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Technological Bases for the Production of Plastic Greases from Alternative Raw Materials

A. Hryhorov[1], E. Kurtyosov[2], D. Usachov[2], O. Kozhemiaka[2], Y. Makarov[2], M. Dement[2]

[1] National Technical University «Kharkiv Polytechnic Institute», 61002, 2 Kirpichova str., Kharkiv, Ukraine

[2] National University of Civil Defense of Ukraine, 18034, 8 Onoprienka street, Cherkassy, Ukraine

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Abstract

The article discusses the prospects of obtaining plastic greases based on alternative raw materials available in Ukraine – production and consumption waste. It is proposed to use waste motor oils or high-boiling fractions of oil sludge, compounded at temperatures of 120-150°C with polymer wax – a product of thermal destruction of HDPE, PP at temperatures of 400-410 °C and a pressure of 0.11-0.13 MPa, to obtain greases that are analogues of Total Multis EP 0/1/2; Shell Retinax A; Mobilgrease XHP 100; Castrol Spheerol EPL 0/1/2; Klüber STABURAGS NBU 4; Divinol Fett 0/1/2. The research conducted has made it possible to propose a combined scheme for the production of PG from alternative raw materials, which additionally allows the production of motor and boiler fuels, as well as adsorbents for the treatment of industrial wastewater.

Keywords: *Plastic greases; Waste oils; Oil sludge; Polymers; Fractions; Thermal destruction; Thermal compounding; Fuels; Adsorbent.*

1. Introduction

Current trends in the development of Ukraine's oil refining and petrochemical industry aim to achieve a balance between the quality of commercial petroleum products, particularly greases, and the cost of their production. The cost of grease production largely depends on the cost of raw materials, their availability and manufacturability, and also determines their competitiveness in both the domestic and global plastic grease markets. Therefore, the search for alternative raw materials to replace the traditional materials currently used for greases in Ukraine is one of the most pressing tasks today.

2. The objective of the research

In Ukraine, traditional petrochemical components are used for the production of greases for various functional purposes, which, in turn, can be explained by the factors presented in Fig. 1. We will analyze the significance and contribution of each of the factors formulated and presented in Fig. 1.

Established technological base. Ukraine has developed production of anti-friction and protective greases based on mineral oil, petroleum derivatives, and soap thickeners [1-2]. The component composition of such greases can be represented by the following general formulas:

$$PG = BO + T + A \quad (1)$$

$$PG = BO + T + F + A + D \quad (2)$$

where BO – base oil; T – thickener; A – additives; F – filler; D – dye.

The technological equipment of existing enterprises - mixing units and reactors [3-4] – is designed specifically for traditional recipes. At the same time, modernization of technologies requires significant investments.

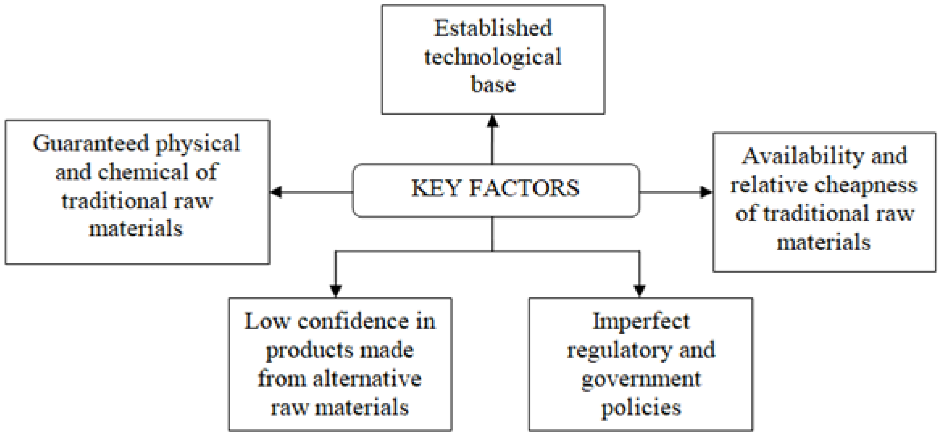

Figure 1. Factors influencing the use of traditional raw materials for the production of greases in Ukraine

