

PERFORMANCE EVALUATION OF (N-BUTANOL WITH TRI-SODIUM PHOSPHATE) FOR REGENERATION OF (10W-40) SPENT OIL LUBRICANT

MUDHAFFAR YACOUB HUSSEIN^{1*}, ZUHAIR KUDHAIR ABBAS²,
QURAIISH ABBAS KADHUM² AND AHMED SAMIR NAJE³

¹College of Engineering-University of Misan, Iraq

²Chemical and Petrochemical Research Center, Corporation of Research and Industrial Development,
Ministry of Industry and Minerals, Baghdad, Iraq

³Collage of Water Resource Engineering, AL-Qasim Green University, Babylon 51031, Iraq

(Received 5 May, 2020; accepted 14 June, 2020)

ABSTRACT

Recycling of used lubricating oils by using solvent extraction method is considered inexpensive and achieving maximum productivity with minimum wasted and one of the most effective processes in recycling of used lubricating oils. In this work, the efficiency of extracting solvent (n-Butanol) without catalyzed solvent and with catalyzed solvent (tri-sodium phosphate : TSP), as the pretreatment method in concentration 30 ppm recycling used oil was investigated experimentally, solvent to oil ratio (2:1, 3:1, 4:1 and 5:1 V/V), speed of mixing (500 rpm), mixing time (30 min) and temperature (25 °C). The performance on three evaluated depended factors that the percent of oil loss (POL), the percent of sludge removal (PSR) and the percent of base oil recovered (yield) were used to estimate the efficiency of extraction process. The optimum solvent to oil ratio or critical clarifying ratio (CCR) were (3.4) without TSP and (3.55) with TSP. The maximum recovered base oil (89.5%) by using n-Butanol with TSP and (81.5%) without TSP, minimum oil loss nearly (1.2%) (1.6%) and maximum sludge removal (9%) and (17%) with TSP and without TSP respectively.

KEY WORDS : Solvent extraction, Lubricating oil, Used oil recycling, POL.

INTRODUCTION

High energy demand associated with large quantities of lubricant produced, as a result disposal issue of these quantities raised up (Aremu *et al.*, 2015). Lubricants lost their properties after a designated period of time due to impurities contaminant as a result of use. Contaminated lubricating oils considered as a valuable reusable resource because of high energy potential (Filho *et al.*, 2010). Utilization of oil quantity needed to produce a certain amount of lubricating oil is nearly 9 times lower than that required to produce the same amount of lubricating oil from crude oil (Ogbeide, 2010). Recycling the contaminated oil to base oil (original form) is the effective economically and environmentally way of disposal (Hsu and

Robinson, 2006). Complex processes suggested for recycling because of high variability and difficulty to describe. It's strongly dependent on, the waste oil origin, the waste oil grade, the collection way, and the intended final use of the oil. Commonly four processes are used for contaminated lubricants (Emam and Shoaib, 2013) : acid/clay re-refining process, vacuum distillation/clay process, distillation/ hydro-processing and distillation/ solvent extraction process. From an economic view not all of them are feasible depended on the resources availability and the quality of the recovered base oil which might contain residue byproducts that also require to be treated along with costly energy demand (Ramasamy and T-Raissi, 2007). Regeneration waste oils treatment method preferred as a solvent extraction, because the

