

THE CHOICE OF METHOD OF DISPERSION THE THICKENER FOR THE PRODUCTION OF THE RECYCLING PLASTIC GREASE

Andrey Grigorov¹, Alena Tul'skaya^{1*}, Mykhailo Nahliuk², Valeriia Karchakova³

¹ Department of Oil, Gas and Solid Fuel Refining Processes, National Technical university Kharkiv polytechnic institute, Kyrypychova str. 2, Kharkiv, Ukraine

² Department of Technical Operation and Car Service, Kharkiv National Automobile and Highway University, Yaroslava Mudrogo str. 25, Kharkiv, Ukraine

³ Ukrainian State Research Institute for Carbochemistry, Vesnina str. 7, Kharkiv, Ukraine

Received August 12, 2019; Accepted October 31, 2019

Abstract

The results of dispersing the thickener in the area of grease using a paddle stirrer (rotational speed = 100 and 1000 rpm) and ultrasonic treatment with a frequency of 44 kHz have been presented. It has been concluded that ultrasonic dispersion allows us to increase the dispersion of the thickener up to 3-5 microns and spread it evenly in the grease comparing to the mechanical dispersion. Increasing the degree of dispersion of the thickener allows improving the stability of the obtained grease, its adhesive properties. Also it allows reducing the duration of the process of greases producing.

Keywords: Grease; Thickener; Dispersion; Viscosity; Colloidal stability.

1. Introduction

The quality of commercial greases and the costs associated with their production are largely determined by the design and arrangement of the main equipment in the technological chain, as well as the choice of its optimal parameters. Dispersion of the thickener through in the grease is one of the main factors which is closely related to the temperature and duration of the process and is also a factor that determines the formation of the structure and properties of the grease.

Nowadays, tough environmental requirements for process management demand the modernization of existing industrial schemes for the production of commodity petroleum products. At the same time the environmental requirements can promote the development of recycling technologies that are focused on the use of hazardous industrial and domestic waste as raw materials. [1]. For the effective implementation of both processes – production and recycling, it has been proposed a technology of obtaining the recycling greases based on the use of waste lubricating oils as a dispersion area and polymer waste as a hydrocarbon thickener. The technology for production of recycling plastic greases is more complicated comparing to the production of plastic greases with soap thickeners. It must be considered that the main components in proposed technology are industrial and household wastes, which are impure and chemically instable. Therefore, the technological production process has to include the following steps: cleaning, washing, drying and grinding the components. The approximate technological scheme of recycling grease production is presented in Fig. 1.

Each of the described stages demands the implementation of specific equipment. At the stage of preparation, there should be applied heated tanks, centrifuges, filters to clean the dispersion medium, chopper, belt conveyors, washing and drying apparatus for preparation the thickener [2]. At the stage of components dispersion, important elements are the dosing devices - by weight (for a polymeric thickener) and dosing pumps (for a dispersion medium and liquid additives). However, the main equipment at this stage is a heated reactor with a

specific device for efficient mixing. It provides the initial formation of the structure of recycling grease. The final formation of the entire complex of consumer properties of the product is achieved by cooling the grease in containers and homogenizing it by mechanical treating in valve or rotary-gap homogenizers (pushing under pressure through 30-50 μm holes). The homogenization of plastic grease provides a uniform distribution of the thickener throughout the grease bulk to increase the colloidal stability of grease [3].

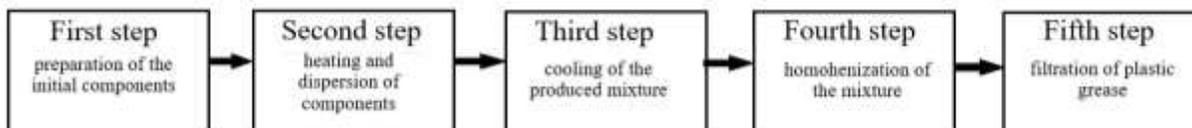


Fig. 1. Scheme of production of recycling greases

A prerequisite for the production of stable uniform greases is effective operation of the mixing device in the reactor [4]. The mixing device provides contact between the dispersion area and the dispersed phase (thickener), reduces the duration of dispersion of the thickener and its distribution throughout the grease, promotes uniform heating of the reaction mixture without local temperature differences across the section and height of the reactor.

