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Quantitative Determination Of Sediments In Crude Oil And Lubricating Oils By Toluene – Hexane Mixture In Basic Media Extraction

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Abstract:

An analytical, accurate and sensitive method covers the determination of sediments in crude oil and lubricating oils by extraction with toluene and n-hexane mixture, the precision applies to a range of sediment levels from 0.01 to 0.40 % mass, although higher levels may be determined.

Extract test portion of a representative samples, contained in a refractory thimble, with hot extraction mixture for 30 minutes until the residue reaches constant mass. The mass of residue, calculated as a percentage, is reported as sediment by extraction.

The samples under this study collected from different places at Kurdistan region, Iraq, shows a sediment range of (0.319 – 3.77) %.

Keywords: Sediments, extraction, toluene, hexane, turbine oil

Introduction:

Solvent extraction is the ability of a solute to distribute itself between an aqueous solution and an immiscible organic solvent. The organic solvent separates and purifies the solutes by extracting into the organic phase, leaving undesirable substances in the aqueous phase [1].

A solvent is used selectively to extract the base oil component from the waste lubricant oil. The additives and carbonaceous impurities that are normally present in used oil should be rejected by the extracting solvent, these impurities settle and flocculate based on gravity [2]; the solvent is then recovered by distillation for reuse. However, a portion of this solvent is lost due evaporation during treatment.

Previous studies have shown that n-hexane has good oil extraction ability on used lubricants mainly due to its efficiency and ease of recovery. Moreover, other factors such as physical properties of the solvent, particularly its low boiling point attribute to the choice of n-hexane as the optimum solvent. Toluene was found to be the second-best extracting agent [3,4].

The extractability of oil depends on the nature of the solvent and oil, extraction temperature, contact time between solvent and the feed, pre-treatment conditions of the oil-bearing resource [5]. Properties such as solubility are necessary to evaluate the solvent performance [6], showed that an increase in solubility parameter difference further enhances the solvent capability to extract the additives and impurities from used oil [7].

Used lubricant contains a large proportion of valuable base oil that may be used to formulate new lubricants if undesired pollutants are removed [8].

Many processes for recycling used lubricating oil involve the use of vacuum distillation followed by a polishing or decolorizing treatment. However, serious problems related to coking and column fouling during distillation and, therefore, some form of pre- treatment to remove additives and contaminants from the oil is required [9]. Furthermore, these types of processes are energy demanding due to the high overpressures, deep vacuums and high temperatures applied [10]. The use of extraction has been studied as a low energy consuming alternative by a number of researchers.

Sediments are formed as a result of the process of sedimentation of particles from a layer of water. In the process, organic compounds are transmitted into the sediments together with the solid particles. It is an effect of hydrophobic organic substances getting absorbed on the particles of the suspension, and a reason why sediments are of very complex physico-chemical structure [11].

Experimental:

Apparatus:

The extraction apparatus from Linetronic Technologies (Switzerland) consist of: Extraction Flask, Condenser, Extraction Thimble, Water Cup, thimble basket and Source of Heat, illustrated in Figures 1 and 2 consisting all elements.

Figure 1. Extraction apparatus

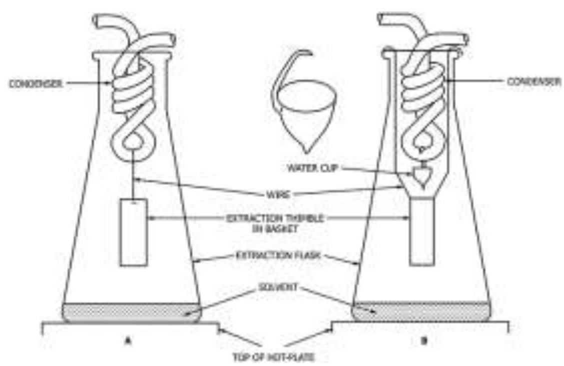

Figure 2. Extraction apparatus elements

Reagents: Toluene (Fluka 99.8%) and n-hexane (BDH 96%), reagent grade used with typical characteristics as shown in table 1.