problems related with other methods solved by solvent extraction, such as product of acidic sludge and the oldest method acid-clay treatment producer that is not used in developing countries (Hamad *et al.*, 2005). The problems of high loss and cost of oil associated solved with vacuum distillation clay process (Clonfero and Schieppati, 1999); furthermore, solved the difficult hydrogenation step, the possibility of formation stable component stay stable in solution as well as gaseous compounds both potentially pollutant associated with using process of distillation-hydrogenation and the cost of catalyst (Ancheyta *et al.*, 2002; Almutairi *et al.*, 2007). The only solved extraction process disadvantage is that the yield still contain some of the color and impurities not significantly enhanced. Hydrogenation or adsorption can be employed as a final step of this recovery process (Wu *et al.*, 2000). In the solvent extraction-flocculation, the maintreatment step simultaneously performed is, extracted undesirable oxidation and polymerized products mainly (chemical contaminants) along with dirt, metals, and dust (segregated dispersed solid contaminants), by utilizing a select solvent a composite solvent or able to dissolve flocculate the impurities and in the same time dissolve the base oil (Elbashir *et al.*, 2002). Flocculation action depends on the conditions of fluid mixing as well as the characteristics of particles; flocculation of dispersed solid impurities occurring because destabilization of these impurities. Brownian motion because mixing destabilization solid contaminants add to velocity gradient of these flocculation happened (Smoluchowski, 1917; Langelier, 1921). The recovered base oil separated from the used solvent at the end of the treatment could be employed in another processes. The objectives of the current study are to investigate the optimum solvent ratio (OSOR) and to inspect the effect of solvent (n-butanol with tri-sodium phosphate) on percentage of oil yield, oil loss percentage (POL), sludge removal percentage (PSR), and percentage of used oils that are drained into wastewater or soil.

Experimental Work

Materials

In this study one type of oil (10w-40) was used, this type is the automobile lubricating oil which was produced by Al-Durra refinery. This type of used oil was utilized in automobile at operating conditions (2500 km) which represented the feedstock of used

oil. The used lubricant oils properties were determined (at the laboratories of Ministry of Science and Technology) based on Iraqi standard procedure. Solvent used in this work was, analytical grade n-Butanol (Hopkin and Williamsind.).

Method of Working Experiments

Pretreatment

Consists of two consecutive steps, first settling and second heating. Spent oils in the experiments were allowed to homogenize and any settleable suspended solids and free water settled down in a container. Then, to eliminate any residue light contaminants and water the sample was heated to 110 °C (Hani and Al - Wedyan, 2011).

Mixing-

For the purpose of determining the critical clarifying ratio (CCR) the spent oil and solvent were brought to contact together at several ratios (2:1, 3:1, 4:1 and 5:1) solvent to oil ratio (SOR), at constant mixing speed (500 rpm), mixing time (30 min) and temperature (ambient) to be able to estimate the critical clarifying ratio (CCR). The base oil was extracted from the spent in this stage, whereas chemical contaminates and physical contaminates were separated and settled down. At the end of this step, the impact of these solvents is determined visually. The solvent is efficient if oil spent and solvent (mixture) was isolated into two layers extract and raffinate, otherwise the solvent is inactive.

Settling

The spent oil and solvent (well mixed mixture) left at room temperature for 24 hours to be allowed to the flocculated particles (dispersed solid) to settle down. Then two layers would form, the bottom liquid phase was heavier contaminants (material), and the upper phase was solvent of extraction and the base oil.

Washing and drying

Black layer of sludge formed at the bottom of container after the mixture of solvent and spent oil leaved to settle for 24 hours. Then, extracted, weighted and marked this layer as the wetted layer (W_{wet}) that washed with the same used solvent to eliminate any residual trapped oil with the sludge, after that heated the washed sludge to isolate the excess solvent, weighted and marked as (W_{dry}). The

percentage of sludge remover (PSR) and the percent of oil loss (POL) were determined based on the following equations (Durrani *et al.*, 2012):

$$PSR = \frac{W_{dry}}{W_o} \% \quad .. (1)$$

$$POL = \frac{W_{wet} - W_{dry}}{W_o} \% \quad .. (2)$$

Here :

W_o : the initial quantity of spent oil.

w_{dry} : the dried sludge.

w_{net} : the wetted sludge.

Recovery

The up layer was fed into unit of distillation for the base oil removing. Figure (1) shows the distillation unit. The removed solvent can be reused again. The recovered base oil will sent for some tests to estimate if it is valid for use again or not. The following equation can be used to determine the percentage of yield (removed base oil) (Hussein *et al.*, 2014; Najee *et al.*, 2016).

$$Yield = \frac{W_{oil}}{W_o} \% \quad .. (3)$$

Here :

w_{oil} : The recovered base oil.

w_o : The initial amount of spent oil.



