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Microstructure of complex lithium lubricants

Larysa Yu. Bodachivska [1], Iryna O. Venger [1], Artem Yu. Verba [2]

[1] *V.P. Kukhar Institute of Bioorganic Chemistry and Petrochemistry of National Academy of Sciences of Ukraine*
1 Academica Kukhara Str., Kyiv, 02094, Ukraine, e-mail: bodach@ukr.net
[2] *"RMA "Innovative Technologies and systems" LLC*
1 Zhmerinskaya Str., Kyiv, 03148, Ukraine, e-mail: info@itis.com.ua

This work presents the results of studies of the microstructure and properties of lubricant samples based on synthesized complex lithium soaps of hydroxylated fatty acids from of waste food oil as a lubricant thickener. The results of scanning electron microscopy confirmed the differences in the peculiarities of the formation of the structural framework of lubricants depending on their composition, thickener concentration, complexing agents, and heat treatment temperature. It was determined that it is the lubricant thickener that binds the dispersion medium through intermolecular interaction, forms a three-dimensional interwoven microstructure, and gives the lubricant improved rheological and volumetric-mechanical properties. Complex lithium soap is more structured than simple lithium soap, although both are formed by a connected three-dimensional mesh microstructure. It has been shown that the rheological and operational properties of lubricant can be improved by using various complexing agents. The lithium complex lubricant samples are characterized by the maximum thickening effect and have lower effective viscosity values at minus 30 °C. With an increase in the concentration of the thickener, the effective viscosity of lithium complex lubricants increased, but the indicators of mechanical stability and drop point remained almost unchanged. It was found that the optimum heat treatment temperature for complex lithium lubricant is 220 °C. If the heat treatment temperature of the lubricant was exceeded, for example, at 240 °C, the microstructure of the complex lithium soap was destroyed, which led to a deterioration in viscosity and colloidal stability. The established rheological, volume-mechanical, and temperature regularities of the structure formation of the dispersed phase of lubricants can be used to select rational modes of obtaining complex lithium lubricant compositions intended for use in friction units of modern machines and mechanisms.

Keywords: lubricant thickener, microstructure, rheology, mechanical stability, complex lithium lubricants

Introduction

Modern advanced technology places stringent requirements on lubricant developers. Lubricants have to withstand friction units' overheating, stay on surfaces at high speeds, not leak out of bearings, not cause metal corrosion, and protect them from the harmful effects of various factors. Lubricants with a wide range of operating temperatures and those that work in the presence of moisture are increasingly in demand. Lithium lubricants (simple and complex) fully meet these requirements.

Lithium lubricants consist of a base oil (mineral or synthetic) and a lithium soap thickener, which is obtained by direct saponification (in situ) of usually stearic/monohydroxystearic acid, as well as various natural fats and synthetic fat substitutes [1]. Lithium lubricants are classified as multipurpose lubricants. They are water-resistant, characterized by good mechanical and thermal stability, a wide range of application temperatures, suitable for lubricating most friction units of industrial equipment and vehicles, and provide long-term protection against oxidation, corrosion, extreme temperatures, and wear.

Lithium complex lubricant (cLi) is an improved version of lithium lubricant, which includes a thickener based on lithium complex. Complex lithium soap (cLi-soap) is a product of cocrystallization of Li-soap of high molecular weight fatty acids and Li-salts of low molecular weight organic and inorganic acids [1]. Lubricants based on cLi-soaps are characterized by improved antioxidant, mechanical and thermal stability, and anti-wear characteristics compared to simple lithium lubricants. Their dropping point exceeds 230 °C. Complex Li-lubricants are durable, and stable and are most often used to fill friction units for the entire period of their operation. They are ideal for high-temperature, low-temperature, and highly loaded industrial applications such as bearings, gears, and heavy machinery.

