Re-Refining of Waste Lubricating Oil by Solvent Extraction

HASSAN ALI DURRANI*, MUHAMMED IBRAHIM PANHWAR**, AND RAFIQUE AKTHAR KAZI***

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ABSTRACT

Re-refining of waste lubricating oil by solvent extraction is one of the potential techniques. The advantages of solvent extraction technique practically offers from environmental and economic points of view have received due attention. In this paper selection of composite solvent and technique to upgrade the used lubricant oil into base oil has been made. The composite solvent 2-propanol, 1-butanol and butanone have two alcohols that make a binary system reasonably effective. This work also attempts to study the performance of the composite solvent in the extraction process for recovering waste lubricating oil. The key parameters considered were vacuum pressure, temperature and the weight ratio of solvent to waste lubricating oil. The performance was investigated on the PSR (Percentage Sludge Removal) and POL (Percent Oil Loss). The best results were obtained using composite solvent 25% 2-propanol, 37% 1-butanol and 38% butanone by a solvent to oil ratio of 6:1 at vacuum pressure 600mmHg and distillation temperature 250°C. The vacuum distilled oil pretreated with the composite solvents was matched to the standard base oil 500N and 150N, found in close agreement and could be used for similar purpose.

Key Words: **Waste lubricating Oil, Re-Refining.**

1. INTRODUCTION

Automotive waste lubricating oil is generated from the transport sectors when loses its effectiveness during operation because it degrades after a time of use, becomes contaminated and creates a serious pollution problem. This may contaminate water and soil. When it is burnt as a low grade fuel it causes air pollution. Waste lubricating oil creates environmntal pollution, if not disposed properly. The waste oil contains various substances which may enter the food chain through a natural cycle via water. This may pose higher risk to humman health, impede the growth of plants and take up water because waste lubricating oil contains hydrocarbons, heavy metals, PCBs (Polychlorinated Biphenyls) and other halogen compounds [1]. This hazardous waste oil needs proper management to make it a value added product by minimizing the quality of oil being improperly disposed off and reducing the waste oil's environmental burden [2]. Therefore, re-refining of waste oil justifies the interest in elimination of pollution and preserving crude oil reserves.

* Ph.D. Scholar, Department of Mechanical Engineering, Mehran University of Engineering and Technology, Jamshoro.
** Professor, Department of Mechanical Engineering, Mehran University of Engineering and Technology, Jamshoro.
*** Professor, Institute of Petroleum and Natural Gas Engineering, Mehran University of Engineering and Technology, Jamshoro

Waste lubricating oil has been re-refined using many techniques such as chemical treatment [3], physical treatment by distillation and thin film evaporation [4] and solvent extraction [5]. Since the chemical (acid/clay) treatment creates environmental problems, therefore, solvent extraction was introduced as a self alternate to replace it [6]. This solvent extraction treatment has received considerable attention in recent years; because it overcomes the problems associated with acid sludge produced from chemical treatment [5] and its cost is one third of the cost of physical re-refinery [7].

In Pakistan, until now, no WLO management systems are available and the level of public awareness is very low in respect of environmental impacts of waste lubricating oils. According to relatively recent studies, it is estimated that 80,000-100,000 tonnes of waste lubricating oil is generated each year from the vehicles only and is not properly disposed off in Pakistan [8].

In solvent-extraction process the performance was evaluated to upgrade the waste lubricating oil into useful product. A composite solvent of (2-propanol, 1-butanol and butanone) different proportion at various solvent oil ratios was used as the 2-propanol, is a polar compound that segregate the impurities in the form of sludge. Where as the basic compound of the composite solvent is 1-butanol that extracts the spent lubricant oil and the butanone known as MEK (Methyl Ethyl Ketone) is the catalyst that expedites the reaction. The performance was predicted through the temperature rang 60-70^{o}C and the two dependent variables, namely the POL and PSR. It was evaluated at laboratory scale and finally the best re-generated oil result was achieved at the temperature of 250^{o}C using composite solvent of 25% 2-propanol, 37% 1-butanol and 38% butanone at SOR 6:1, based on the performance that is maximum sludge removal, minimum oil losses and maximum sedimentation, it was used on pilot scale level, regenerated the used oil at different operating variables. The regenerated used oil recovery was found about 68% and oil sample properties of regenerated oil matched to the standard base oil, 500N and 150N, found in closed agreement [9]. The regenerated oil can be used for similar purpose therefore, suitable to restore its original quality as new lubricant when added additives.