It is known that the effectiveness of reactant mixing essentially depends on the design and rotation speed of the stirrer, which has to be determined experimentally for each mixture. The following types of mixing devices are used in industry and laboratory practice: paddle, propeller, anchor, screw (worm), scraper, magnetic, centrifuge, and vibratory mixers [5].

Blade propeller mixers are most commonly used in the production of greases due to their high efficiency and the possibility to control the intensity of mixing. These mixers are suitable for mixing in a laminar mode, and for intensive, turbulent mixing. The device has spiral-curved blades - the angle of inclination in length from 45° at the shaft hub and up to 20° at the end of the blade. Typically, the diameter of the blades of the stirrer is not more than 0.5 of the diameter of the cross section of the reaction zone of the device. The width and angle of the blades are determined experimentally [6].

Recently, industrial technologies for producing greases implemented with mechanical agitation and method of ultrasonic dispersion. Ultrasonic dispersion includes the destruction and dispersion of a solid fraction in the lubricant. Ultrasonic dispersion is based on the difference in the speed of sound waves in liquid and solid area, the formation of reflected and standing waves at the interface, and unevenness of the solid phase, which leads to the ultrasonic cavitation effect and acoustic flow [7]. The cavitation effect is characterized by the occurrence of a shock wave near the irregularities of the surfaces of the particles and the appearance of cavitation bubbles due to the pulsation, high-speed (speed of about 600 m/h) microflows. These microflows contribute to the filling of the space between the particles with a liquid, and the separation of particles of the dispersed phase from each other occurs under the action of shock waves arising in the liquid when the cavitation bubbles collapse [8]. The homogeneity of mixture increases due to cavitation and the occurrence of vortex acoustic microflows, gradient in the structure, and, consequently, the properties of the processed mixture.

Ultrasonic dispersion technology is used to produce plastic lubricants with improved anti-friction properties due to the introduction and uniform distribution in the plastic lubricant fullerene soot [9] and particles of graphite [10].

Considering all these facts, the purpose of current research is to study the effect that different modes of mixing the heated initial mixture have on the formation of the structure and properties of recycling greases obtained from it.

2. Experimental

To achieve this goal, we have produced samples of grease based on used engine oil SAE15W-40(API SF/CD) by dispersing of 5 % (mass) waste products (crushed to 2 × 2 mm) from low-density polyethylene in this oil, with the further homogenization and filtration. Two

samples were obtained by dispersing the thickener in the lubricant in a heated reactor with a paddle propeller stirrer (see fig. 2) at of 430 K and a stirrer rotation speed 100 and 1000 rpm.

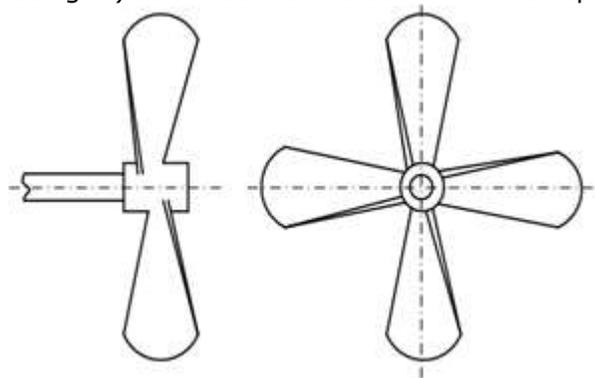


Fig. 2. Blade propeller mixer

The third sample was obtained using an ultrasonic dispersant USDN-1; the oscillation frequency of the emitter was 44 kHz. During dispersion, the reaction mixture has been also heated up to 430 K. The distribution of the thickener particles through the grease has been studied by applying an optical microscope at magnification $\times 250$ in transmitted light (Fig. 3).

