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Regeneration of waste hydraulic oils as a recycling method in relation to the environment

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Abstract: The article deals with the possibilities of regenerating operating fluids, assessing the composition of new, used, and regenerated oils by evaluating their toxicity and proposing the environmentally friendly regeneration method. The focus lies on two methods of regeneration of waste operating fluids: distillation and electrostatic cleaning. Oil samples, regenerated through these methods, were analyzed using gas chromatography with mass detection. The variance in composition among new, used, and regenerated oils depends on the method of regeneration. Properties of hydrocarbons exhibiting ecotoxic, mutagenic, teratogenic, carcinogenic, and other effects were identified using safety data sheets and databases like Pubchem, ChemicalBook. Analyzing HLP 46 oil (samples of new, unused, used and regenerated oil) revealed that the most toxic hydrocarbons (acetane, heptacosane, nonacosane) were absent after regeneration through electrostatic cleaning. Comparing the composition of operating fluids before and after regeneration, it was established that the most environmentally favorable regeneration method is electrostatic cleaning, which maintains the original properties of the operating fluids intended for use. Operating fluids regenerated via electrostatic cleaning contain fewer toxic hydrocarbons, making them more favorable concerning human health and the environment.

Introduction

Transport-mechanization devices utilize operating fluids for their proper operation (Kireš and Labuda 2017). These fluids encompass various types of oils, such as hydraulic, engine, transmission, transformer, turbine, and others. Based on the origin of the raw material used in their production, oils can be categorized as petroleum (mineral), synthetic, vegetable, animal, or mixed oils (www.tribotechnika.sk 2021).

Hydraulic mechanisms use compressed liquid as their working medium. These systems serve as a prevalent source for transferring energy to various industrial appliances, facilitating a wide array of technical movements (Dusa et al. 2018). One of their primary advantages is the high power-to-weight ratio and the compact dimensions upon installation. Hydraulic oil is designated for transmitting force necessary for operations within the hydraulic system. Its principal roles include lubrication, sealing, cooling, corrosion protection and ensuring compatibility with all hydraulic system components (Hnilicová 2015). The hydraulic fluid forms a lubricating film that effectively separates precisely manufactured parts.

The film's optimal length should sufficiently fill the gaps between these moving components (Dobrotová and Krilek 2015). However, the use of these operating fluids poses a risk, particularly to the environment. For example, in the event of an oil substance leakage into water, it can result in an oxygen deficit in aquatic environment, reduce the access of light to the depth, and prevent the process of photosynthesis. Such disruptions can lead to the eutrophication of aquatic ecosystems, a widespread global issue affecting rivers, lakes, seas, and even ocean ecosystems (Nowak et al. 2019). Moreover, oil pollution significantly alters the physical, chemical, and biological properties of soil. It diminishes water evaporation from the soil and increases the hydrophobicity of soil aggregates (Kout et al. 2022). The greasy coating restricts air circulation between the soil and the atmosphere, preventing the soil from "breathing" and hindering CO₂ exchange with the air (Curiel-Alegre et al. 2022). Petroleum substances act as herbicides on most plant species (Jeevanantham et al. 2019).

The disposal of used waste oil is commonly conducted through incineration. This process involves the reclamation of used oils and lubricants for energy purposes. Once specific

modifications, such as the elimination of water and mechanical impurities, are made, the oils are combusted, for instance, in rotary cement kilns (www.tribotechnika.sk 2014).

The current state of scientific research and technology enables the reutilization of used oils after their regeneration. This practice not only aligns with environmental protection (Hybská et al. 2017) but also supports the conservation of the raw materials utilized in production and proves economically advantageous in terms of cost savings.

Regeneration of used oils

Electrostatic cleaning

The continued use of worn and contaminated operating oils cause the failure of the used mechanisms and their hydraulic circuits in 85% of cases. The main cause is greasy oil and solid impurities, such as sawdust and metal powder, which prevent the performance of the oil's function. The oil cannot form a thin layer to ensure the smooth running of the machinery. Eliminating these impurities will extend the service life of the mechanisms and also reduce the electricity consumption (www.hirecofluid.sk 2020). Electrostatic liquid cleaning devices (Figure 1) use electrostatic forces, specifically following Coulomb's law, to attract polar liquids. As the liquid and oil pass through a set of anodes and cathodes, negatively charged particles pass to the positive part of the device, while positively charged particles migrate to the negative part. This process is referred to as electrophoresis. Non-polar particles are drawn through electrophoresis during the deformation of the electric field -. Collectors, strategically shaped inserts placed between the electrodes, attract individual particles due to electrostatic forces. The capacity (absorbability) of the collectors is high and can only be expressed in kilograms (Internal company document KLEENTEK s.r.o. 2022).