The authors of [1, 2] showed that the main component of cLi-soaps is monohydroxycarboxylic acids with a different arrangement of the hydroxyl group in the hydrocarbon radical of the acid about the carboxylic group – 12-HoSt, obtained as a result of hydrogenation of castor oil; 6-HoSt, synthesized by free radical addition of cyclohexane to α-oleic acid; 9(10)-HoSt, obtained by hydroxylation of oleic acid; 6-HoSt, synthesized by the free radical addition of cyclohexane to α-olefins followed by oxidation of 2-alkylcyclohexanol, accompanied by ring breakage. The complexing agent was dicarboxylic acids (H_2Dc): azelaic acid (H_2Az), adipic acid (H_2Ad), sebacic acid (H_2Se), or hydroxybenzoic acid in combination with boric acid in cLi-soap. Mineral and synthetic base oils, which differ in viscosity, aniline point, and other physical and chemical characteristics, were used as dispersion media. Also in these works, the optimal values of the molar ratio of cLi-soaps and dicarboxylic acids are given. It is noted that an increase in the proportion of H2Dc reduces the thickening ability of cLi-soap, decreases the drop point, and worsens the rheological characteristics of cLi-lubricants and their microstructure.

Lithium complex lubricant is the most common lubricating grease in the high-end lubricant market. Lithium complex soap holds base oil by intermolecular interaction and shows the entangled fiber structure, where its composition and structure influences the rheological properties [3]. Yeong et al. [4] found the yield stress and viscosity of lithium lubricant increase as the increase of thickener concentration, as well as storage modulus, attributing to the high volume fraction of thickener concentration results in a stronger gel network. Delgado *et al*. [5, 6] found that the soap fiber shows higher physical entanglement with the increase of soap concentration, and the relative elastic characteristics increases with the decrease of base oil viscosity due to the lower viscosity of base oil increase the affinity for the soap.

Investigations at the Competence Center of Tribology Mannheim show that the lubricant service life for roller bearing lubrication, even at high temperatures, does not only depend on classic oil aging. In numerous roller bearing tests and by means of rheological measurements, it could be shown that the loss of the lubricating effect is a consequence of the change in the thickener structure. The fiber structure and the inner network are of the greatest importance for the lubricating properties of soap greases [7]. Adhvaryu et al. investigated the change in the fiber structure of lubricating greases as a function of antioxidants. Unsaturation and fatty acid chain length of soap molecule dictate the fiber shape and distribution, which can be translated to hardness, thermo-oxidative stability, shear stability, water tolerance, and other important properties of grease [8]. They define the breakdown of the structure as damage to the lubricating properties. Couronne and Vergne found that the fiber length is shortened by thermal aging [9]. Gonçalves et al. found also that the thickener matrix changes with thermal aging [10]. Shen et al. investigated the thickener structure of a lithium–calcium thickened grease and also showed that the network structure is gradually destroyed with a longer thermal aging time and that the stability of the grease structure decreases significantly [11].

Hodapp *et al*. [12] noted that the type and viscosity of the base oil does not affect the absolute value of the complex viscosity and the shape of the filaments formed by this thickener. The complex viscosity of the lubricants only depended on the thickener concentration. High-frequency shear modulus data, however, indicated that the thickener lithium 12-hydroxystearate formed stiffer networks/filaments in poly-α-olefins than in mineral oils. As expected, the viscosity increased with increased thickener concentrations, but microscopy and high-frequency rheometry revealed that the thickness, length, and stiffness of the individual filaments did not change. In mineral oil, the 12-hydroxystearate thickeners yielded higher viscosity than the corresponding stearates with the same metal ion. The filamentous lithium thickeners created stronger networks than the roundish aggregates formed by magnesium and zinc stearate.

Saatchi *et al*. [13] investigated the mechanism of oil release from the lithium complex in soap and nonsoap thickeners of greases. It is assumed that the leakage occurs from the viscous flow of unbound oil in the porous structure made of the effective media (the region surrounding the thickener particles).

Zhang *et al*. [14] investigated on microstructure, friction and rheology of four lithium greases formulated with four different base oils (paraffinic oil, naphthenic oil, poly-α-olefin and polyol ester). It was found that naphthenic oil-based grease exhibited the best colloidal stability. Lithium fibers in polyol ester-based lubricant showed a relatively flat network compared to the three-dimensional entangled structure of the other three greases. Tribological performance of greases differed from their corresponding base oils, which might be attributed to the different microstructure of lithium soap fibers. Three-dimensional entangled structure was more suitable for friction-reducing than flat network structure. Moreover, noise test revealed that naphthenic oil-based lubricant was suitable for low-noise bearing lubrication. This study provided fundamental guidance for the selection of base oil which is beneficial for the development of high-performance lubricant products.