The major aim of this study was to re-refine vehicle waste lubricating oil to generate base oil by means of solvent extraction process and to study the effects of various operating variables on the properties of the refined base oil.

2. EXPERIMENTAL WORK

Experimental work was carried out first at laboratory to measure the effectiveness of solvents by two dependent variables i.e. PSR and POL and then solvent extraction process was used to extract the base oil at pilot scale.

2.1 Laboratory Scale Experiment

In this work, a glassware experimental setup was made in petroleum refinery laboratory of Institute of Petroleum & Natural Gas Engineering, Mehran University of Engineering & Technology, Jamshoro, Pakistan. The major steps involved in solvent extraction process are shown in Fig. 1. Waste lubricating oil was collected from different service stations and local vehicles repair workshops/ garages. The collected oil was mixed in a single container. It was believed that it can represent a typical feed stock to a re-refining plant for re-generating automotive waste lubricating oil.

After settling, oil sample (Woil) was mixed with the solvent (Wsol) in a conical flask. It was dehydrated at 200^{o}C under vacuum pressure (600mmHg), cold and then mixed with a composite solvent (2-propanol, 1-butanol and butanone) of different proportion at various solvent oil ratios and mixture was stirred rigorously for 15 minutes.

Different solvent oil ratios were used, varying from 2:1-6:1 to calculate the extraction performance of this composite solvent as described in [10]. The experimental technique

was followed for the separation of sludge phase (additives, impurities and carbonaceous particles) from solvent. Oil phase and the effectiveness of solvent extraction process can be measured by two dependent variables i.e. PSR and POL [11-12].

2.1.1 Percentage of Sludge Removal

After mixing, the solvent oil mixture was heated at 60°C for 30 minutes, the sludge was separated and the mixture of oil and solvent was withdrawn by vacuum. The residue (sludge) was weighed and marked as Wet Sludge (W wet). The wet sludge was then mixed with same solvent and put in an oven at 100°C for 24 hours to evaporate the solvent present in the sludge throughout sedimentation as described in [11] and was maintained at room temperature. It was weighed as Dry Sludge (Wdry) and PSR was calculated by Equation (1) shown in Fig. 2 and Table 1.

$$PSR = \frac{W_{dry}}{W_{oil}} \times 100\% \quad (1)$$

2.1.2 Percent Oil Loss

POL is defined as the loss of oil that trapped in the sludge during solvent-extraction process in mass and did not dissolve in the solvent but quite settled with sludge. It is the amount of waste lubricating oil in sludge phase per 100 grams of used oil. This factor was calculated from the same washing process as stated in [13] and calculated as shown in Fig. 3 and Table 1.

$$POL = \frac{W_{wet} - W_{dry}}{W_{oil}} \times 100\% \quad (2)$$

2.1.3 Sedimentation

Sedimentation is a process where the particles or system of particles under the action of gravity settles out from suspension fluid. This was determined by using tubes of 2cm diameter to make the observation possible by watching. The tubes filled with solvent (25% 2-propanol, 37% 1-butanol and 38% butanone) used the solvent oil ratio 6:1 proportions. It was strongly agitated in inverted tubes 30 times to promote flocculation by velocity gradients.

The height of the settling was measured every 30 seconds from the front scale that almost finished within 30 minutes time. Sedimentation result shows that the complete settlement take place at 1cm in 30 minutes time as shown in Fig. 4.

