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Research Article

WASTE MINERAL OILS RE-REFINING WITH PHYSICOCHEMICAL METHODS

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ABSTRACT
Refining of waste mineral oils is usually done by distillation or acid/clay methods, or a combination of both. In this study, waste oil refining was carried out in order to obtain higher efficiency than acid /clay method by using more environmentally friendly physicochemical methods which could be an alternative to acid /clay method and distillation methods. For this purpose, demetalization and decolorization stages were applied in order. It was observed that the oils obtained had equivalent parameters to base oils.

Keywords: *Waste Mineral Oil, Recycling, Re-refining*

1. INTRODUCTION

Waste mineral oil is any industrial oil that has become unsuitable for the use to which it was initially assigned. These especially include used oils from combustion engines, transmission systems, turbines and hydraulic systems, the different sectors of the car industry and industrial shipping activities.

Mineral oils are petroleum origin and synthetic lubricants used in generators and other machines, especially in automotive engines. It is one of the most dangerous sources of pollution. Engine oil is not clean after draining from a motor because it collects dirt particles and other chemicals while the engine is running. Used, out-of-service lubricating oils are defined as waste oil, waste engine oil, waste lubricating oil or used lubricating oil depending on where it is used. The United States Environmental Protection Agency defines waste oils (used oils) as lubricating oils obtained from refinery or synthetic methods of crude oil contaminated with physical and chemical impurities (Zitte, 2016).

Used lube oil normally tends to have a high concentration of potentially harmful pollutant materials and heavy metals which could be dangerous to both living and non-living things on the earth. Used lube oil may cause damage to environment when dumped into ground or into water streams including sewers. This may result in ground water and soil contamination (Hopmans, 1974). Therefore, development of environmentally safe, sustainable and cost-effective solution is required for recycling of used lubricant (Stehlik, 2009). Nowadays due to different treatment and finishing methods, there are currently available many new Technologies (Bridjanian, 2006).

The direct effects that these types of oil can have on health include the following: Irritation of lung tissue due to the presence of gases that contain aldehydes, ketone, aromatic compounds, etc. The presence of chemical elements such as Cl (chlorine), NO_2 (nitrogen dioxide), H_2S (hydrogen sulphide), Sb (antimony), Cr (chrome), Ni (nickel), Cd (cadmium), and Cu (copper) that affect the upper respiratory tract and lung tissue. They produce asphyxiating effects that prevent oxygen transportation due to their content of carbon monoxide, halide solvents, hydrogen sulphide, etc. Carcinogenic effects on the prostate and lungs due to the presence of metals such as lead, cadmium, manganese, etc. (web 1). Direct effects on the environment that stand out include the following: The pollution of soils, rivers and the sea due to their low biodegradability. On coming into contact with water, they produce a film that prevents oxygen circulation. Uncontrolled combustion can lead to the emission of chlorine, lead and other gas elements into the atmosphere, with the corresponding effects (web 1).

It is also understood that used engine oil contains some components which mix with the oil when the engine is worn. These include iron, steel, copper, lead, zinc, barium, cadmium, sulfur, water and ash. Used waste engine oil contains hazardous pollutant chemicals, so it is more harmful to the environment than crude oil (Zitte, 2016), which can cause both short-term and long-term adverse environmental effects (Zitte, 2016). Only one ton of used waste oil makes one million tons of clean water unusable (US EPA, 1996). Even in sewage treatment, 50-100 ppm oil remains in water without disposal (US EPA, 1996). In general, it has been found that five liters of oil can cover a small lake (Zitte, 2016). The oil film formed on the water surface causes the BOD value to decrease and causes toxic effects. As a result, drinking water sources are polluted, dead plants and animals are observed (Zitte, 2016). Diran (1997) reported that oil in surface water seriously disrupts water's life support capacity. The oil prevents sunlight and oxygen from entering the water, making it difficult for fish to breathe and photosynthesis in aquatic plants. Thus, it kills plants and fish in aqueous environments. Toxic substances in used oil can also kill small organisms that support the rest of the food chain (Diran, 1997; Arner, 1992). Lubricant consumption is 5.3 million tons in Europe and 39 million tons in the world (Giovanna, 2003). The uses of oils are shown in Fig. 1 (Diphare, 2015).