Availability and relative cheapness of traditional raw materials. The mineral oil used in the production of greases is obtained from primary oil refineries, followed by deasphalting and dewaxing. Such oil contains up to 0.03% by weight of sulfur, has a kinematic viscosity at 100°C of 7.5-8.5 mm^2/s, and a viscosity index of 90-120 units [5]. Compared to synthetic oil, for example, it is less expensive and more accessible for grease production.

The main types of thickeners for mineral base oil are alkali soaps – metal salts of fatty acids, where the cation is an alkali or alkaline earth metal. Such soaps form a three-dimensional network that retains the base oil and imparts a certain consistency to the grease. The main types of alkali soaps used to thicken greases are shown in Table 1.

Table 1. Types of alkali soaps used for thickening greases [6-7].

Type of soap	Cation	Substance
Calcium soap	Ca^{2+}	Calcium stearate
Lithium soap	Li^{+}	Lithium stearate
Aluminum soap	Al^{3+}	Aluminum stearate
Sodium soap	Na^{+}	Sodium salt of fatty acid

Alkaline soaps require affordable, relatively inexpensive fatty acids (stearic acid, technical palmitic acid, or animal fat) and alkalis ($Ca(OH)_2$, NaOH, or LiOH) for their production, which react at 100–150°C.

Additives to grease are active additives that are injected into the grease base (base oil + thickener) to improve the operational properties of the grease [8]: anti-oxidant, anti-wear, anti-corrosion, adhesion. These additives were considered affordable because most of them were produced at enterprises in the petrochemical industry of Ukraine.

Fillers for greases are solid or semi-solid additives that improve the grease's physical and chemical properties [9], reduce its cost, or provide special functions (e.g., heat resistance, adhesion, anti-friction properties). The main types of fillers and their sources are presented in Table 2.

Table 2. Types of fillers used in greases.

Filler type	Function	Source of origin
Kaolin	Improves thermal stability and homogeneity	Zhytomyr region
Bentonite	Structuring, stability	Volyn, Zakarpattia
Talc	Reduces friction, improves gliding	Import / Carpathians
Clay	Cheap filler	All regions of Ukraine
Chalk ($CaCO_3$)	Filling, reducing cost	Mykolaiv, Kharkiv

Guaranteed physical and chemical properties. State standards (DSTU) and technical specifications of Ukraine (TU U) are focused on traditional components (base oil + alkali soaps). The majority of consumers (especially state-owned enterprises) require compliance with old standards, where alternative raw materials do not yet have legalized characteristics.

Low confidence in products made from alternative raw materials. There is a stereotype on the market that greases made from secondary raw materials are poor-quality, unstable materials. Without certification and technical testing, many consumers avoid using them.

Imperfect regulatory and government policy. Ukraine lacks updated national standards (DSTU) for greases based on alternative/secondary resources. Bureaucratic difficulties with legalizing new types of greases based on alternative raw materials are holding back manufacturers. It should also be noted that without official approval, new greases is not purchased by state-owned companies.

However, in the current geopolitical situation surrounding Ukraine, most of the raw materials that were previously widely used in the production of greases are now in short supply. This was facilitated by both insufficient domestic hydrocarbon reserves [10] and a significant reduction in the industrial capacity of Ukraine's oil refining and petrochemical industries [11]. Under such conditions, domestic production volumes of greases are unable to meet existing demand, although more and more imported raw materials are being used for their production.

Therefore, against the backdrop of rising global oil prices, a shortage of alkali metals, the development of a circular economy, and environmental legislation in Ukraine and the EU, the production of greases from alternative raw materials, in particular secondary raw materials, is becoming a relevant strategic direction that needs to be developed.