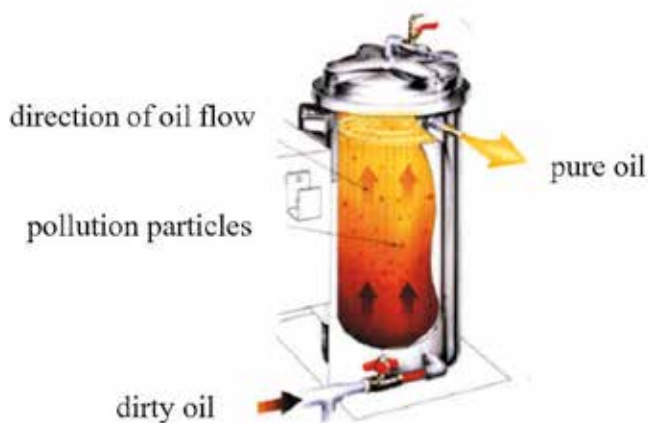


Figure 1. Principle of electrostatic cleaning (source: Internal company document KLEENTEK s.r.o. 2022)

The application of electrostatic cleaning in operating oils allows for the simultaneous removal of mechanical solid impurities (such as dust, abrasives, metals and other particles) within the size range of 2-100 μm . This process is effective in eliminating soft impurities linked to oxidative and thermo-oxidative stress, ranging in size from 0.01-2 μm , as well as ferromagnetic impurities across the entire spectrum of particle

sizes without adversely affecting the soluble additives in the oil. In instances of higher water content, electrostatic cleaning can be coupled with a dehydrator. Electrostatic cleaning equipment can retain and remove insoluble contaminants down to 0.01 μm , which include metallic and non-metallic impurities, dust, corrosion products, impurities caused by oxidation, such as carbon particles, deposits, varnishes, tar. The utilization of these cleaning devices present several advantages (Table 1): it enhances the performance of hydraulic components, mitigates malfunctions caused by clogging in pumps, motors, and valves, reduces operational costs, diminishes energy consumption, facilitates simple installation, displays the status of the device, eliminates the need for frequent oil changes, and ensures a rapid return on investment. Electrostatic cleaning equipment is widely applicable in many sectors, including manufacturing, mining, industry and for various types of oils (petroleum and white oils, biodegradable oils, polyalphaolefins (PAO), diester-based oils) and polyesters, as well as for glycol oils and liquids based on esters of phosphoric acid (www.hirecofluid.sk 2020; Internal company document KLEENTEK s.r.o. 2022; Hybská et al. 2017).

Item	Average cost reduction of
Oil costs	90 %
Filter costs	80 %
Production outages due to changing oils and filters	80 %
Labour costs when changing oils and filters	80 %
Cost of repair and replacement of components	70 %
Production stoppages due to machine breakdowns	56 %
Labour costs to eliminate faults	56 %
Non-conforming products - confusion	40 %
Energy for the drive and function of machines	5 %

Table 1. Benefits of predictive maintenance (KLEENTEK) (source: Internal company document KLEENTEK s.r.o. 2022)

Distillation

Liquid industrial wastes are primarily disposed of in incinerators. However, material recovery of waste is more acceptable for the environment.

The distillation of waste oils is one of several methods suitable for oil regeneration. This method, however, is a technically demanding process due to the complex nature of waste oils. These oils typically encompass a blend of hydrocarbons with a high boiling point, special additives, solid particles, and by-products from the oxidation of thermal decomposition of their components. Additionally, waste oils also contain water, acquired from various engine failures or during the collection of waste liquids.

Waste oils are introduced into the heated medium within the film distillation evaporator, where distillation takes place

using a thin liquid film. Driven by gravitational force, this liquid film flows down with vapor removal from the internal heated evaporation surfaces at a pressure of 0.1 kPa to 110 kPa and at a temperature of 80–330 °C.