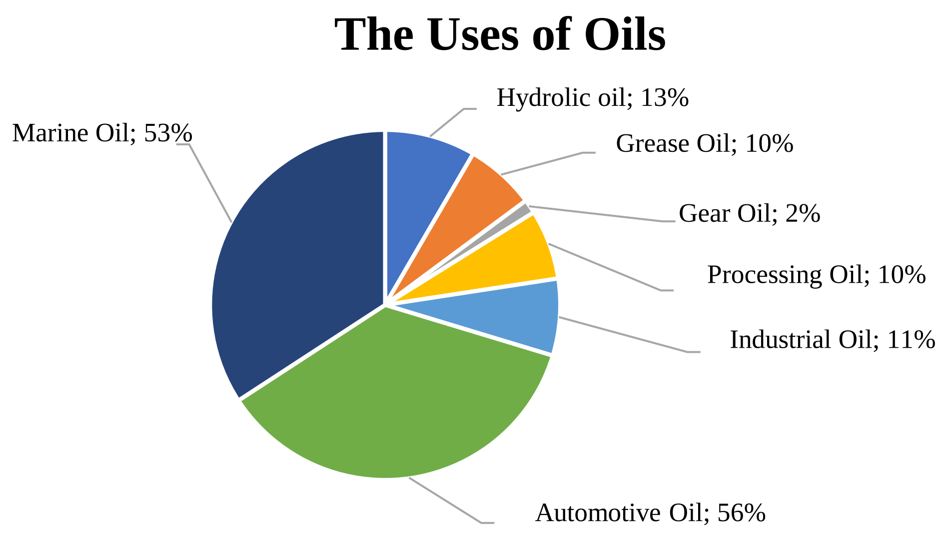

Fig. 1. Application areas of oils

After understanding the pollutant effects of waste oils, intensive studies have been carried out worldwide for the disposal and recovery of waste oils. The recovery and disposal of waste oils is possible by three methods (Fig. 2). These methods are reprocessing, re-refining or direct destruction (Diphare, 2015).

Reprocessing: It is the production of fuel by separating water and sediments in the waste oil. For this purpose, the water and contaminated components in the waste oils are removed by precipitation, adsorption and filtration; sometimes distillation and thermal cracking (pyrolysis) are applied before filtration.

Re-Refining: After some pretreatments, fractional distillation methods are applied to obtain base oil. Lubricating oils can be produced by adding additives to the base oils.

Disposal: Especially waste oils containing PCB (polychlorobenzenes) are disposed of directly by incineration without any pretreatment.

The refining of waste oils is possible by using highly advanced technology. In this study, by refining the waste oils with physicochemical methods, it is tried to determine the more economical alternative to distillation methods.

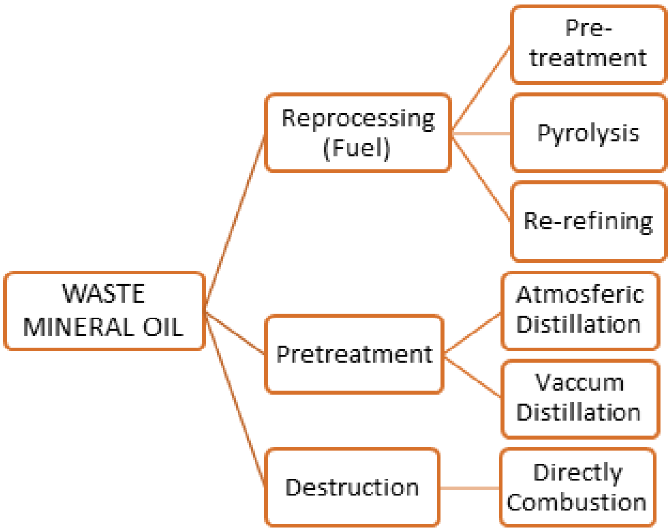

Fig. 2. Waste mine oil recovery and disposal methods

2. MATERYAL VE METOD

The waste oils used in the experiments were obtained from the auto services that change the oil. The viscosity measurements of waste oils and obtained oils were determined by Shanghai God NDJ-55 rotary viscometer, density measurements by Anton Paar DMA35 densimeter, flash point measurements by Teknosem Tan-400 device and color measurement by X-RITE SP 62 device. Metal measurements were determined by the Merlab GBC Avanta model atomic absorption device. Aluminum oxide (Al_2O_3), sodium silicate, tetraethylenepentamine (TEPA) used in the experiments were obtained from Merck.