The main prospects for the development of greases production technologies based on secondary raw materials in Ukraine can be presented in the form of a structural diagram (see Fig. 2).

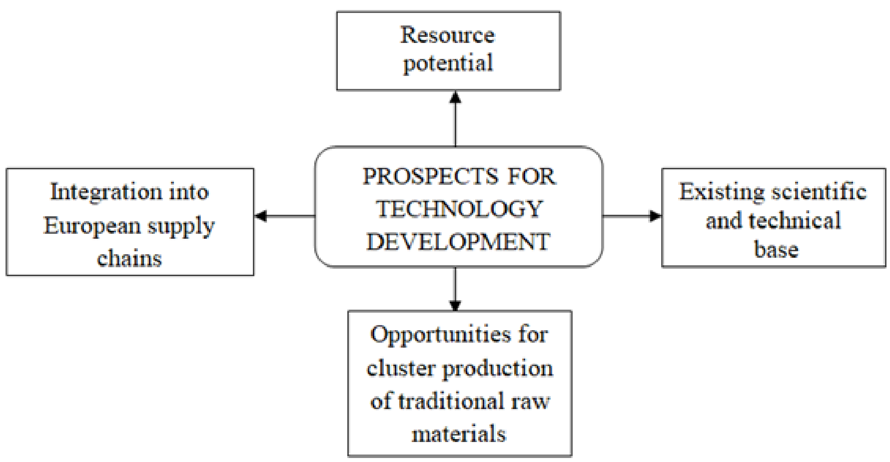

Figure 2. Key prospects for the development of greases production technologies based on secondary raw materials in Ukraine.

Considering the prospects for the development of greases production technologies based on secondary raw materials as a dispersion medium with a hydrocarbon composition similar to that of traditional mineral oils, the following should be used: used hydraulic, motor, turbine, and process oils; heavy hydrocarbon fractions obtained from the processing of production waste, such as oil sludge [12]. The use of waste oils, in particular motor oils, has a significant advantage over fractions obtained from oil sludge, which is the presence of residual additive potential [13] – antioxidants, anti-wear agents, etc. It is known that this potential can average 30-50 % of the initial value, which allows for a reduction in the use of additional quantities in greases.

Polymer waste, represented by polyethylene (LDPE, HDPE) and polypropylene (PP) products that have lost their consumer properties, can be used quite successfully as the main thickeners for such base oils. These solid polymers can be used in greases after preliminary preparation (grinding, washing, and drying) [14] or in the form of polymer wax after thermal destruction of the source polymers. Polyolefin thickeners are characterized by compatibility with various types of base oils, which manifests itself in the stability of the resulting greases during their operation and long-term storage.

Therefore, the practical application of greases made from alternative raw materials instead of anti-friction and protective greases obtained from traditional raw materials requires a more detailed study of the properties that can ensure the basic functions of greases. The purpose of this article is to establish the ranges of physical and chemical quality indicators that characterize the properties of greases made from alternative raw materials. The stated research objective is achieved through the following tasks: selection of mutually compatible raw material components, preliminary preparation of components and their technological processing into grease samples, laboratory testing of the obtained grease samples.

3. Materials and methods

Materials. The base oil for obtaining greases was used motor oil of viscosity class SAE10W-40 API SL, SAE15W-40 API CI-4/E7; hydrocarbon fraction of oil sludge (with a boiling point above 360(380) °C) selected from a storage pond at an oil refinery in the Kharkiv region, Ukraine. Pre-prepared polymer products represented by HDPE (grade 50100, manufacturer Borealis, Austria) and PP (grade H030, manufacturer Karpatnaftohim, Ukraine) were used as polymer thickeners.