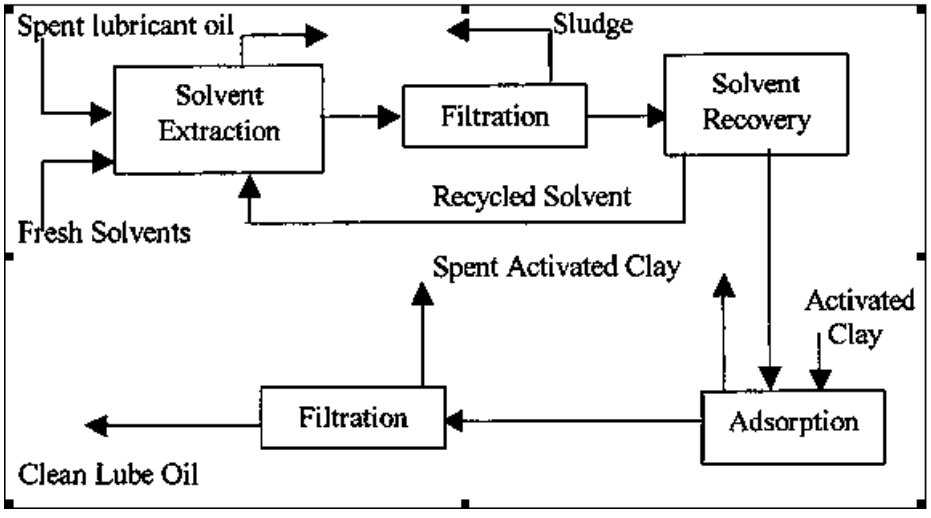

FIG. 1. SOLVENT EXTRACTION PROCESS DIAGRAM

2.2 Pilot Scale

A batch system experimental rig on pilot scale was designed, fabricated and installed in the Department of Mechanical Engineering, Mehran University of Engineering and Technology, Jamshoro, Sindh Pakistan. Various technologies were studied while designing and fabricating of pilot scale rig [14], like Meinken technology, KTI (Kinetics Technology International) technology, Mohawk technology, BERC or NIPER technology (Bartlesville Energy Research Center USA, and PROP technology (Phillips Petrol Company). It has four working processes i.e. dehydration, extraction flocculation, solvent recovery and vacuum distillation. It does not need to be cleaned so frequently because instead of using steam leads, vacuum was produced mechanically to a reduction in the quantity of waste water as shown in Fig. 5. Experimental work on chemical treatment and solvent extraction has been carried out already on this experimental rig similar to laboratory experimental work as a part of Ph.D. work and results were little improved than laboratory scale because of the proper dehydration and distillation [15-16].