The film distillation evaporator (Figure 2) is utilized for processing liquid wastes (whether under vacuum or atmospheric conditions) in combination with a distillation vapor cooler. This equipment comprises a vertically oriented block of hollow distillation body (5) featuring an internal evaporation surface (6). The heat necessary for the process is transferred from the heating block (4) to the external heated surface of the hollow distillation body. At the upper section of the distillation body (5), alongside the internal evaporation surface (6), a distributor is installed to facilitate the inflow of liquid waste onto the evaporation surface.

Steam extraction from forced evaporation surfaces is concurrent or counter current, depending on the direction of flow of the liquid film.

The advantage of using distillation with a film evaporator for the regeneration of waste oils is the efficiency and simple design of the device, suitable even for low-volume operation. This method minimizes the clogging of heat exchange surfaces, and cleaning these surfaces from deposits is easy. Additionally, during distillation, waste oils remain in the column for only a short time, they are not exposed to long-term high temperature, and thus a higher quality of the distillation product is achieved (Štibrányi et al. 2009).

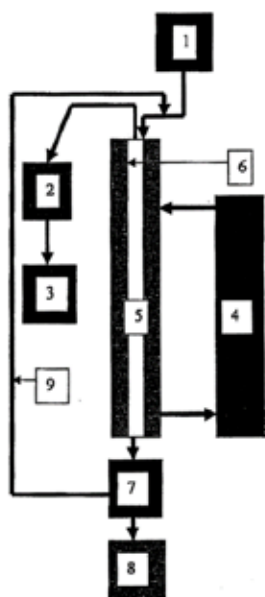


Figure 2. Film distillation evaporator for waste oils (Štibrányi et al. 2009) *Explanations:* 1 – waste oil tank, 2 – cooler, 3 – oil condenser at the outlet of distilled vapours with distilled oil tank, 4 – heating block, 5 – distillation body, 6 – internal evaporation surface, 7 – separator, 8 – distillation residue tank, 9 – oil recirculation circuit

Materials and Methods

Material

Hydraulic oil, made from a mixture of petroleum raffinates, was used for the experiment. The oil was analyzed: new, used and regenerated.

Determination of the composition of oils

All samples were dissolved in cyclohexane (purity 99.5%) before analysis. Device parameters and measurement conditions are listed in Table 2.

Device parameters GC-MS	
Device name	Shimadzu GCMS-TQ8040
Input device	autosampler AOC6000
Type of stationary phase	SLB-5MS
Measurement conditions GC-MS	
Column temperature	80 °C
Injection temperature	280 °C
Injection mode	Split (split, multi-program, divided)
Dividing ratio	10
Initial temperature	80 °C
Keep holding on	5 min.
Rate of temperature rise	10 °C/min.
Final temperature	300 °C
Keep holding on	15 min.
Total program time	42 min.
Method of measurement	TIC (total ion chromatogram) 33-55 m/z

Table 2. Device parameters and measurement conditions.

The identification of substances is based on the comparison of the mass spectrum of the samples with the spectra of substances from the NIST spectrum library. Supelco C7 - C40, Saturated Alkanes Standard was used to determine hydrocarbons.

Processing and evaluation of results

Excel and data from databases like Pubchem, ChemicalBook.

Results and discussion

According to the Waste Catalog (Slovak legislation - Decree No. 365/2015 Coll.), mineral hydraulic oil HLP 46 is included in subgroup 13 01 Waste hydraulic oils: 13 01 09 chlorinated mineral hydraulic oils, 13 01 10 non-chlorinated mineral hydraulic oils. Both new and used oil are classified as flammable liquid IV, posing a hazard, and have irritating effects on the respiratory tract, skin, and eyes.

Per Regulation (EC) no. 1272/2008 on the classification, labelling and packaging of substances and mixtures, operating fluids of petroleum (mineral) origin are categorized as flammable liquids of hazard category 3 if the highest permitted working temperature exceeds the flash point.

Analysis of the composition of operating fluids

Compounds listed in Table 3, were determined using gas chromatography with a gas spectrometer (GC – MS) method.