In industrial production, the selection of raw materials is often based on the criteria of availability and cost. Aromatic, alicyclic, and aliphatic amines with up to 20 carbon atoms in the hydrocarbon chain are commonly used to make lithium greases. Lithium soaps are obtained by saponification of 12-hydroxystearic acid with LiOH and organic acids [1, 3]. In works [15-18], hydroxylated fatty acid soaps based on vegetable oils or secondary fatty raw materials were synthesized, and thixotropic systems with improved performance characteristics were developed.

Therefore, the purpose of this work is to obtain samples of complex lubricants based on synthesized lithium soaps of hydroxylated fatty acids from waste food oil and to study the effect of the microstructure of these samples on rheological and volumetric-mechanical properties.

Experiment

Waste food oils (WFO), a sample provided by a local catering establishment, were used as an alternative source of secondary fatty acids. 99.5 % potassium iodide, 35 % hydrogen peroxide, 99.8 % sodium hydroxide, 99 % lithium hydroxide, 13 % hydrochloric acid, 12-hydroxystearic acid, 99.8 %, 99.5 % diethanolamine, 99.8 % formic acid, 99.7 % adipic acid, 99.6 % sebacic acid were purchased from Chemlaborreactiv (Ukraine).

Samples of complex lithium lubricants were evaluated according to the following indicators penetration (ISO 2137), dropping point (ASTM D 2265), colloidal stability (GOST 7142, method A), viscosity (GOST 7163). The scanning electron microscopy (SEM) images were taken using brand appliance CAMSCAN.

Hydroxylated fatty acids were synthesized by epoxidation of unsaturated fatty acids of waste food oil followed by hydrolysis by acylglycerol grouping and oxirane rings. Fatty acid aminoamides of waste food oils were synthesized by epoxidation of oils followed by the opening of oxirane cycle and transamidation of acylglycerols with diethanolamine according to the method described in patent [15].

Results and Discussion

A complex lithium lubricant is a highly structured colloidal-dispersed system in which the internal dispersed phase, a thickener based on a lithium complex (complex lithium soap), binds the dispersion medium (mineral or synthetic oils) into a single system. Complex lithium soap (cLi-soaps) is a product of cocrystallization of cLi-soap of high molecular weight fatty acids and Li-salts of low molecular weight organic and inorganic acids [1]. Indeed, having a polar hydrophilic component and a developed nonpolar lipophilic part, the thickener is actively concentrated at the interface of two mutually insoluble phases, reducing the surface or interfacial tension.

Chemical modification of waste food oils was carried out to obtain an emulsifier-stabilizer as a thickener for lubricants – saponified hydroxylated fatty acids (WFO_{-OH}) a solution of lithium hydroxide. The WFO hydroxy acids were synthesized by epoxidation of unsaturated fatty acids (oleic, linoleic, linolenic, erucic) or their mixture, which is part of used food oils, followed by hydrolysis by acylglycerol groups and oxirane rings [15].

Using WFO_{-OH}, samples of complex lithium lubricants (cLi-lubricants) based on a base oil of group IV according to the API (American Petroleum Institute) classification with a viscosity of 9.5 mm^2/s at 100 °C were made. The complex lithium lubricant is manufactured according to the conventional technology containing lithium fatty acid soaps [1, 15] and includes the following steps synthesis of hydroxylated fatty acids of WFO; production of lithium soaps of hydroxylated fatty acids (saponification of hydroxylated fatty acids with lithium hydroxide solution) in the base oil environment; introduction of a complexing agent (adipic acid (H_2Ad) or sebacic acid (H_2Se); dehydration of the resulting oil-soap mixture; thermomechanical treatment of the mixture and its cooling; homogenization of the lubricant.