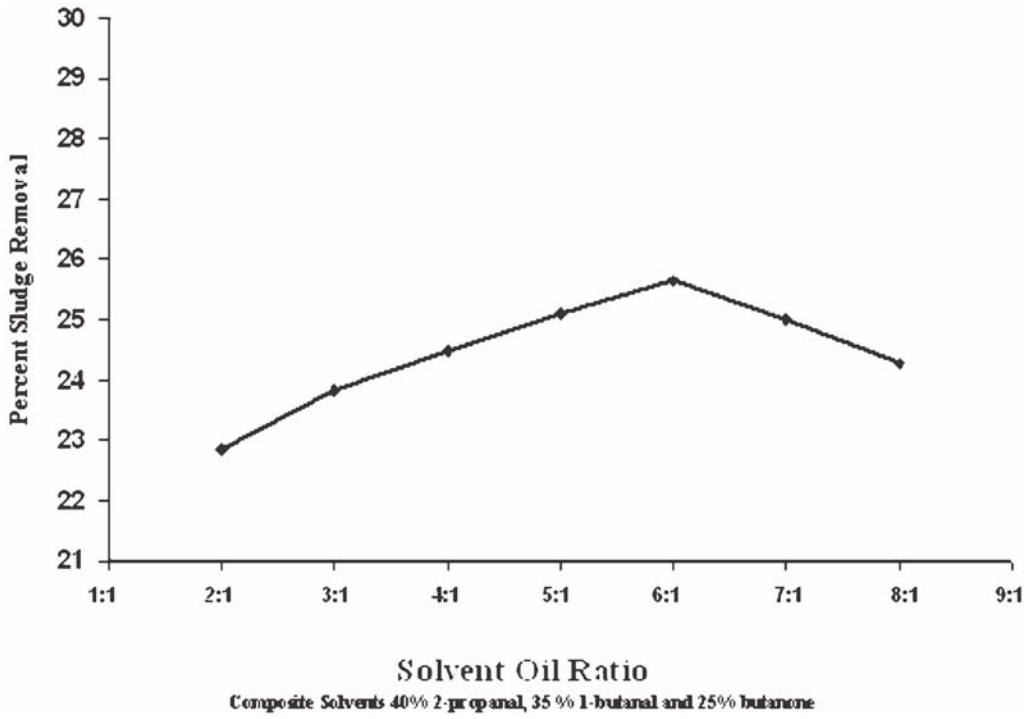

FIG. 2. PERCENT SLUDGE REMOVAL

TABLE 1. OPTIMUM SOLVENT OIL RATIO AND AMOUNT OF SLUDGE REMOVAL

Solvent-to- Oil Ratio	Sludge					Oil Extracted Solvent/Oil		
	Oil (g)	Solvent (g)	Extract Solvent/ Oil Mixture (g)	Affinate (g)	Sludge (g)	Oil (g)	Solvent (g)	Loss (g)
2:1	50.10	100.11	131.80	18.83	12.94	35.39	99.35	1.49
3:1	50.15	150.16	181.11	19.45	13.48	34.81	149.47	1.32
4:1	50.13	200.10	228.76	21.93	13.94	34.67	199.36	1.3
5:1	50.16	250.13	275.73	24.78	14.27	34.28	249.28	1.22
6:1	50.10	300.18	323.75	26.85	14.54	33.97	299.34	1.10
7:1	50.12	350.12	379.89	20.39	14.10	34.17	349.3	1.18
8:1	50.17	400.15	430.83	19.48	13.65	34.44	399.32	1.27

The 2 liters WLO was poured in a measuring cylinder and stirred rigorously for 30 minutes and then left for 24 hours for gravitational settlement to remove heavy particles. After natural settlement oil was pumped and passed through the feed heat exchanger to preheat the WLO. After preheating, WLO entered in the dehydrated column, where the WLO was heated by furnace at temperature of 180°C and circulated continuously for 3 hours. During