Hydrocarbon name	Chemical formula
octane	C ₈ H ₁₈
decane	C ₁₀ H ₂₂
octane, 2,3,3-trimethyl-	C ₁₁ H ₂₄
undecane	C ₁₁ H ₂₄
tridecane	C ₁₃ H ₂₈
hexadecane	C ₁₆ H ₃₄
pentadecane, 2,6,10,14-tetramethyl-	C ₁₉ H ₄₀
eicosane	C ₂₀ H ₄₂
heneicosane	C ₂₁ H ₄₄
heptacosane	C ₂₇ H ₅₆
nonacosane	C ₂₉ H ₆₀

Table 3. List of specified compounds in operating fluids.

Analyzed samples

Hydraulic oil HLP 46 serves as a pressure fluid for both industrial and mobile hydraulic systems. It is a high-performance hydraulic oil made from high-quality petroleum raffinates. Due to the high thermal stability of the base oils contained in the hydraulic oil, the aging process is significantly reduced, especially at elevated temperatures. This significantly contributes to lower sludge formation, improved cleanliness, and reliability of the hydraulic system. The outstanding

New oil	Used oil	Oil after regeneration
pentadecane, 2,6,10,14-tetramethyl-(pristane)	pentadecane, 2,6,10,14-tetramethyl-(pristane)	pentadecane, 2,6,10,14-tetramethyl-(pristane)
undecane	nonacosane	undecane
eicosane	octane	eicosane
octane, 2,3,3-trimethyl-	undecane	heneicosane
heneicosane	heptacosane	octane, 2,3,3-trimethyl
	eicosane	
	tridecane	
	hexadecane	
	octane, 2,3,3-trimethyl-	
	heneicosane	

Table 4. Composition of hydraulic oil HLP 46.

resistance to oxidation ensures an extended service life (change time) of the oil. Excellent wear protection technology works under both low and high loads. The oil provides reliable corrosion protection even in the presence of water. Hydraulic components are optimally protected under all operating conditions (Tatralube HYD HLP 46 safety data sheet).

Samples regenerated by electrostatic cleaning.

The composition of hydraulic oil HLP 46 regenerated by electrostatic cleaning (ELC) confirms the effective removal of hydrocarbons formed during the oil operation. This process effectively eliminated the identified hydrocarbons (tridecane, hexadecane, eicosane, heneicosane, octane, 2,3,3-trimethyl) from the used oil, while also maintaining the original composition of oil (Table 4). According to information obtained from the operator of the equipment, the original ingredients and components were preserved, as in the case of a new oil sample. .

Samples regenerated by distillation.

In samples of regenerated hydraulic oil other hydrocarbons were also determined, which the original new oil did not contain. Only some hydrocarbons were removed through the distillation of used oils (Table 5).

New oil	Used oil	Oil after regeneration
pentadecane, 2,6,10,14-tetramethyl-(pristane)	pentadecane, 2,6,10,14-tetramethyl-(pristane)	pentadecane, 2,6,10,14-tetramethyl-(pristane)
undecane	nonacosane	decane
eicosane	octane	heptacosane
octane, 2,3,3-trimethyl-	undecane	undecane
heneicosane	heptacosane	octane, 2,3,3-trimethyl-
	eicosane	heneicosane
	tridecane	eicosane
	hexadecane	
	octane, 2,3,3-trimethyl-	
	heneicosane	
	decane	

Table 5. Composition of hydraulic oil HLP 46.

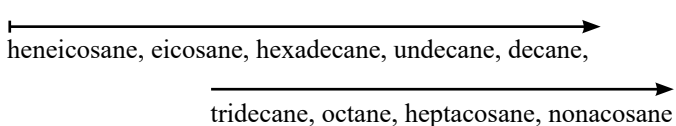
Toxicity assessment

The oil contains hydrocarbons that exhibit ecotoxic, mutagenic, teratogenic, and carcinogenic effects at various concentrations (Table 6). The data are derived from safety data sheets, Pubchem, ChemicalBook, and toxicity publications. Out of the 11 hydrocarbons determined by the analysis, we did not find any information about the toxicity of one specific hydrocarbon (octane, 2,3,3-trimethyl).