Fig. 1. Bench scale of distillation unit.

RESULTS AND DISCUSSION

Figures 2, 3 and 4 showed the influence for process of solvent extraction on the percentage of base oil yield, POL and PSR as a function of the solvent.

Used oil ratio (SOR), were determined using equations (1), (2) and (3) respectively at time of mixing (30 min), speed of mixing (500 rpm) and ambient temperature (25 °C). The POL and SOR were reduced with increasing SOR whereas the yield of base oil was increased for system including used oil and n-Butanol at different magnitude without TSP and with TSP.

Figure 2, shown that n-Butanol with TSP had the highest percentage of yield (recovered base oil) nearly (92%) followed by (85%) without TSP. Figures 3 and 4 shown that the solvent n-Butanol had the lowest percentage of oil loss nearly (1%), followed

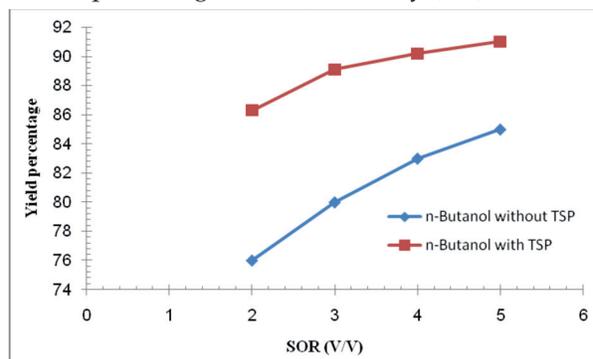


Fig. 2. The effect of solvent: oil ratio on the base oil yield percentage.

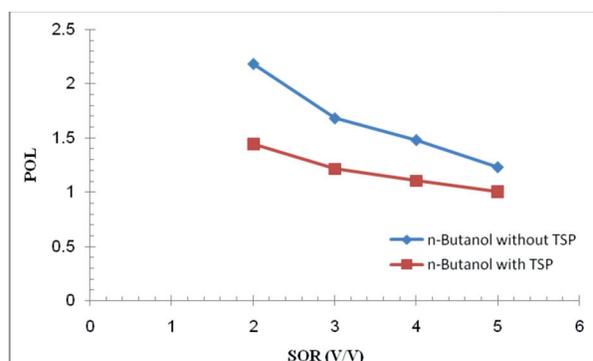


Fig. 3. The effect of solvent:oil ratio on the POL.

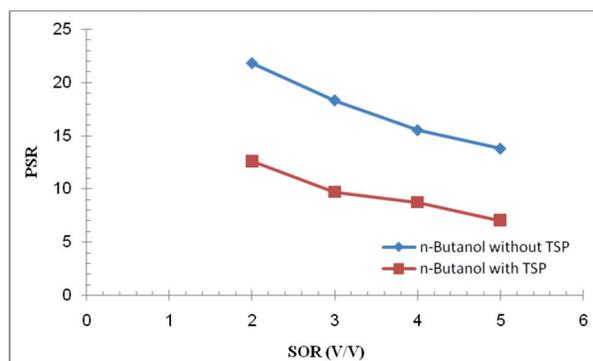


Fig. 4. The effect of solvent: oil ratio on the PSR.

by n-Butanol (1.23%) and PSR (7%) and (14.78%) with and without TSP respectively at SOR (1:5). The different in key parameters percentage (POL, PSR and yield) is due to the addition of TSP, which helps to coagulation and allows for improvement of used oil purification as well as removal of impurities that are in a suspension state or have not been removed in the course of other physical methods. Coagulation is a coalescence of colloid particles.

Figure 5 shown that the value of (CCR) for n-Butanol with TSP is 3.4 and 3.55 (the ratio of solvent to oil) without TSP, which was the better possible results (lower POL, higher PSR and higher base oil yield).