Table1. Typical Characteristics for reagent grade toluene

Property	Toluene	n-hexane
Chemical formula	$C_6H_5CH_3$	C_6H_{14}
Assay (%)	99.8	96
Molecular weight (gm/mol)	92.141	86.18
Boiling point (ºC)	111	68.7
Melting point (ºC)	- 95	- 95
Density (gm/ml)	0.87	0.6603
Vapor pressure (torr at 20 ºC	60	124
Solubility	0.52 gm/L	Insoluble

Procedure:

(10 gm) of the sample placed into the thimble as soon as possible, (5 ml) of sodium hydroxide of 0.01N added, 160 ml toluene and 40 ml of n-hexane to the extraction flask, and then, thimble set in the extraction apparatus and extract with toluene for 30 minutes after the solvent dropping from the thimble is colorless.

When testing samples having high water contents, the water cup shall be used. In this procedure, any water in the material is removed as its toluene azeotrope and collected in the water cup, where it separates into two phases. The toluene layer overflows into the thimble. If the cup becomes full of water, cool the apparatus slightly and empty the cup.

After the extraction is completed, the thimble must dry for one hour at 120ºC. Cool in a desiccator and weigh to the nearest 0.2 mg [12].

Calculations: The sediment content of the samples as percentage by mass of the original sample calculated by equation (1): [12]

$$s = \frac{m3 - m1}{m2 - m1} \, x100 \qquad (1)$$

S: Sediment content of the sample as a percentage by mass

m_1: mass of the empty thimble (gm)
m_2: mass of the thimble plus tested sample (gm)

m_3: mass of the thimble plus sediment (gm)

Results and discussion:

The process of petroleum products accumulation is a result of high sorption capacity of components of bottom sediments [13].

Length of the period of time throughout which the oil's components and products of their degradation remain within the bottom sediments is dependent on numerous factors, such as: type of sediment, temperature, type of oil, nutrients content, pace of biological decomposition, etc. [14]. The power of sorption of hydrocarbons in the sediment's changes depending on hydrocarbon's type, and organic matter content in the sediment [15].

Determination of bottom sediments requires observance of the following stages of the proceeding; preliminary processing of the samples of bottom sediment (drying, grinding), isolation and enrichment, which consists of transfer of the analyte from the primary to the secondary matrix, purification and fractionation of the extracted material and identification and final determination of the selected group of analytes [16].

The solvent should dissolve base oil and precipitate other substances to form sludge n-hexane, toluene, heptane, butane, hexanol and acetone were selected due to their good solubility parameters for base oil and good anti-solvent effect for nonpolar or slightly polar polymeric additives, metals and carbonaceous particles.

Variation of oil recovery with the use of various solvents, n-Hexane gave the highest recovery of 74.39% while acetone gave the lowest of 7.43%. The difference between them is related to the solubility of the base oil in these solvents. The performance of the extraction agents can also be related to the different interaction between the solvent and oil molecules, due to the size of the main carbon chain and the solvent molecules configuration [17].

The polarity of solvent is significant to the extraction efficiency in removing oil contaminants. Thus, a solvent has good dissolving ability when its polarity is similar to that of oil.

The recovery increased with increase in solvent carbon chain. This is mainly because carbon atoms lose hydrogen ions and act as a carrier medium for the oil molecules.

The extracting solvent must have two important properties; should be miscible with the base oil contained in the waste oil being processed and; must reject from the solution the used oil impurities allowing their aggregation to particle sizes big enough to separate from the liquid by sedimentation.