By the above technology, using the appropriate raw materials, a sample of simple lithium lubricant without a complexing agent (sample 1) and samples (2, 3, 4, 5) of complex lithium lubricant with the following content, % wt.: lithium soaps of hydroxy acids of oils – 15.0, synthetic oil – the rest, heat treatment temperature 220 °C (sample 1); complex lithium soap of hydroxy acids of oils (complexing agent H_2Se) – 20.0, synthetic oil – the rest, heat treatment temperature 220 °C (sample 2); complex lithium soap of hydroxy acids of oils (complexing agent H_2Ad) – 20.0, synthetic oil – the rest, heat treatment temperature 220 °C (sample 3); complex lithium soap of hydroxy acids of oils (complexing agent H_2Se) – 20.0, synthetic oil – the rest, heat treatment temperature 240 °C (sample 4); complex lithium soap of hydroxy acids of oils (complexing agent H_2Se) – 22.0, synthetic oil – the rest, heat treatment temperature 220 °C (sample 5).

Table 1 presents the results of the rheological and bulk-mechanical properties of lithium lubricant samples. The obtained cLi-lubricants samples are characterized, first of all, by high thermal stability, which confirms the value of the dropping point. The dropping point is the temperature at which a thixotropic system passes from a plastic solid to a liquid state. A lubricant is high-temperature if at least one of the following requirements is met: its dropping point is higher than 250 °C, and the temperature limit of application is higher than 150 °C [19]. The obtained samples of cLi-lubricants (2, 4, 5) are characterized by a higher dropping point than sample 1 of a simple lithium lubricant. Table 1 presents the results of studies on the mechanical stability of lubricants.

Mechanical stability – the ability of a lubricant to resist mechanical stress and restore lost volumetric and mechanical properties – is one of the most important parameters for assessing the operational properties of lubricants [1]. Lubricant, which quickly breaks down and is poorly restored after stress relief, leaks from bearings, axle boxes, *etc*., causing dry friction and overheating. Mechanical stability is largely determined by the properties and concentration of the thickener, as well as the stability of its structural framework, which is manifested in the magnitude of the bonding forces between its individual components.

Table 1. Properties of samples of lithium lubricants

Sample No.	Samples of complex lithium lubricants	Properties of samples of complex lithium lubricants					
		Dropping point, °C	Penetration at 25 °C, $m \cdot 10^{-4}$			Colloidal stability, % of extracted oil	Effective viscosity at -30 °C and strain-rates 10 s^{-1}, Pa·s
			After 60 double strokes (P_1)	After 10,000 doubles strokes (P_2)	Mechanical stability, change (ΔP)		
1	Sample 1	204	240	260	20	13.4	1138
2	Sample 2	>250	256	274	18	14.2	800
3	Sample 3	213	212	236	24	16.6	–
4	Sample 4	250	268	289	21	16.2	890
5	Sample 5	>250	242	258	16	12.6	989

He criterion for mechanical stability is the change in the penetration index of the lubricant after prolonged mechanical destruction (P_2-P_1 = ΔP). According to ISO 2137, the lubricants were subjected to destruction in a standard penetrometer mixer. P_1 was determined after 60, and P_2 after 10.000 double strokes.

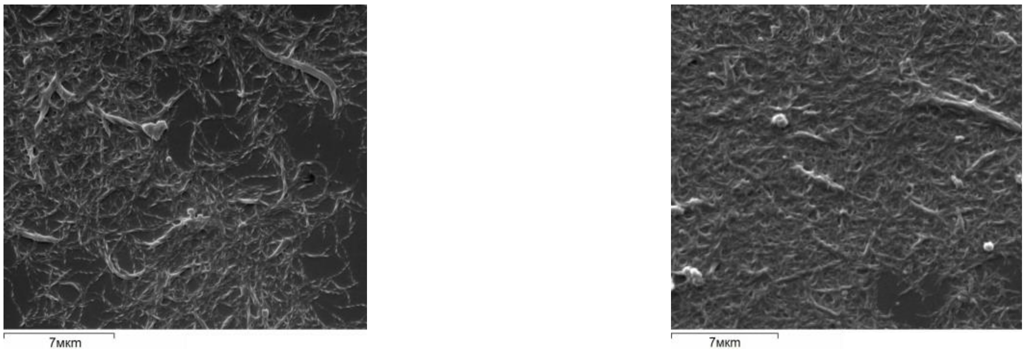
The change in the state of the lubricant (ΔP) characterizes the stability of its structural framework: for $\Delta P < 30$ – mechanical stability is excellent, for $\Delta P = 30 \div 60$ – good, for $\Delta P = 61 \div 100$ – satisfactory, for $\Delta P > 100$ – unsatisfactory [20]. The data in Table 1 indicate that cLi-lubricants have high mechanical stability (ΔP does not exceed 24 units).