Physicochemical refining stages of pre-analyzed waste mineral oils are as follows.

Pretreatment (Dewatering and Dematilization): It can be defined as dewatering step. To this end, 1 liter of waste oil was added to a 2 liter beaker, 10 mL of 20% diammonium phosphate solution was added and heated at 120°C for 120 minutes with careful stirring. The mixture was then allowed to cool. After 8 hours, a viscous mixture was formed as a precipitate at the bottom of beaker. The supernatant was carefully transferred to another beaker so that no precipitate was contaminated.

Chemical Refining: The supernatant transferred to another beaker was stirred with heating to 30°C. 1.5% ethylene glycol was added, stirring for 5 minutes to reduce the viscosity of the oil. Then 1% sodium silicate was added and stirred at 30°C for 10 minutes. Subsequently, 2% Al_2O_3 was added thereto and heating was continued with stirring to 50°C. 1% tetraethylenepentamine was added and stirred at constant temperature for 15 minutes. Subsequently, stirring was continued by careful addition of about 8-10% acid active bentonite to 160°C. Occasionally, the waste oil was sampled and dripped onto the filter paper to control the color. When the color was sufficiently lightened, the addition of bentonite was stopped and stirred for a further 10 minutes. The mixture was allowed to cool and filtered under vacuum at 60°C. The obtained oil was analyzed.

3. RESULT AND DISCUSSION

During the prolonged use of lubricating oils, black viscous structures are formed as a result of interactions between additives and metals in the oil, carbonation and polymerization reactions. Saturated hydrocarbons (30 to 70%), aromatic hydrocarbons polyaromatic (20 to 40%), polar compounds (5 to 25%) and asphalt based (0 to 10%). Waste oil also contains metal toxic metal pollutants (Pb, Hg, Zn, Cd, As and persistent organic pollutants PCB, PCT (Ouffoue, 2013; Boadu, 2019). In our country, waste oils are recovered according to the Regulation on Control of Waste Oils published by the Ministry of Environment (Resmî gazete, 2008). Although it is aimed to produce base oils from waste oils according to the regulation criteria, due to high refining plant costs and lack of technical knowledge, many recycling companies use the reprocessing method to produce fuel in existing plants. In the recovery of oils by reprocessing, catalytic decomposition or direct thermal decomposition at high temperatures is generally employed to obtain low viscosity fuel-equivalent products. However, the production of lubricating oil by adding some chemical additives to the base oils obtained by refining of waste oils according to the purpose of use is extremely important both in terms of commercial and national economy.

The recycling regulation applied in our country prohibits the production of fuel from waste oils. Heavy fines and even imprisonment are often applied to firms that produce fuel through the reprocessing method. Therefore, it is clearly known that companies with little experience in recycling are looking for more economical and practical methods to produce base oil from waste oils instead of fuel. The most common recovery methods of waste oils are as follows (Secretariat of the Stockholm Convention on Persistent Organic Pollutants, 2007):

Acid / Clay Method: In this process, after dehydration and distillation of used lubricant oil, re-refining or reprocessing operation is done using sulfuric acid. Clay is used to remove certain impurities. The acid/clay process has minimal environmental safety. The main by-product of this process is the large amounts of acidic sludge. Based on the concentration of contaminants, the type of lubricant oil and the regenerated oil quality, this process can be as a reprocessing or regenerative method. These two methods are different in terms of the heating rate (distillation unit) and the generated by-products. this method has many disadvantages: It also produces large quantity of pollutants, is unable to treat modern multigrade oils and it's difficult to remove asphaltic impurities. To reduce these hazardous contaminants from this method, the acid treatment stage of the process can be done under the atmospheric pressure to remove the acidic products, oxidized polar compounds, suspended particles and additives (Rahman *et al.*, 2008; Hani *et al.*, 2011; Hamawand *et al.*, 2013; Udonne *et al.*, 2013; Abu-Elella *et al.*, 2015).