At CCR conditions the maximum yield oil (89.5%) by using n-Butanol with TSP and (81.5%) without TSP, minimum POL (1.2%) (1.6%) and maximum PSR (9%) and (17%) with TSP and without TSP respectively.

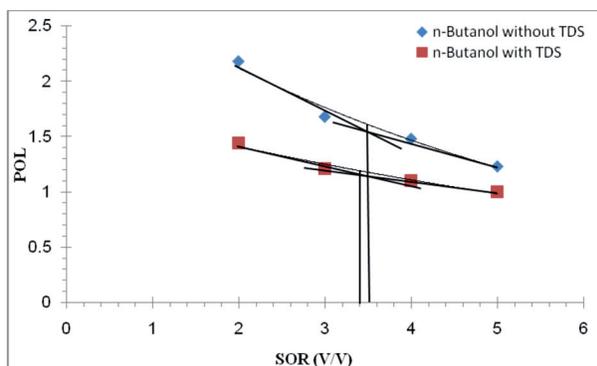


Fig. 5. Graphical estimation of critical clarifying ratio (CCR) based on POL.

The outcomes indicated in Table 1 were obtained at all conditions of solvent:oil ratio (SOR), (2:1, 3:1, 4:1 and 5:1), with mixing time (30 min), mixing speed (500 RPM) and temperature (25 °C). Generally, all the results obtained at (SOR) 3:1 and higher are considered to be identical and acceptable when compared with the Iraqi standard properties for type (10W-40) of lubricating oil. Note that the results using (TSP) with waste oil as a pretreatment is better than the results without (TSP). These outcomes are cheaper in composition and better in performance than those fixed by (Durrani *et al.*, 2012), where yield (80%) of the base oil from Iraqi waste oil using (MEK = 0.4), (n-Butanol = 0.35) and (isopropanol = 0.25) composite solvent.

CONCLUSION

The studied solvent was good in the waste oil regeneration process. The results in agree with the properties of standard oil. Mostly, all the results obtained at (SOR) 3:1 and higher are considered to be identical and acceptable when compared with the Iraqi standard properties for type (10W-40) of lubricating oil. The outcomes of product are considered cheap in composition and better in performance compared with a literature. Solvent n-Butanol with TSP showed a better performance than solvent n-Butanol without TSP. Reducing the percentage of used oils released to the wastewater or eventually to the soil leads ultimately to environmental protection.

Table 1. Properties of standard oil, spent oil and recovered base oil at all (SOR)

Properties	Standard oil	Spent oil	Recovered base oil							
			n-But without TDS. Ratio 2:1	n-But with TDS. Ratio 2:1	n-But without TDS. Ratio 3:1	n-But with TDS. Ratio 3:1	n-But without TDS. Ratio 4:1	n-But with TDS. Ratio 4:1	n-But without TDS. Ratio 5:1	n-But with TDS. Ratio 5:1
Viscosity kinematic at 100 °C	14.607	8.2	10.4	12.6	11.9	13.1	13.4	14.6	14.8	15.6
Viscosity index	Minimum limit = 95	90	92	94	93	96	95	101	100	118
Flash point open	225	179	190	210	212	224	222	227	231	232
Pour point	Maximum limit = -33	-18	-22	-27	-29	-32	-31	-33	-33	-34
Total acid number	Maximum limit = 5	8.1	6.5	5.4	5.8	4.8	5	4.1	4	3.5
Nouk test	Maximum limit = 15	17.6	17.1	15.5	16.2	15.1	14.8	14.6	14.2	12.1
Yield (%)	/	/	76	86.3	80	89.1	83	90.2	85	92

ACKNOWLEDGEMENTS

The authors thank for College of Engineering-University of Misan and Ministry of Higher Education Iraq for support this research.