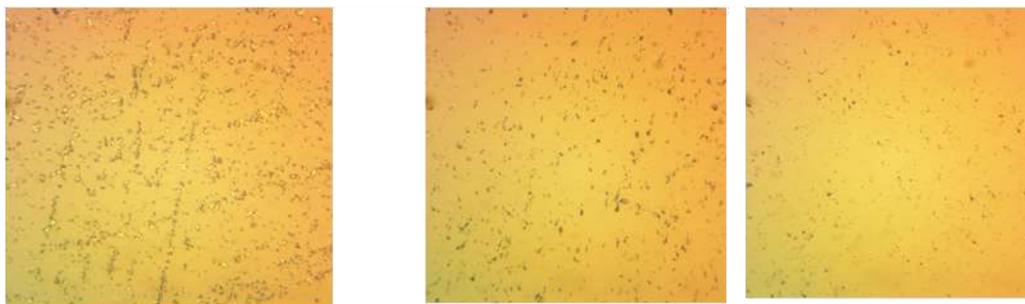


Fig. 3. Grease under the transmitted light: a)-mechanical stirring at 100 rpm; b)-mechanical agitation at 1000 rpm; c)-ultrasonic mixing at 44 kHz

When the heated reaction mixture is stirred by a propeller mixer, the thickener is distributed in the grease area. Different parts of the agitator blade at different angles keep constant contact with the lubricant, which creates intense axial vertical flows that capture all the lubricant layers and ensures mixing in the entire reactor volume. However, when the rotation speed of the stirrer is 100 rpm (see Fig. 3), it is not possible to distribute the thickener over the lubricant area, due to the moving of layers in the laminar mode (Fig. 4, a).

When the rotational speed of the agitator raises up to 1000 rpm, vortex flows of the mixture occur in the plane of rotation from the center of the reactor, where a region of reduced pressure occurs, into which the layers of the mixture are drawn below and above the agitator. This type of mixing responds to the turbulent flow mode and provides uniform distribution of the thickener over the lubricant area (see Fig. 3, b) than with stirring at 100 rpm. The particle size of the thickener varies from 7-10 microns.

The application of ultrasonic dispersion (see Fig. 3, c) comparing to propeller mixers allows to achieve the best dispersion of the thickener inside the lubricant and reduce the particle size of the dispersion phase to 3-5 microns.

Then, in order to study the properties of the obtained grease samples, there have been defined some quality indicators that characterize the rheological properties and the stability of the greases during storage (see Table 1).

For plastic greases obtained on metal soaps, an increase of thickener dispersion degree leads to an increase of the effective dynamic viscosity of grease. While using the solid polymer waste as a thickener there have been obtained different results (see Table 1): with an increase

of the thickener dispersion degree and the uniformity of its distribution over the lubricant area, the effective dynamic viscosity of the lubricant decreases (by 30 Pa/s in average). It can be explained by the presence of large (up to 30 μm) aggregates of thickener particles that provide relatively high resistance to the movement of layers of grease in the gap between the measuring cylinders in determining the effective dynamic viscosity of the lubricant. As the mechanical or ultrasonic stimulation of the grease intensifies, the aggregates have been crushed into smaller particles (particles within 3-5 μm), and, as a result, the effective dynamic viscosity has been reduced. These results correspond to the hypothesis that the viscosity of the aggregate colloidal system is always much higher than the viscosity of this system in a disaggregated state. [11].

Table 1. The quality indicators of the obtained greases

N ^o	Name of indicator	The value for the sample of grease		
1	The method of dispersion of thickener	Propeller stirrer at 100 rpm	Propeller stirrer at 1000 rpm	Ultrasonic dispersion at 44 kHz
2	Form of grease	Homogeneous ointment with a loose black texture		
3	Effective dynamic viscosity η , at 273 K and average deformation speed gradient 10 s ⁻¹ , Pa·s	95	72	65
4	Colloidal stability X_{kc} , %	13.9	9.34	7.78
5	Solubility - in water - in gasoline	Non-soluble Soluble		

It is known that an increase of thickener dispersion degree usually leads to an increase in the stability of the dispersion, which is fully confirmed by the data given in Table. 1. Thus, the colloidal stability of samples of the studied greases improves from 13.9% to 7.78%, depending on the method of thickener dispersing inside the lubricant. It is obvious that a lubricant sample, in which the thickener is dispersed using ultrasound at a frequency 44 kHz, has better colloidal stability compared to lubricant samples, where mechanical dispersion was carried out.