Viscosity is the most important operational characteristic that determines the conditions for filling the lubricant into the friction units at low temperatures and affects the starting torque of the bearings. For lithium lubricant samples, the viscosity at a temperature of minus 30 °C and an average strain rate gradient of 10 s-1 is Li-lubricant (sample 1) 1138 Pa·s, cLi-lubricants (samples 2, 4-5) is within 800-989 Pa·s (Table 1). The results show that cLi-lubricants have a lower viscosity compared to simple lithium lubricant.

Figs. 1-4 shows the morphological characteristics of Li-lubricant and cLi-lubricants samples with different component compositions. The formation of the structural framework of lubricants occurs due to the formation of hydrogen bonds between the molecules of the thickener and the dispersion medium [21]. In the process of emulsification, in the manufacture of lubricant compositions, the thickener molecules form reverse micelles in the form of fibers [1]. The fibers, colloidally suspended in the base oil, collide and entangle together, forming a connected three-dimensional mesh microstructure that holds the dispersion medium, as confirmed by SEM images. This microstructure affects the rheological and bulk-mechanical properties of the lubricants.

The results of scanning electron microscopy confirmed differences in the features of the formation of the structural framework of the lubricant thickener depending on its composition, concentration, complexing agents, and the temperature of the lubricant heat treatment. The dispersed phase of lubricants based on lithium soaps of hydroxylated fatty acids and based on complex lithium soaps of hydroxylated fatty acids has significant differences between sample 1 (Fig. 1 a), and sample 2

(Fig. 1 b). Complex lithium soap is more structured than simple lithium soap, although both are formed by entangled fibers. With the same soap concentration in the lubricant, the number of fibers in complex lithium soap is many times greater. This explains the better rheological and operational properties of complex lithium lubricants (Table 1).



a b

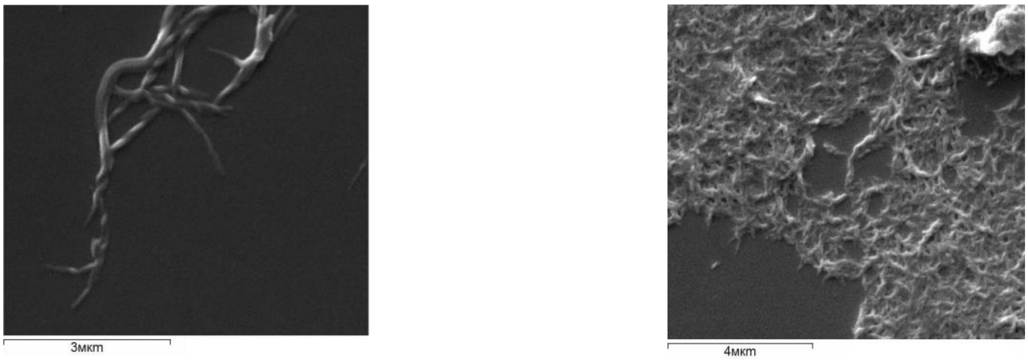
Fig. 1. Microstructure of lithium lubricants: a) simple lithium lubricant; b) complex lithium lubricant