REFERENCES

- Aremu, M.O., Araromi, D.O. and Gbolahan, O.O. 2015. Regeneration of used lubricating engine oil by solvent extraction process. *International Journal of Energy and Environmental Research*. 3 (1) : 1-12.
- Ancheyta, J., Betancourt, G., Marroquín, G., Centeno, G., Castañeda, L. C., Alonso, F., Muñoz, J. A., Gómez, Ma, T. and Rayo, P. 2002. Hydroprocessing of Maya heavy crude oil in two reaction stages. *Applied Catalysis A: General*. 233 : 159.
- Almutairi, A., Bahzad, D. and Halabi, M. A. 2007. A comparative study on the performance of a catalyst system for the desulfurization of two kinds of atmospheric residues. Kuwait Export and Eocene residual oils. *Catalysis Today*. 125 : 203.
- Clonfero, E. and Schieppati, R. 1999. Reduction of polycyclic aromatic hydrocarbons from thermal clay recycled oils using technical adsorbents. *Polycyclic Aromatic Compounds*. 16 : 41.
- Durrani, H.A., Panhwar, M.I. and Kazi, R.A. 2012. Determining an Efficient Solvent Extraction Parameters for Re-Refining of Waste Lubricating Oils. *Mehran University Research Journal of Engineering & Technology*. 31 (2) : 265-270.
- Emam, E.A. and Shoaib, A.M. 2013. Re-refining of used lube oil, i- by solvent extraction and vacuum distillation followed by hydrotreating. *Petroleum & Coal, International Journal for Petroleum Processing, Petrochemistry and Coal Processing*. ISSN 1337-7027. 55 (3) : 179-187.
- Elbashir, N.O., Al-Zahrani, S.M., Abdul Mutalib, M.I. and Abasaheed, A.E. 2002. A method of predicting effective solvent extraction parameters for recycling of used lubricating oils. *Journal of Chemical Engineering and Processing*. 41 : 765-769.
- Filho, J. L.A., Moura, L.G.M. and Ramos, A.C.S. 2010. Liquid-liquid extraction and adsorption on solid surfaces applied to used lubricant oils recovery. *Brazilian Journal of Chemical Engineering*. 27 (4) : 687 - 697.
- Hsu, C.S. and Robinson, P.R. 2006. *Practical Advances in Petroleum Processing*, Volume 1 and Volume 2". Springer Science, New York.
- Hamad, A., Al-Zubaidy, E. and Fayed, M. E. 2005. Used lubricating oil recycling using hydrocarbon solvents. *Journal of Environmental Management*. 74 : 153.
- Hani, F.B. and Al-Wedyan, H. 2011. Regeneration of base-oil from waste- oil under different conditions and variables. *African Journal of Biotechnology. Antje Hansmeier*, ISBN: 978 - 90 - 386-2264.
- Hussein, M., Amer, A. A. and Gaberah, A. S. 2014. Used lubricating oils re-refining by solvent extraction. *American Journal of Environmental Engineering and Science*. 1 (3) : 44-50.
- Langellier, W.F. 1921. Coagulation of Water with Alum by Prolonged Agitation. *Eng. News-Record*. 86 : 924-928.
- Naje, A.S., Chelliapan, S., Zakaria, Z., Ajeel, M.A., Sopian, K. and Hasan, H.A. 2016. Electrocoagulation by solar energy feed for textile wastewater treatment including mechanism and hydrogen production using a novel reactor design with a rotating anode. *RSC Advances*. 6 (12) : 10192-10204.
- Ogbeide, S.O. 2010. An investigation to the recycling of spent engine oil. *Journal of Engineering Science and Technology Review*. 3 (1) : 32-35.
- Ramasamy, K.K. and T-Raissi, A. 2007. Hydrogen production from used lubricating oils. *Catalysis Today*. 129 : 365.
- Smoluchowski, M.1917. Versucheiner mathematic chen Theorie der Koagulations kinetic Kolloider Losunger. *Zeit. Phys. Chemie*. 92 : 129-168.
- Wu, J. C.S., Sung, H.C., Lin, Y.F. and Lin, S.L. 2000. Removal of tar base from coal tar aromatics employing solid acid adsorbents. *Separation and Purification Technology*. 21 : 145.