Adhesion properties of greases that characterize the speed of their operation in bearings have also been studied and defined (Fig. 4).

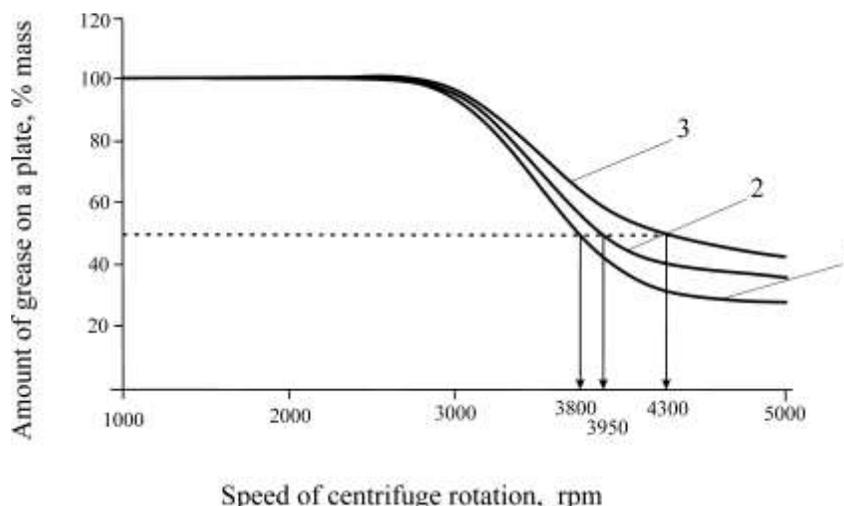


Fig. 4. The dependence of the adhesion properties of grease on the speed of centrifuge rotation: 1 - mechanical agitation at 100 rpm; 2 - mechanical stirring at 1000 rpm; 3 - ultrasonic mixing at 44 kHz

A layer of grease 0.1 mm thick was applied to clean, degreased metal plates and then tested in a centrifuge. The test rotation speed range of the centrifuge was 1000–5000 rpm. Adhesion properties (%) were determined by the mass of lubricant on the plate before and after the test in a centrifuge. Moreover, the boundary value of reducing the mass of lubricant on the plate relative to the initial value was taken at the level of 50% (by mass) in accordance with the practical recommendations of the manufacturers of bearings for their filling with grease.

As has been shown in Fig. 4, increase of thickener dispersion degree is followed by increase of distribution and adhesion of the thickener through in the lubricant (from 3,800 to 4,300 rpm). The lubricant sample subjected to ultrasonic dispersion of the thickener reaches the limit value at a rotor speed of 4300 rpm, which is the maximum value of all the samples examined.

Duration of the main technological operation (τ , s) is one of the most important technological parameters that have a large impact on the quality and price of the final commodity product. It should be noted that the higher speed of mixer rotation is, the less time has to be spent on dispersing the thickener. So, at 100 rpm. it is 3000 s, and at 1000 rpm –1800 s. In contrast to the dispersion of a mechanical stirrer, ultrasonic dispersion reduces the processing time to 900 seconds.

The use of ultrasonic dispersion of the thickener, along with the reduction of the processing time, allows providing the safety of the technological operation, due to the absence of elements rotating at high speeds, as well as to simplify the technological scheme of the production of recycling plastic lubricants. So, due to the high degree of dispersion of the thickener by the lubricant volume, from the technological scheme of production (see Fig. 1), it is possible to exclude the stage of homogenization and filtration of the lubricant. In the reactor, designed to disperse the components, to melt the thickener, there is no need to supply external thermal energy, and heating the mixture to the required temperature (in this case up to 160°C) is due to ultrasonic action, which greatly reduces production costs.

3. Conclusions

It has been found that ultrasonic dispersion provides a higher degree of thickener dispersion and its uniform distribution comparing to mechanical. As a result, the colloidal stability of the resulting grease has been raised up to 7.85%, the adhesion properties improved (the grease maintains the centrifuge rotor speed of 4300 rpm), and the process duration has been reduced to 1800 s.

However, during the ultrasonic dispersing of the thickener in the lubricant, there has been observed a slight decrease in the effective dynamic viscosity of the final product, which can probably be explained by the destruction of the aggregates formed by the thickener particles.