Methods. The main methods of preliminary preparation of the dispersion medium – used motor oil – were settling for 3 hours at a temperature of 90-100°C. With this preparation, the residual content of water and mechanical impurities (up to 10 μm in size) in the oil was < 0.01 % and < 0.15 %, respectively. The high-boiling hydrocarbon fraction of oil sludge was obtained by settling its hydrocarbon part for 3 hours at 110°C, followed by distillation according to ASTM D86. Polymer wax was obtained by thermal destruction in a laboratory reactor at a temperature of 400-410°C and a pressure of 0.11-0.13 MPa. From the liquid products of destruction obtained, the fraction with a boiling point above 340°C was removed by distillation (using the ASTM D86 method). The greases samples were obtained by dispersing the thickener in a dispersion medium at temperatures of 130-160°C using a mechanical stirring device (speed 150-170 rpm) for 2 hours. The mixture obtained in this way was homogenized by pressing it through holes with a diameter of 500 μm under a pressure of 15 MPa. The research methods used to determine the physicochemical quality indicators of greases included: drop point (ASTM D566); penetration (ASTM D 1403-10(E)); effective dynamic viscosity (ASTM D 1092); colloidal stability (ASTM D 1742); volatility (ASTM D972,); lubricating properties (DIN 5151 350/4); water resistance (DIN 51 807/1); corrosion impact on metals (ASTM D4048); corrosion effects on metals (D1743); tribological characteristics using Four-Ball Wear Test (ASTM D 2266), emission spectral analysis implemented on the MFS-11 installation.

4. Results and discussion

The main process of greases production from alternative raw materials is the thermal dispersion of the dispersed phase – the thickener in the dispersion medium – the base waste oil. This process is quite complex because a physicochemical interaction occurs between the components (disperse phase and dispersion medium), which forms the structure and properties of greases. Polymer waxes obtained by the destruction of HDPE/PP are well compatible with waste motor oils due to their common nonpolar hydrocarbon nature [15]. Their interaction can be used to create budget greases. The mechanism of interaction between components in the production of greases from waste oil and polymer wax is presented in Table 3.

Table 3. Mechanisms of interaction between the dispersion medium and the dispersed phase for greases based on waste oil and polymer wax.

Type of interaction	The process	Result
Physical compatibility	The dispersed phase partially dissolves in the dispersion medium	Formation of a homogeneous or gel-like mixture
Solubilization	Dispersion of the dispersed phase by the volume of the dispersion medium	Creation of a heat-resistant gel or paste-like product
Polar interaction (partial)	Oxidized fragments of the dispersion medium interact with the terminal groups of destroyed polymers	Improving system stability
Combination of mechanical mixing and heat treatment	When heated to 120–150°C, complete dispersion occurs	Creating a base for grease

The interaction between the high-boiling, heavy fraction separated from oil sludge by distillation is somewhat different. Typically, this fraction has a boiling point in the range of 360(380)–500 °C and contains C_{20}–C_{50} hydrocarbons, resins, asphaltenes, and acids, which characterize its high polarity [16]. The mechanism of interaction between components in the production of greases from petroleum sludge fraction and polymer wax is presented in Table 4.

Table 4. Mechanisms of interaction between the dispersion medium and the dispersed phase for greases based on oil sludge fraction and polymer wax.

Mechanism	The process	Result
Physical compatibility (partial)	Partial dissolution of the dispersed phase in the dispersion medium	Homogeneous or gel-like system
Solvation of asphaltenes	The dispersed phase acts as a stabilizer for resinous particles	Fraction stabilization, sediment reduction
Thermoplastic dispersion	The dispersed phase structures the dispersion medium	Formation of plastic mass
Film formation	When cooled, a gel/ grease is formed	Increase in density

Our theoretical propositions regarding the interaction between components that leads to the formation of a new product – greases – were confirmed by experimental research. Studies of grease samples obtained from alternative raw materials allowed us to determine the ranges of their physicochemical quality indicators, which are presented in Tables 5–8.

Table 5. Intervals of physical and chemical quality indicators of greases based on used motor oils and polymer wax from HDPE 50100.