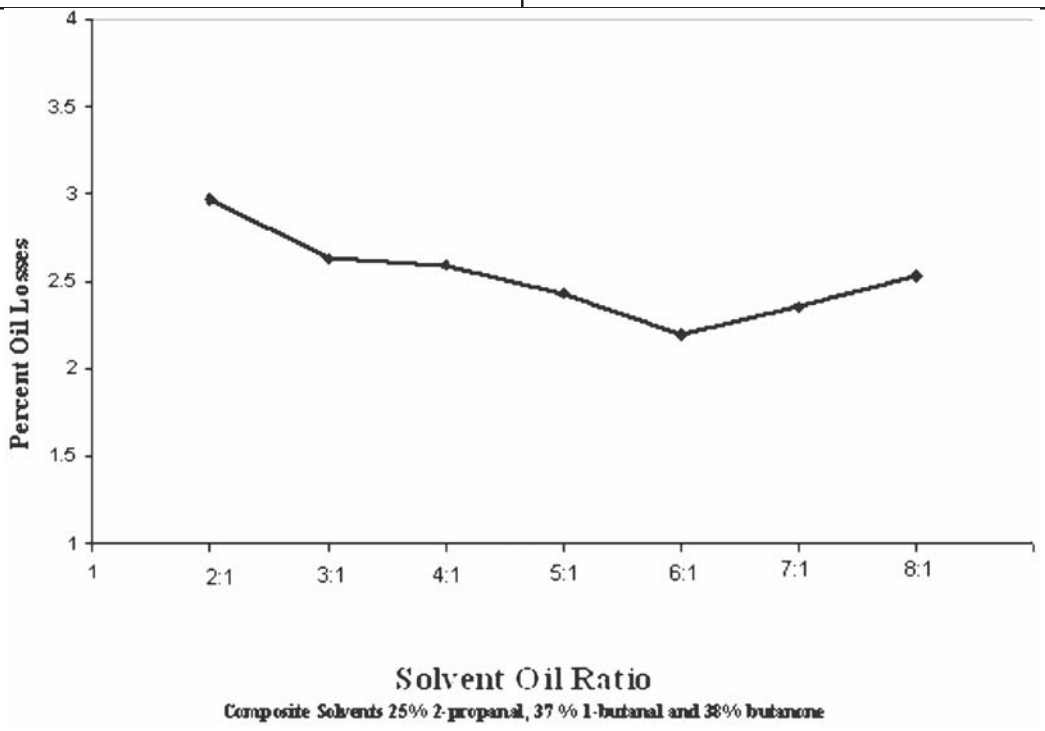

FIG. 3. PERCENT OIL LOSSES

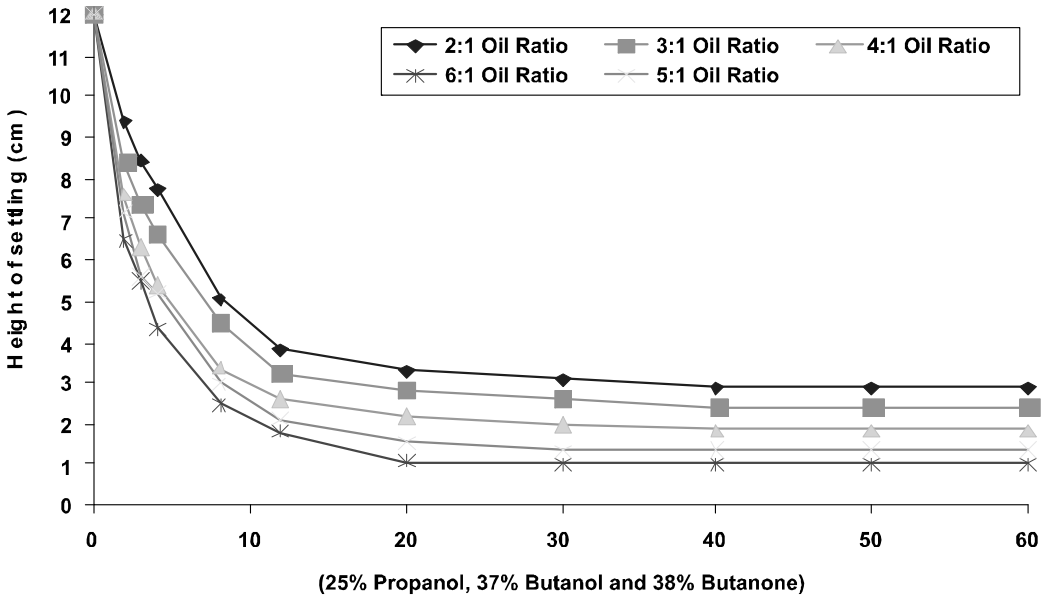

FIG. 4. USED OIL SEDIMENTATION

dehydration WSO was separated from the water and light hydrocarbons, the oil separation degree is due to the function of residence time in the column and drawn through a vacuum pressure 600 mmHg. The collected vapors were condensed by the vapor condenser and drained from the accumulator tank. After dehydration used oil was pumped to dehydrate WLO tank, passed to water cooler through heat pump to reduce the temperature of used oil to 40°C. From the dehydrated WLO tank was pumped to solvent mixture tank, where composite solvent 25% 2-propanol, 37% 1-butanol and 38% butanone was added with solvent waste lubricating oil in the ratio 6:1 and mixed in a mixer for about 20 minutes time. After solvent mixing, the solvent oil solution was transferred to settler tank, where the oil-mixture settled down by a natural gravitation for around 3 hours. The light particles remained on top and heavy particles at bottom. The solvent-oil mixture was drawn from the upper layer by feed pump from the settler tank to vertically long flash distillation column where solvent- oil mixture was initially heated to 60°C temperature for 30 minutes under vacuum pressure 600 mmHg retained. The solvent vapors by vacuum pump lifted the solvent vapors upward through vacuum pressure separated from the oil and collected in the accumulator tank, where as the impurities collided with other moving particles and formed larger particles that settled down at the bottom of the distillation column as the lubricating oil-solvent mixture at temperature range of 90-120°C was heated in accordance to the boiling point of the solvent-oil mixture and for 3 hours at similar vacuum pressure. The light hydrocarbons moved upward and the heavy hydrocarbons moved downward. The oil was separated from the light hydrocarbons in the form of vapors transferred through the vacuum pump, condensed and collected in a diesel accumulator tank. Fuller's earth was added 4 (Wt %) to (Vol %) to oil and was heated at 250°C for 3 hours, the heated oil was passed through cooler where temperature of the oil was reduced and pumped to treated oil tank. The oil through filter-pump then was fed to filter assemblies cleaned from impurities to remove whole impurities and collected in a treated oil tank for browsing. The entire Solvent Extraction Process is given in diagram shown in Fig. 6 where the operating variables are shown in Table 2.