The choice of a method of dispersing the thickener in the production of recycling greases should be determined by the properties of the final product, ease of implementation of the technological scheme, environmental requirements, and economic feasibility of applying this technological solution.

References

- [1] Directive 2008/98/EC of the European parliament and of the council. Official Journal of EC L 312/3, 22 November 2008, pp. 3–30.
- [2] Myimirin VN. Secondary refining of polymer materials in Europe: new and proved approaches (in Russian), Vtorichnaya pererabotka polimernykh materialov v Evrope: novyye i proverennyye resheniya. Polimernyye materialy, 2013; (9): 22-29.
- [3] Fuks IG. Composition, properties and production of plastic grease (in Russian), SHibryaev SB. Sostav, svoystva i proizvodstvo plastichnykh smazok. Gosudarstvennaya akademiya nefti i gaza im. I. M. Gubkina, 1992
- [4] Ischuk YUL. Composition, properties and production of plastic grease (in Russian), Sostav, struktura i svoystva plastichnykh smazok. Nauk. dumka, 1996
- [5] Karpushkin SV. Calculation and choice of mechanical mixers of vertical apparatus: tutorial (in Russian), Raschëty i vyibor mekhanicheskikh peremeshivayuschih ustroystv vertikalnykh emkostnykh apparatov: uchebnoe posobie. Tambov: Izd-vo Tamb. gos. tehn. un-ta, 2009.

- [6] Patmaeva YV. Production of greases: technological approaches (in Russian), *Proizvodstvo smazok: tehnologicheskie resheniya Vestnik Kazanskogo tehnologicheskogo universiteta*, 2014; (1): 333-335.
- [7] Hmelev VN, Leonov GV, Barsukov RV, TSyiganok SN, SHalunov AV. Ultrasound multifunctional and special equipment for intensifying of industrial and agricultural processes (in Russian), *Ultrazvukovyye mnogofunktsionalnyie i spetsializirovannyye apparaty dlya intensifikatsii tehnologicheskikh protsessov v 175 promyshlennosti, selskom i domashnem hozyaystve*. Alt. gos. tehn. unt, BTI. – Biysk: Izd-vo Alt. gos. tehn. un-ta, 2007
- [8] Intensifying of ultrasound cavitation processes in heterogenous systems in high-viscosity and non-Newtonian fluid Speciality (in Russian), *Povyshenie effektivnosti ultrazvukovogo kavitatsionnogo vozdeystviya na himiko-tehnologicheskie protsessyi v geterogennyih sistemah s nesuschey vyisokovyazkoy ili nenyutonovskoy jidkoy fazoy Spetsialnost 05.17.08 – Protsessyi i apparaty himicheskikh tehnologiy Dissertatsiya na soiskanie uchenoy stepeni kandidata tehnikeskikh nauk Nauchnyy rukovoditel – d.t.n., dotsent SHalunov A.V. Biysk – 2014.*
- [9] Plastic grease: (in Russian), *Plastichnyy smazochnyy material: Pat. №2268291 RF, MPK8 S10M 125/02, C10N 30/06 / V.P. Budtov, B.M. Ginzburg, D.G. Tochilnikov, A.A. Shepelevskiy*. Zayavl. 26.03.2004. Opubl. 20.01.2006, Byul. № 1.
- [10] Production of grease composition (in Russian), *Sposob polucheniya smazochnoy kompozitsii: Pat. № 2428462 RF, MPK8 S10M 177/00, C10M 125/02, S01V31/04 / E.V. Kim, E.V. Golubev*. Zayavl. 24.02.2010. Opubl. 10.09.2011, Byul. № 5.
- [11] Matvienko VN, Kirsanov EA. Viscosity and structure of disperses (in Russian), *Vyazkost i struktura dispersnyih system. Vest. Mosk. Un-Ta.* – 2011; 52(4): 243-276.

To whom correspondence should be addressed: Dr. Alena Tul'skaya, Department of Oil, Gas and Solid Fuel Refining Processes, National Technical University Kharkiv Polytechnic Institute, Kyrypchova str. 2, Kharkiv, Ukraine, E-mail fuel.khpi@gmail.com