Hydrocarbon	Test type	Monitored parameter	Test organism	Concentration
Octane C₈H₁₈ [22]	Toxicity to fish	LC ₅₀	<i>Oryzias latipes</i>	0.42 mg/l
	Toxicity to daphnia and other aquatic invertebrates static test	EC ₅₀	<i>Daphnia magna</i>	0.38 mg/l
	Toxicity to algae growth inhibition NOEC (no detectable effect concentration)	NOEC	<i>Pseudokirchneriella subcapitata</i>	5.8 mg/l
	Toxicity to fish (chronic toxicity) LOEC	LOEC	<i>Oryzias latipes</i>	0.069 mg/l
	Fish toxicity (chronic toxicity) NOEC (no detectable effect concentration)	NOEC	<i>Oryzias latipes</i>	0.028 mg/l
	Toxicity to daphnia and other aquatic invertebrates (Chronic toxicity) static test NOEC (concentration with no detectable effect)	NOEC	<i>Daphnia magna</i>	0.17 mg/l
Decane C₁₀H₂₂ [3]	Toxicity to fish semi-static test	LC ₅₀	<i>Oncorhynchus mykiss</i>	>1000 mg/l
	Toxicity to daphnia and other aquatic invertebrates static test	EC ₅₀	<i>Daphnia magna</i>	>1000 mg/l
	Toxicity to algae static test	EC ₅₀	<i>Pseudokirchneriella subcapitata</i>	>1000 mg/l
Undecane C₁₁H₂₄ [4]	Toxicity to fish semi-static test	LC ₅₀	<i>Oncorhynchus mykiss</i>	>1000 mg/l
Hexadecane C₁₆H₃₄ [5]	Toxicity to fish semi-static test	LL ₅₀	Fish	1028 mg/l
	Toxicity to algae	EL ₅₀	<i>Pseudokirchneriella subcapitata</i>	>3200 mg/l
Heptadecane C₁₇H₃₆ [6]	Acute toxicity - oral	LD ₅₀	Rat	> 5000 mg/kg
	Acute toxicity - inhalation	LD ₅₀	Rat	> 5266 mg/m ³
	Acute toxicity - dermal	LD ₅₀	Rabbit	> 3160 mg/kg
Eicosane C₂₀H₄₂ [24]	Acute toxicity - oral	LD ₅₀	Rat	> 5000 mg/kg
	Acute toxicity - inhalation	LC ₅₀	Rat	5266 mg/m ³
	Acute toxicity - dermal	LD ₅₀	Rabbit	> 3160 mg/kg
Heneicosane C₂₁H₄₄ [23]	Inhalation toxicity test	LC ₅₀	<i>Aedes aegypti</i>	5 g/cm ³
Heptacosane C₂₇H₅₆ [20]	Toxicity test	LD ₅₀	<i>Plutella xylostella</i>	1.30 µg/larva
			<i>Spodoptera exigua</i>	2.16 µg/larva
			<i>Spodoptera litura</i>	3.35 µg/larva
Nonacosane C₂₉H₆₀ [28]	Toxicity test	LD ₅₀	<i>Spodoptera litura</i>	2.70 µg/larva
			<i>Spodoptera exigua</i>	3.54 µg/larva
			<i>Plutella xylostella</i>	0.72 µg/larva

Table 6. Hydrocarbons and their toxicity.

Based on the toxicological data from Table 6, hydrocarbon toxicity increases in the direction of the arrow:



 heneicosane, eicosane, hexadecane, undecane, decane,
 tridecane, octane, heptacosane, nonacosane

In studies by Rim et al. (2004), the toxicity of n-decane was tested in rats. The lethal dose (LD₅₀) was established at 72,300 mg/m³ in 2 hours. N-decane was confirmed as a carcinogen. During the 28-day experiment, the test organisms were exposed to different doses of N-decane through inhalation: 47 ppm (the

low dose), 201.8 ppm (the medium dose) and 800 ppm (the high dose). Although no significant organ changes were observed as a result of inhaling N-decane, all three test groups of organisms experienced hair loss due to exposure to the substance.

Eicosane exhibits carcinogenic effects and is classified among substances that disrupt the endocrine system (EDC). Hydrocarbons are toxic compounds that can inhibit microbial processes. They are methanogens. In a study conducted by (Morais et al. 2021), the toxicity of hexadecane was examined at concentrations of 0–30 mM. The study confirmed its toxic impact on cultures of hydrogenotrophic methanogens (*Methanobacterium formicicum* and *Methanospirillum hungatei*).