Adipic acid and sebacic acid were used as complexing agents in the preparation of complex lithium soap. Accordingly, the cLi-lubricant samples: sebacic acid - H_2Se (sample 2, 4, 5), adipic acid - H_2Ad (sample 3). One of the main indicators confirming the presence of a lithium complex in the lubricant is the high dropping point, over 250 °C, compared to simple lithium lubricants (sample 1), in which the dropping point is over 180 °C [1]. Therefore, according to the data in Table 1, it can be concluded that when the complexing agent H_2Se (sample 2, 4, 5) is introduced into the thickener composition, a complex molecular complex is formed between the salts obtained during saponification. However, when the complexing agent H_2Ad (sample 3) is introduced into the thickener composition, a mixture of dilithium and lithium soaps is formed instead of a complex lithium soap. This confirms the conclusions of the authors of the works [1, 15] that complex lithium soap is formed when a dicarboxylic acid with a chain length of at least 9 carbon atoms is introduced into the thickener, and in adipic acid, the chain length consists of 8 carbon atoms.

The maximum thickening effect of the dispersed phase of the lubricant upon introduction of H_2Se is explained by the structural features of the formed complex lithium soap. Indeed, in Fig. 1 b we observe that sample 2 of complex lithium lubricant with H_2Se complexing agent has a more structured intertwined three-dimensional mesh microstructure, which provides better binding of the dispersion medium and better colloidal stability, lower rheological properties (Table 1).

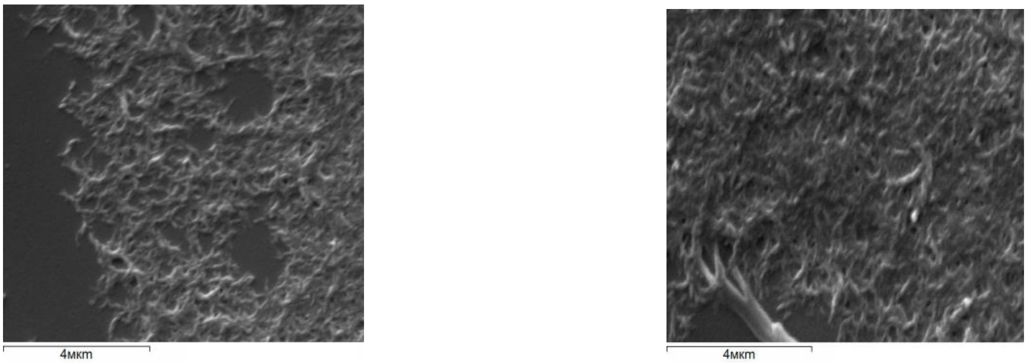
As can be seen from the SEM images (Fig. 2 a, b), changing the heat treatment temperature of the lubricant leads to the formation of different types of microstructure. At high temperatures of heat treatment of cLi-lubricant (240 °C), an amorphous dispersed phase is formed (sample 4), capable of retaining the dispersion medium in its cavities (Fig. 2 a). This is confirmed by the high values of viscosity and colloidal stability indicators. At a heat treatment temperature of cLi-lubricant 220 °C (sample 2), a strong, bonded three-dimensional mesh microstructure of the thickener is formed, the thickening ability of the dispersed phase, the dropping temperature, penetration, colloidal stability, and rheological properties are improved (Table 1).

To determine the effect of thickener concentration on rheological characteristics, we prepared sample 2 cLi-lubricant with a cLi-soap concentration of 20 % and sample 5 cLi-lubricant with a cLi-soap concentration of 22 %. It was found that the viscosity of lubricants increased with increasing thickener concentration (Table 1). SEM images (Fig. 3) demonstrate that with increasing thickener concentration, there is a gradual development of soap fibers, the number and length of which increase, achieving a structured fibrous microstructure [12]. Sample 5 of the lubricant with a thickener content of 22 % has better colloidal and mechanical stability. The change in the thickener concentration in the lubricant (sample 5) did not affect the dropping point value, but the rheological properties deteriorated with increasing thickener concentration (Table 1).



a b

Fig. 2. Microstructure of complex lithium lubricants at different heat treatment temperatures: a) 240 °C; б) 220 °C



a b

Fig. 3. Microstructure of complex lithium lubricants at different thickener concentrations, %: a) 20%; б) 22%

Thus, the rheological and volumetric-mechanical properties of lubricants depend on the bound three-dimensional mesh microstructure of the dispersed phase. This complex molecular complex manifests in the magnitude of the bonding forces between its components. The formation of a stable structural framework of lubricants depends on the optimal composition, concentration of thickener, complexing agents, and heat treatment temperature. Otherwise, instead of complex lithium lubricants, a simple mechanical mixture is formed.