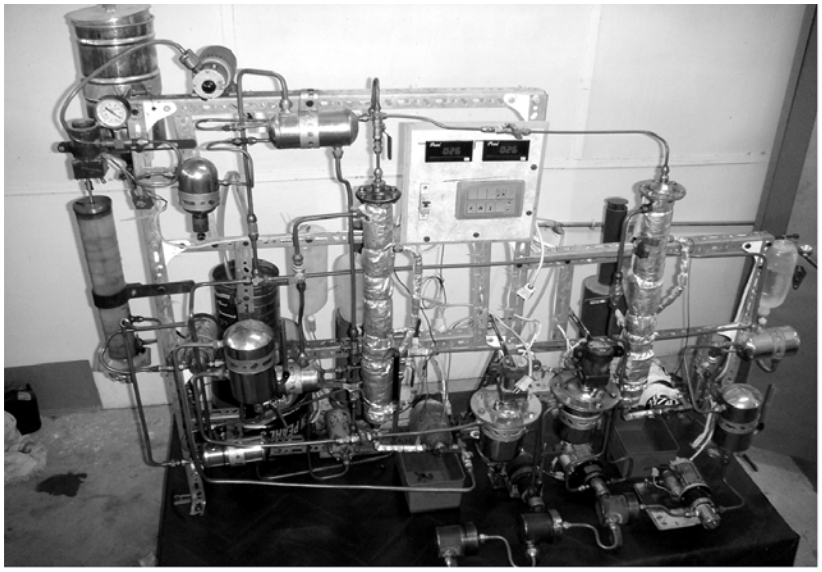

FIG. 5. EXPERIMENTAL RIG

3. RESULTS AND DISCUSSION

The WLO was regenerated into base oil by solvent extraction method. The effective solvent oil ratio of composite solvent 25% 2-propanol, 37% 1-butanol and 38% butanone was evaluated. The results for optimum solvent composition are tabulated in Table 1 that represent the mass balance of the 6 experiments. The percentage of oil recovery for the solvent to oil ratio of 6:1 has further improved solvency oil recovery with improved physical properties of regenerated oil. Solvent to oil ratio lower than the above lead to reduce the solvency power and also not to improve the properties of the regenerated oil this is may be due to ash contents