Indicator name	Units	The value of the indicator
Base oil	-	SAE10W-40, SAE15W-40
Polymer wax content	%	25.0-50.0
Consistency according to NLGI	Grade	0/1/2
Drop point	°C	65-105
Penetration at 25 °C	$mm \times 10^{-1}$	350-225
Water resistance	-	Stable
Corrosive effect on metal	-	Withstands (steel)
Density at 15.5 °C	kg/cm^3	810-890
Separation of oil (ASTM D 1742)	%	7-15
Friction indicator, 1450 rpm/800 H/1 hour	mm	0.50-0.75

Table 6. Intervals of physical and chemical quality indicators of greases based on used motor oils and polymer wax from PP H030.

Indicator name	Units	The value of the indicator
Base oil	-	SAE10W-40, SAE15W-40
Polymer wax content	%	25.0-50.0
Consistency according to NLGI	Grade	00/0/1
Drop point	°C	55-75
Penetration at 25 °C	mm×10^{-1}	450-330
Water resistance	-	Stable
Corrosive effect on metal	-	Withstands (steel)
Density at 15.5 °C	kg/cm^3	800-870
Separation of oil (ASTM D 1742)	%	10-18
Friction indicator, 1450 rpm/800 H/1 hour	mm	0.55-0.85

Table 7. Intervals of physical and chemical quality indicators of greases based on oil sludge fraction and polymer wax from HDPE 50100.

Indicator name	Units	The value of the indicator
Base oil	-	Oil sludge fraction (t_{boil} > 340°C)
Polymer wax content	%	25.0-50.0
Consistency according to NLGI	Grade	4/5/6
Drop point	°C	85-120
Penetration at 25 °C	mm×10^{-1}	180-90
Water resistance	-	Stable
Corrosive effect on metal	-	Withstands (steel)
Density at 15.5 °C	kg/cm^3	890-903
Separation of oil (ASTM D 1742)	%	5.0-9.0
Friction indicator, 1450 rpm/800 H/1 hour	mm	0.88-1.00

Table 8. Intervals of physical and chemical quality indicators of greases based on petroleum sludge fraction and polymer wax from PP H030.

Indicator name	Units	The value of the indicator
Base oil	-	SAE10W-40, SAE15W-40
Polymer wax content	%	25,0-50,0
Consistency according to NLGI	Grade	2/3/4
Drop point	°C	75-110
Penetration at 25 °C	mm×10^{-1}	190-270
Water resistance	-	Stable
Corrosive effect on metal	-	Withstands (steel)
Density at 15.5 °C	kg/cm^3	880-891
Separation of oil (ASTM D 1742)	%	6.0-10.5
Friction indicator, 1450 rpm/800 H/1 hour)	mm	0.95-1.15

Analyzing the information presented in Tables 5-8, we note that greases obtained on the basis of used motor oils and HDPE 50100 polymer wax, PR H030, due to the content of active additive elements (Zn – 835 ppm, P – 740 ppm, B – 150 ppm, Ca – 2200 ppm, Mg – 170 ppm, S – 3500 ppm), exhibit improved anti-wear properties, as indicated by the friction indicator (0.50-0.85 mm). It is known that the presence of Zn and P indicates the residue of anti-wear additives, B – the residue of anti-oxidant and anti-wear additives, Ca and Mg – the residue of detergent and dispersant additives, S – the residue of anti-seize additives [17-19]. In terms of drop point, which determines the upper temperature limit of grease performance, such greases are analogous to simple, inexpensive, or universal greases based on calcium thickeners or naphthenic soaps and mineral oils. These include the greases listed in Table 9.

Table 9. Foreign analogues of greases with a drop point of up to 100 °C.