Vacuum Distillation: In this method, used lube oil collected is heated at a temperature of 120°C to remove the water added to the oil during combustion. Then the dehydrated oil is subjected to vacuum distilled at a temperature of 240°C and pressure 20 mmHg. This results the production of a light fuel oil (the light fuel oil can be used as fuel source for heating) and lubricating oil at 240°C. The advantages of vacuum distillation process over atmospheric pressure distillation are: Columns can be operated at lower temperatures; more economical to separate high boiling point components under vacuum distillation; avoid degradation of properties of some species at high temperatures therefore thermally sensitive substances can be processed easily. However, the remaining oil generated at this temperature (240°C) contains the dirt, degraded additives, metal wear parts and combustion products like carbon and is collected as residue. The residue is in the form similar to that of tar, which can be used as a construction material, for example, road and bitumen production. The disadvantage of this method is the high investment cost and/or the use of toxic materials such as sulphuric acid (Havemann, 1978; Puerto-Ferre *et al.*, 1994; Kanna *et al.*, 2014).

Hydrogenation: To avoid formation of harmful products and environmental issues based on above methods, some modern processes have been used and the best one is hydrotreating (Bridjanian, 2006). This method follows vacuum distillation. In this process, the distillate from vacuum distillation is hydrotreated at high pressure and temperature in the presence of catalyst for the purpose of removing chlorine, sulphur, nitrogen and organic components. The treated hydrocarbons resulted in products of improved odour, chemical properties and colour Another important aspect of this method is that, this process has many advantages: Produces of high Viscosity Index lube oil with well oxidation resistance and a good stable colour and yet having low or no discards. At the same time, it consumes bad quality feed. In addition to that, this method has advantage that all of its hydrocarbon products have good applications and product recovery is high with no (or very low) disposals. Other hydrocarbon products are: In oil refinery the light–cuts can be used as fuel in plant itself. Gas oil may be consumed after being mixed with heating gas oil and the distillation residue can be blended with bitumen and consumed as paving asphalt, because it upgrades a lot its rheological properties. Also, it can be used as a concentrated anti-corrosion liquid coating, for vehicles frames (Durrani, 2014). The disadvantage of this method is that the residue resulting from the process is of high boiling range of hydrocarbon product fractionated into neutral oil products with varying viscosities which can also be used to blend lube oil.

Solvent de-asphalting process: This method has replaced acid-clay treatment as preferred method for improving oxidative stability and viscosity as well as temperature characteristics of base oils. Base oils obtained from Solvent Extraction are of good quality and contains less amounts of contaminants. In contrast to acid-clay treatment, it operates at higher pressures, requires skilled operating system and qualified personnel. The solvent selectively dissolves the undesired aromatic components (the extract), leaving desired saturated components, especially alkanes, as a separate phase (the raffinate) (Rincon *et al.*, 2005). Different solvents types have been used for solvent extraction such as 2-propanol, 1-butanol, methyl ethyl ketone (MEK), ethanol, toluene, acetone, propane etc. used propane as solvent (Quang *et al.*, 1974; Rincon *et al.*, 2003). He found out that propane was capable of dissolving paraffinic or waxy material and intermediately dissolved oxygenated material. Asphaltenes which contain heavy condensed aromatic compounds and particulate matter are insoluble in liquid propane. These properties make propane ideal for recycling the used engine oil, but there are many other issues that have to be considered. Propane is hazardous and flammable therefore this process is regarded as hazardous method. In general, involves solvent losses and highly operating maintenance. Also, it occurs at pressures higher than 10 atm and requires high pressure sealing systems which makes solvent extraction plants expensive to construct, operate and the method also produces remarkable amounts of hazardous by-products (Quang *et al.*, 1974; Rincon *et al.*, 2003; Rincon *et al.*, 2005; Hamawand *et al.*, 2013).

Modern Technologies for Used Oil Re-Refining: Pyrolytic distillation method, pyrolysis process, thin film evaporation (TFE), including combined TFE and clay finishing, TFE and solvent finishing, TFE and hydrofinishing, thermal de-asphalting and clay finishing or hydrofinishing etc. In addition, environmentally friendly and affordable solvent extraction and adsorbents are being developed as a means of removing contaminants in used lube oil. The thin film technique is an alternative to vacuum distillation process. (Kalnes, 1990, Hamed *et al.*, 2005).

Thin Film Evaporation (TFE) with hydro-finishing: These methods are utilized to segregate oil and foreign components via a TFE, and purify it through hydro-finishing to prevent the secondary pollution. First, the moisture and light oil contained in the used oil are eliminated and then vacuum distillation of free components is required to permit for continuous separation of a TFE. Finally, the oil is encountered to hydro-finishing to eliminate chlorine, nitrogen, oxygen, and sulfur compounds. There is a difference between these both methods, that clay is used for absorption (Kalnes, 1990).