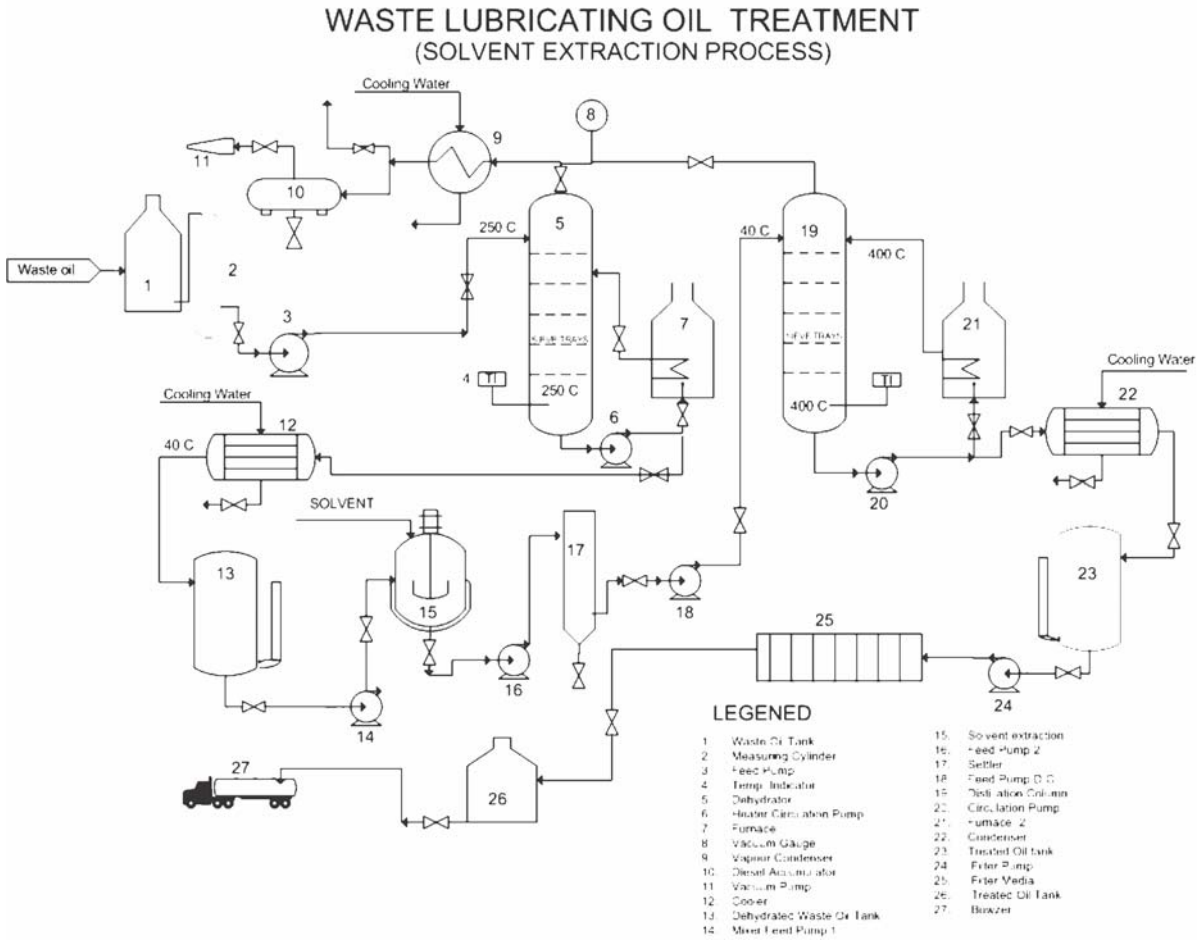

FIG. 6. PROCESS FLOW DIAGRAM OF EXPERIMENTAL RIG

TABLE 2. PROCESS VARIABLES OF SOLVENT EXTRACTION

Process Variables	1st Run	2nd Run	3rd Run
Solvent Oil Mixture (6:1)	2000ml	2000ml	2000ml
Fuller's Earth (wt%)-(vol%)	3	3.5	4
Distillation Temperature °C	150	200	250
Distillation Vacuum Pressure	500 mmHg	550 mmHg	600 mmHg
Distillation Time	1.5 hours	2 hours	3 hours
Gravitational Settling Time	24 hours	24 hours	24 hours
Oil Recovery %	58	62	68

present in the oil. This means that larger solvent to oil ratio will lead to dissolution of some contaminants in the phase especially the ash forming material, which was considered to be undesirable. As a result of the above mentioned facts, the SOR of 6:1 was considered to be the better solvent to oil ratio used for treatment of waste lubricating oil.

The capability of a polar solvent to segregate sludge (contaminants) from waste lubricating oil is closely related to its solubility parameters. The test results show that using higher percentage of butanone than 25% produces lower sludge separation, lower ash reduction and higher percent oil recovery. Since the butanone has lower solubility parameters value than the two alcohols; 2-propanol has the solubility parameter (7.4) that is larger than 1-butanol of (7.0) and both alcohols are much larger solubility difference than butanone of (2.8), therefore it was further added in 25% butanone to improve sludge separation and ash reduction.