Grease name	Country / Brand	Thickener type	Drop point	The area of application
Total Multis EP 0/1/2	Total (France)	Calcium soap	~90–95 °C	Universal, for moderate loads
Shell Retinax A	Shell Netherlands/ UK)	Calcium soap	~95 °C	Regular calcium grease for chassis
Mobilgrease XHP 100	ExxonMobil (USA)	Calcium soap	~95 °C	Basic grease for vehicles
Castrol Spheerol EPL 0/1/2	Castrol (UK)	Calcium soap	~90–100 °C	For general industrial use
Klüber STABURAGS NBU 4	Klüber (Germany)	Calcium soap	~85–95 °C	Anti-corrosion, for slow bearings
Divinol Fett 0/1/2	Zeller+Gmelin (Germany)	Calcium soap	~80–100 °C	For cargo equipment, lubrication units

The properties of the obtained greases can be improved by introducing various natural and technogenic fillers that increase penetration, drop point, and colloidal stability. To improve the tribological properties of PGs, additional anti-wear, anti-seize, and anti-pitting additives should be introduced at the production stage [20-21].

The greases we obtained based on oil sludge fraction is characterized by higher values (90-270 units) of consistency, which is determined by PG penetration (degree of softening) in accordance with ASTM D217 / ISO 2137, high colloidal stability (5.0-10.5 %), which is explained by the interaction between the raw material components. At the same time, according to the friction indicator, such greases, due to the absence of additives, are characterized by significantly worse tribological properties than PGs obtained on the basis of used motor oils. The tribological characteristics of such greases are determined solely by the properties of the dispersion medium and thickener, which ultimately contributes to their use as protective greases. Based on theoretical and experimental studies, we propose a combined scheme for the production of greases from alternative raw materials, shown in Fig. 3.

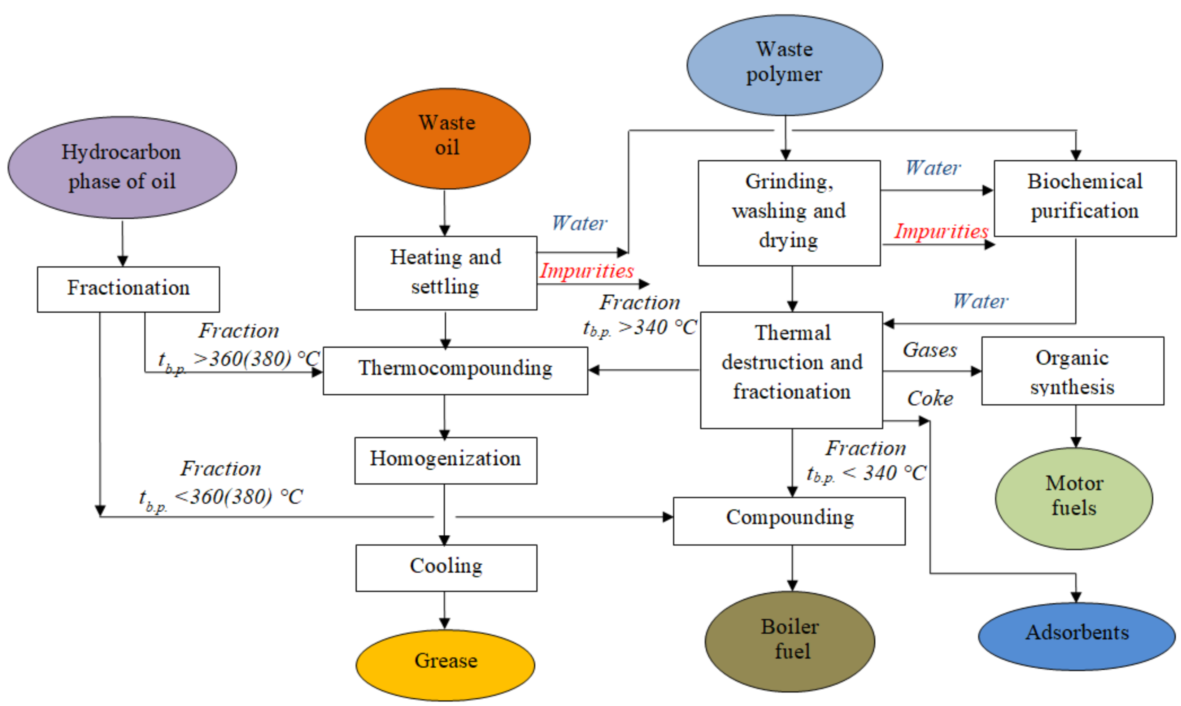

Figure 3. Scheme for obtaining greases based on waste oils, petroleum sludge fractions, and polymer wax.