Hydrocarbon	New	used	Regenerated	
			electrostatically	by distillation
octane		+		
decane		+		+
octane 2,3,3-trimethyl-	+	+	+	+
undecane	+	+	+	+
tridecane		+		
hexadecane		+		
pentadecane 2,6,10,14-tetramethyl-	+	+	+	+
eicosane	+	+	+	+
heneicosane	+	+	+	+
heptacosane		+		+
nonacosane		+		

Explanation: + the oil contains the specified hydrocarbon

Table 7. Overview of the presence of hydrocarbons in hydraulic oil HLP 46 samples.

Exposure to n-octane in test organisms (rats) resulted in acute toxicity, manifested by temporary lethargy, which was ceased once inhalation stopped. The LC_{50} for acute inhalation toxicity of n-octane was determined to exceed 23.36 mg/l. Rats subjected to repeated exposure to n-octane showed no significant clinical and histopathological changes compared to control test species (Sung et al. 2010).

Alkanes are available carbon sources for *Acinetobacter baylyi*. In a study by (Li et al. 2021), it was confirmed that although hexadecane exhibits genotoxic effects, it did not affect the metabolic activities of this bacterial species.

The mutagenic activity of undecane was evaluated in a bacterial reverse mutation test on *Salmonella typhimurium* and *Escherichia coli* strains. The study, conducted by (Api et al. 2020), involved treating these bacterial strains with undecane in acetone at concentrations up to 5000 $\mu\text{g}/\text{plate}$. The findings revealed no significant increase in the mean number of revertant colonies at any concentration tested. Thus, the study did not demonstrate mutagenic effects of undecane under the conditions of the Ames test.

In the study conducted by Ajayi et al. in 2018, the research focused on examining the contact toxicity and fumigation effects of tridecane. Separately, Poonsri et al. in 2015 determined the lethal dose (LD_{50}) of heptacosane for *Plutella xylostella* larvae, establishing it at 1.58 μg per larva following 48 hours of exposure.

Junhirun et al. (2018) determined a lethal dose (LD_{50}) of nonacosane of 2.7 μg for the species *Spodoptera litura*, 3.54 μg for the species *Spodoptera exigua*, and 0.72 μg for *Plutella xylostella* after 24 hours of exposure. In the case of heptacosane, LD_{50} of 3.35 μg was determined for the species *Spodoptera litura*, 2.16 μg for the species *Spodoptera exigua* and 1.30 μg for *Plutella xylostella* after 24 hours of exposure.

Studies conducted by (Dahlgren et al. 2007. Nurdiana et al. 2019) observed a prevalence of autoimmune disease or symptoms associated with autoimmune disorders in residents living in the vicinity of oil fields where pristane (Pentadecane. 2.6.10.14-tetramethyl-) was found in their blood. These studies suggest a potential link between pristane exposure and the development of autoimmune diseases, notably systemic Lupus erythematosus (SLE). Animal models of pristane intoxication further support its consideration as a casual factor in SLE disease development.

The most common symptoms of overexposure to mineral oil in mammals are headache, blurred vision, dizziness, feeling drunk, vomiting, confusion, fever, and general malaise. Exposure levels of 500-1000 ppm for 30-60 minutes can lead to a euphoric state followed by ataxia (problems with balance and dexterity in movement), drowsiness and dizziness. Higher exposure levels (1000-3000 ppm) can result in irritation, headache, nausea, and vomiting, as well as eye, nose, and throat irritation. Exposure to levels exceeding 5000 ppm can result in dizziness, deep anesthesia, pulmonary edema, and death within minutes (Harbison et al. 2015).

According to Table 7, which shows the frequency of individual hydrocarbons in samples of new (unused), used and regenerated hydraulic oil HLP 46, it was observed that the most toxic hydrocarbons (octane, heptacosane, nonacosane) were not present in the regenerated oil samples.

Conclusion

Based on the results obtained from the analysis of the composition of operating fluids (new, used and regenerated) and after the regeneration of these samples by two methods (electrostatic cleaning, distillation), we found that electrostatic cleaning stands out as the most environmentally favorable

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