TFE with solvent finishing: This method is used to segregate oil and foreign substances via a TFE, and request the solvent-finishing with the flow process analogous to TFE with hydro-finishing.

Solvent extraction hydro-finishing: This method combines solvent extraction and hydro-finishing by eliminating the foreign substances using the solvent and then fortifying oil quality by hydro-finishing. First, the moisture is eliminated and segregated the used oil. Then the mixture of solvent and used oil is encountered to hydro-finishing to eliminate sulfur, nitrogen and oxygen for purification purposes.

TDA with clay finishing and TDA with hydro-finishing: The dehydrated used oil is vacuum-heated at 360 ◦C. The ash remains at the bottom, and the oil is divided to 3 types, i.e. vacuum gas oil, base oil (as lubricant) and asphalt residues. Next, the base oil is encountered to hydro-finishing or clay-finishing under highpressure (107 Pa) for continuous utilization (Hamad *et al.*, 2005).

Pyrolysis of waste oil: From research conducted by Arpal (Arpal *et al.*, 2010), a fuel named as diesel-like fuel was produced by applying pyrolytic distillation method. Lam *et al.* (2012 and 2016), describe pyrolysis as a thermal process that heats and decomposes substance at high temperature (300-1000°C) in an inert environment without oxygen. Pyrolysis process is not yet widespread but it has been receiving much attentions nowadays due to its potential to produce energy-dense products from materials. Examples of pyrolysis process includes microwave pyrolysis process and conventional pyrolysis process (Oladimeji *et al.*, 2018).

Various process combinations can be formed by configuring atmospheric distillation, vacuum distillation, solvent extraction, hydrogenation, acid / clay methods in different ways. Generally the first stage in all processes is atmospheric distillation. The cost of the recycling processes consisting of combinations of atmospheric and vacuum distillation method, distillation and solvent extraction method is quite high. Advanced processes can be considered for high capacity plants. In the waste oil recycling regulations, Category III contaminated oils are allowed to be burned only in disposal plants such as cement factories, lime quarries and Category I and II waste oils are allowed to be recycled. (Resmi Gazete, 2008). Therefore, it is not possible for many licensed recycling plants to process high capacity waste oil. Due to high investment costs and high taxes, it is not feasible to establish small capacity processes.

In the oldest known acid / clay method in chemical refining, water and low boiling point components are first removed from the waste oil by atmospheric distillation. Subsequently, the waste oil is reacted with sulfuric acid at low temperature to remove carbons, asphaltenes and other sulfur compounds by precipitation from the oil, and then to be refined by treatment with various bleaching clays (Bhushan, 2016). The major problem in the acid /clay process is the removal of high concentrations of acid and sülfür, asphaltenes, and carbons in sediment, as well as a highly contaminated bleaching clay. Since the disposal process is very expensive and costly, waste oil recycling plants which were established primarily to prevent environmental pollution, cause greater environmental problems. Low process efficiency (60-65%) is also a disadvantage.

The method used in this study consists of a series of physicochemical processes in which base oils are obtained from waste oils, which are less costly, without the need for high process cost distillation. In this study, carbon and metallic components in the waste oil were precipitated together with asphaltenes using coagulatory agents such as aluminum sulfate and sodium silicate instead of acid. Sulfur components, one of the biggest problems in waste oils, were removed from the oil by interacting with amine groups. Thus, acid-free sediment removed from the waste oil can be used in asphalt production, will not pose an environmental risk, and there will be no disposal problems. Active bentonite (acid active bentonite) was used as clay. The purpose of using clay is to remove polymeric materials, colloidal materials remaining in the oil phase by adsorbing with clay.

During the physicochemical refining, asphaltenes and other impurities were precipitated in waste oil with the help of coagulants at low temperatures, while the double bonds formed in the high molecular weight oxidation molecule interacted with amines to ensure oxidation stability. Thus, more stable base oils were obtained from the distillation methods without the need for hydrogenation. 85% base oil was recovered by the new method used in this study, and the acid-free precipitate amount was 10-15%. However, in the acid / clay method, the yield is as low as 60-65% and the amount of sediment is higher than 25-35%. The analysis results of the waste oil used in the experiments and the base oil obtained from the experiments are given in Table 1 and Table 2. The results were also compared with 20W50 oil.