It has been determined that the maximum thickening effect of the dispersed phase, lower values of the effective viscosity at minus 30°C, an increase in the drop point, and a stable structural framework of the complex lithium lubricant are provided by the introduction of a complexing agent such as sebacic acid. The optimum concentration of the thickener is 20-22 %, but increasing the concentration led to an increase in the effective viscosity of the lubricants, while the indicators of mechanical stability and drop point remained almost unchanged. The optimum heat treatment temperature for complex lithium lubricant is 220 °C. If the heat treatment temperature of the lubricant was violated, the microstructure of the complex lithium soap was destroyed, which led to a deterioration in viscosity and colloidal stability.

It was found that complex lithium soap is more structured than simple lithium soap, although both are formed by a connected three-dimensional lattice microstructure.

Conclusion

The use of lithium soaps of hydroxylated fatty acids from of waste food oil and the introduction of a complexing agent made it possible to obtain a complex molecular complex, a complex lithium soap - a bound three-dimensional mesh structure with tangled soap fibers that retains the base oil in the lubricant and gives the lubricant appropriate performance characteristics.

The established rheological, volume-mechanical, and temperature regularities of the structure formation of the dispersed phase of lubricants can be used to select rational modes of obtaining complex lithium lubricant compositions intended for use in friction units of modern machines and mechanisms.

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Мікроструктура комплексних літієвих мастил

Лариса Ю. Бодачівська[1], Ірина О. Венгер[1], Артем Ю. Верба[2]

[1] *Інститут біоорганічної хімії та нафтохімії ім.В.П. Кухаря Національної академії наук України*
вул. Академіка Кухаря, 1, Київ, 02094, Україна, e-mail: bodach@ukr.net
[2] *ТОВ «НВО «Іноваційні технології і системи»*
вул. Жмеринська, 1, Київ, 03148, Україна, e-mail: info@itis.com.ua

В даній роботі представлено результати досліджень мікроструктури та властивостей зразків мастил на основі синтезованих комплексних літієвих мил гідроксильованих жирних кислот відпрацьованої харчової олії, як загусника мастил. Результати скануючої електронної мікроскопії підтвердили відмінності в особливостях формування структурного каркасу мастил залежно від їхнього складу, концентрації загусника, комплексоутворюючих агентів та температури термообробки. Визначено, що саме загусник мастила зв’язує дисперсійне середовище за рахунок міжмолекулярної взаємодії, утворюює тримірну переплетену мікроструктуру і надає мастилу покращені реологічні та об’ємно-механічні властивості. Комплексне літієве мило більш структуроване, ніж просте літієве, хоча обидва утворені зв’язаною тримірною сітчастою мікроструктурою. Показано, що реологічні та експлуатаційні властивості мастила можна поліпшити за допомогою різних комплексоутворювачів. Зразки мастил на основі літієвого комплексу характеризуються максимальним загущувальним ефектом і мають нижчі значення ефективної в’язкості за мінус 30 °C. Зі збільшенням концентрації загусника ефективна в’язкість комплексних літієвих мастил збільшувалась, але показники механічної стабільністі та температури краплепадіння залишались майже без змін. Виявлено, що оптимальна температура термічної обробки комплексного літієвого мастила становить 220 °C. При порушенні температури термічної обробки мастила, наприклад за 240 °C, відбулось руйнування мікроструктури комплексного літієвого мила, що призвело до погіршення показників в’язкості та колоїдної стабільності. Встановлені реологічні, об’ємно-механічні та температурні закономірності структуроутворення дисперсної фази мастил можуть бути використані для вибору раціональних режимів одержання комплексних літієвих мастильних композицій призначених для використання у вузлах тертя сучасних машин і механізмів.

Ключові слова: загусник мастил, мікроструктура, реологія, механічна стабільність, комплексні літієві мастила