The addition of a base promotes a fast flocculation of the impurities, which are separated from the base oil by the solvent. The flocculation is enhanced in the presence of OH groups that neutralize the electrostatic repulsion. Furthermore, addition of a solution containing ions that neutralize those charges, break this stability. Participation of alcohol OH groups in ion exchange reactions with the electrolyte ions helps to neutralize the electrostatic repulsion.

Effective separation of thickening agent from primary base oil is mainly influenced by the properties of the degrading agent used such as solubility in water, concentration and temperature. Alkali-hydroxides are highly soluble in water compared to alkali-carbonates and they tend to have a strong affinity of hydrolysing grease molecules in suspension [17].

Acknowledge of the sediment content of crude oils and fuel oils are important both to the operation of refining and the buying or selling of these commodities.

The results listed in table 1; figure 1 shows flow chart of all results.

Table 2. Sediments in different samples

No.	Sample	Sediments %
1	Used turbine oil	3.15
2	Used lubricating oil	3.77
3	Khormala crude oil	0.592
4	Petroleum products dew	2.93
5	Used lubricating oil A2	1.636
6	Magnum 15W40 (used oil)	2.524
7	Magnum 15W40	2.71
8	Arya oil 15W40 (recycled)	6.319
9	Used lubricating oil (diesel cars)	0.319
10	Fuel oil	0.711

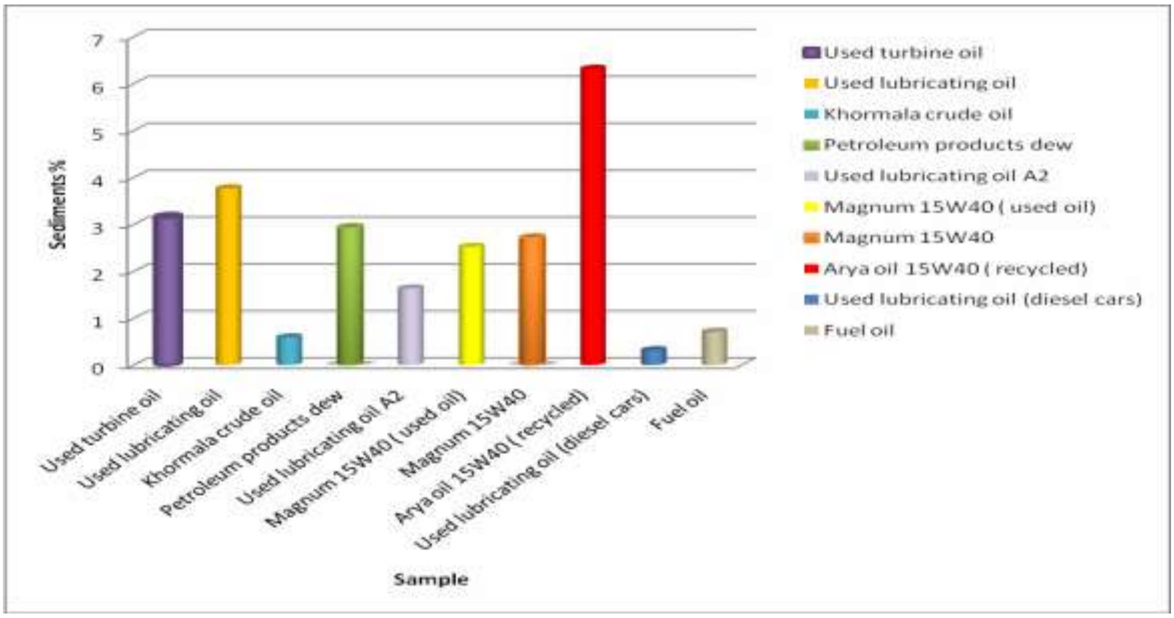

Figure 3. Flow chart of results

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Conclusion:
Sediments in different samples of crude oil, lubricating oil (virgin, used and recycled) and turbine oil from Koya city – Kurdistan region – Iraq local market was estimated by extraction. Results shows that sediments in some samples in a high level.

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