According to the presented scheme, the hydrocarbon phase of oil sludge obtained under refinery conditions during separation in a tricant is subject to fractionation to obtain a high-boiling fraction ($t_{b.p.}$ >360(380)°C), which acts as a dispersion medium for the production of PG. Fractionation is carried out in packed or rectification columns at atmospheric pressure (P = 0.1-0.12 MPa) or under vacuum (P = 0.0015 MPa). A by-product is a fraction ($t_{b.p.}$ <360(380)°C), which is sent to the compounding unit for the production of boiler fuel.

If used motor oil is used as a dispersion medium, it is first settled for 3 hours at a temperature of 60-80°C to remove ballast – water and impurities. The water undergoes biochemical treatment and is then used in heat exchange equipment in the thermal destruction and polymer fractionation unit. Impurities are the only waste product in the proposed scheme and can be used in the production of building materials, road construction.

Waste polymer (HDPE, PP, or a mixture thereof) is crushed, washed, and dried with air heated to 80-100°C. After washing, the water is fed to a biochemical treatment unit, and the impurities are used in the production of building materials or for road construction. The crushed and purified polymer enters the thermal destruction and fractionation unit, where, at temperatures of 400-410°C and pressures of 0.11-0.13 MPa, products with a lower molecular weight than the original polymer are formed. The liquid destruction products are fractionated, and the resulting fraction ($t_{b.p.}$ >340°C), together with the purified dispersion medium, is sent to the thermocomounding unit. The fraction ($t_{b.p.}$ <340°C), which is a by-product, is sent to the compounding unit with fuel fractions of oil sludge, fuel oil, and oil residues to obtain boiler fuel. Other by-products of thermal polymer destruction are gases and coke. Gases (ethane-ethylene, propane-propylene, and butane-butylene fractions) are sent to the organic synthesis unit for the production of motor fuels, in particular automotive gasoline. After surface activation, coke can be used as an adsorbent for the treatment of industrial wastewater.

In the thermocompounding unit, at temperatures of 120-150°C for 3 hours, a reaction mixture is formed, which then goes to the homogenization and cooling unit, where the final formation of the greases structure takes place. During thermocompounding, fillers and additives can be introduced into the mixture to improve the operational properties of the greases.

5. Conclusions

The production of plastic greases based on alternative raw materials is a technically feasible, economically viable, and environmentally sound solution. It is particularly promising for Ukraine, given the significant amount of accumulated and annually generated waste - used oils, oil refining industry waste, and used polymer products.

Due to the process of thermal compounding at a temperature of 120-150°C of a prepared dispersion medium based on waste motor oils and high-boiling fractions of oil sludge with polymer wax obtained by thermal destruction of HDPE, PP, the samples of greases were obtained. These greases are not inferior in their physical and chemical quality indicators to the best foreign analogues: Total Multis EP 0/1/2; Shell Retinax A; Mobilgrease XHP 100; Castrol Spheerol EPL 0/1/2; Klüber STABURAGS NBU 4; Divinol Fett 0/1/2.

Based on theoretical and experimental studies, a rational combined scheme for the production of greases from alternative raw materials has been proposed. It includes units for preliminary preparation of raw material components; a unit for thermal destruction and distillation of polymers; units for thermocomounding, dispersion, and cooling. The proposed scheme allows, along with the target product – greases – the additional production of motor and boiler fuels, adsorbents, and other by-product-based products.

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To whom correspondence should be addressed: prof. A. Hryhorov, National Technical University «Kharkiv Polytechnic Institute», 61002, 2 Kirpichova str., Kharkiv, Ukraine, E-mail: grigorovandrey@ukr.net