of different lubricating oils *i.e.* 1. Contaminated lube oil 2. Virgin lube oil 3. MEK recovered oil, 4. 1-butanol recovered lube oil 5. 2-propanol recovered lube oil. Error band shows experimental uncertainty $\pm 5\%$.

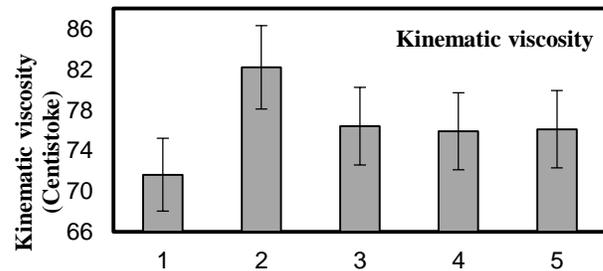


Fig. 13 Comparison of kinematic viscosity of different lubricating oils *i.e.* 1. Contaminated lube oil 2. Virgin lube oil 3. MEK recovered oil, 4. 1-butanol recovered lube oil 5. 2-propanol recovered lube oil. Error band shows experimental uncertainty $\pm 5\%$.

CONCLUSION

Lubricating oil has several applications both in industry and for household purposes. Therefore, the extraction process for recovery of contaminated lubricating oil needs to be considered for further study. This study develops a method by which contaminated lubricating oil can be recovered effectively and inexpensively at large scale with minimal amount of oil losses. The study shows the performance of three cheap and regeneratable solvents at different solvent to oil ratios and extraction temperatures of 25°C and 50°C. The base oil was separated from impurities and regenerated from solvent-oil mixtures by treating each mixture under vacuum distillation. Quality tests were also performed to find the

properties of lubricating oils. The solvent to oil ratio that resulted in least oil losses was found graphically based on experiments. It is concluded that methyl-ethyl-ketone showed the minimum percent oil losses when compared with other solvents and solvent extraction conducted at higher extraction temperature of 50°C resulted in further reduction of percent oil losses because of increased solubility. The quality tests also showed the properties of the recovered base oil samples from all solvents to be in close proximity with the results of virgin lubricating oil. However, the properties of methyl-ethyl-ketone recovered base oil were found more close to virgin lubricating oil than any other recovered base oil. The differences observed on comparing with virgin lubricating oil can be minimized by introducing additives and conducting the process under more controlled conditions.

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