Table 1. Physicochemical properties of waste oils, refined oil and original oil

Parameter	Waste Motor Oil	Refined Oil	20W50
Colour (ASTM D1500)	>8	3,5	4.5
Density 15 C (ASTM D1298)	0,9253	0,8870	0,8700
Viscosity 40 C (ASTM D445), CST	106,37	118	91,0
Viscosity 100 C (ASTM D445), CST	12,66	11,02	10,5
Flash Point (ASTM D97), C	210	225	230
TAN (ASTM D2896), mg KOH/g	2,74	0	0
TBN (ASTM D2896), mg KOH/g	4,66	0	0
Sulfur	>8	4	2,5

Table 2. Analysis of metals in waste oil, refined oil and the original oil

Parameter	Waste Motor Oil ppm	Refined Oil	20W50
Fe	58,6	0	0
Cr	3,2	0	0
Pb	2813	1,86	0
Cu	21,4	2,7	0
Ca	1570	9,56	543
Zn	566	4	0

As it can be seen from the results, the waste motor oils were refined in two stages without the need of hydrogenation and advanced distillation techniques and base oil was obtained again. As in the acid / clay process, it does not generate environmental waste, the processing time is much shorter, the amount of bleaching clay usage is much less, the yield is higher. In Table 3, waste mineral oil recycling methods are compared in terms of process time, recovery cost, efficiency and process setup cost.

Table 3. Comparison of waste mineral oil recycling processes

Regenerative Technologies	Energy requirement	Recycling rate (%)	Quality of regenerative oil	Economic costs	Acidic sludge	Residual oil sludge	Hazardous chemical materials
Acid/clay	Low	63	Good	Low	Much	Much	H_2SO_4
Distillation	High	50	Good	Low	Little	Much	H_2SO_4
Solvent de-asphalinting	High	70-65	API	High	Little	Much	$H_2SO_4$and Organic solvent
TFE with hydrofinishing	High	72	API	High	None	Little	None
TFE with clay finishing	High	72	API	High	None	Little	None
TFE with solvent finishing	High	72	API	High	None	Little	Orgsmic solvent
Solvent extraction hydrofinishing	High	74	API	High	None	Little	Oganic solvent
Thermal de-asphalting	High	74-77	API	High	None	Little	None
Physicochemical Regeneration	Very low	85	Good	Very low	None	Little	No

As can be seen from Table 3, chemical regeneration is the most ideal recycling method of waste mineral oil given the low process cost and high efficiency. In distillation methods, thermal cracking is inevitable because waste oils are processed at high temperatures. In recycling models containing distillation, hydrogenation is essential to produce base oil with high efficiency Otherwise, low quality base oils containing unsaturated oil molecules are obtained in distillation methods that do not contain hydrogenation. Since the unsaturated oil molecules are precipitated together with asphaltenes in chemical refining process, high quality base oils are obtained without hydrogenation.

Since the gasification rate will be increased as a result of thermal decomposition in distillation methods which do not contain hydrogenation, the base oil ratio obtained will be low. Therefore, chemical regeneration is the more preferable method if it is aimed to obtain base oil as a result of waste oil recovery. If it is desired to obtain fuel, known distillation methods by thermal decomposition at high temperature may be used. However, the law does not allow the production of fuel from waste oils.

In our country, hydrogenation-based recovery method is not available due to high process cost. In other distillation-based methods, not only base oil but also fuel-equivalent products are obtained. The Ministry of Environment emphasizes the production of base oil instead of fuel equivalent product with various regulations and measures. The way to prevent fuel production is by applying chemical regeneration, which may be an alternative to distillation methods involving thermal decomposition.

In the chemical regeneration process, it is not possible to obtain fuel, on the contrary, base oil is obtained with high efficiency. The Ministry of Environment should take steps to promote the production of base oils, which have a much higher economic value than fuel, by chemical regeneration. Products that can be produced from recovered base oils (such as grease) are included in the incentive scope and directing the recovery companies to this area will prevent the production of illegal fuel.

As a result, the consumption of lubricating oils produced from limited oil resources as fuel is a major disadvantage for the national economy and the production of base oil from waste oils is very advantageous both for the national economy and for the more conscious use of oil resources. The method determined as a result of the experiments is a method that can be preferred because of low investment costs and no environmental risk without the need for advanced technology processes.

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