Comparing the two solvent composition results, it was found that 25% 2-propanol, 37% 1-butanol and 38% butanone was the most suitable solvent produced higher percent oil recovery with improved quality of oil. For solvent striping, vacuum distillation process was used. There was a problem of foaming of the oil-solvent mixture. This foaming problem led to carry over of the liquid mixture from the still pot to the column, to the condenser, and at the same time to the vacuum lines and this foaming problem was affected by the distillation temperature. In order to define distillation temperature, different temperatures were investigated 125, 150, 180, 200, 225, 250 and 300°C. It was found that the best heating rate with no foaming was around 220-250°C. Higher temperature than 250°C caused severe foaming, so it is important to hold the heat rate constant. Since sudden rise in temperature accelerates foaming, while slow gradual heating rate reduces foaming and for minimum pressure that was used without causing foaming is 600 mmHg. Lower pressure than this resulted in suction of the liquid to the vacuum lines.

The result of investigation indicated that percentage of oil recovery further improved, lowered the sulphated ash content means dissolved contaminants in the solvent phase especially the ash forming, which was considered to be undesirable. Fuller's earth (clay) was used 4 (wt %) to solvent oil ratio (vol %).

The optimum solvents composition provides highest oil recovery and ash reduction. It was found that 25% 2-propanol, 37% 1-butanol and 38% butanone at SOR 6:1 was the most suitable solvent composition to be used with Pakistani WLO produced around 68% oil recovery and the physical properties treated oil was determined using ASTM Standard method. Table 3 represents the physical properties of the virgin and treated oil. Regenerated base-oil was collected from each run and analyzed. The test results in 3rd run were found quite encouraging as the viscosity and flash point increased that is the main function of vacuum created in the distillation process. The vacuum was operated at 600mm Hg at temperature 250°C, similar trends was observed for specific gravity. Pour point was found almost insensitive of the changes in operating variables. Physical properties measured on the oil sample compared with virgin oil indicate that the results found here are similar to those in reference [9] and also as the composite solvent mixture 25% 2-propanol, 35% 1-butanol and 40% butanone found the optimum solvent composition to be used with Iraqi waste lubricating oil produced oil recovery [17].

Optimum solvent mixture 25% 2-propanol, 35% 1-butanol and 40% butanone found the suitable solvent composition to be used with Iraqi waste lubricating oil produced oil recovery 80% [17]. This little difference in composite solvent composition might be attributed to the nature of the base oil crude which is a mixed base for Iraqi crude.

In this experimental work the optimum conditions were determined and results summarized above are: P=600mmHg and T=250°C and fullers' 4 (wt%)-(vol%) and the extraction yield was 68%. The base oil re-generated possesses the physical properties that are required for the formulation of new lubricants.

4. CONCLUSIONS

In this re-refining study proper selection of a composite solvent to recover base oil from waste lubricating oil has been formulated. The study was carried out on a waste lubricant oil mixture drained from different automobiles. The four working process stages were studied, namely: dehydration, extraction flocculation, solvent recovery and vacuum distillation. In dehydration process the best vacuum pressure 600 mmHg and temperature were 200°C. The most excellent oil recovery by extraction was obtained using solvent to oil ratio 6:1 with solvent composition of 25% 2-propanol, 37% 1-butanol and 38% butanone are ash reduction and 68% oil recovery and removal sludge from the waste oils. Distillation of solvent treated oil shows that there are no major differences in oil properties and the fullers' earth gave a good color result.

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TABLE 3. REGENERATED BASE-OIL PROPERTIES

Physical Properties	Standards	Pilot Scale Re-Generated Properties Oil	Fresh Base Oils [4]	
			500N	150N
Kinematic Viscosity at 100°C cSt	ASTM D-445	7.20	10.1	4.91
Kinematic Viscosity at 40°C cSt	ASTM D-445	58.43	90.3	28.5
Specific Gravity	ASTM D-1298	0.8753	0.8861	0.8749
Density	ASTM D-1298	0.8750	0.8856	0.8744
Flash Point (COC)°C	ASTM D-92	222	244	200
Pour Point°C	ASTM D-97	-14	-9	-15
Sulphated Ash Content (wt%)	ASTM D-874	0.021	0.01	0.01
Colour	ASTM D-1500	4.0	4.0-4.5	4.0-4.5

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