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# Technological Control of Production of Plastic Lubricants by Rheological Properties

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**Abstract**

The article substantiates the importance of technological control of the production of plastic lubricants which characterizes physical, chemical or operational properties. Effective dynamic viscosity has been considered the most appropriate characteristics for technological control of for production of plastic lubricants. Effective dynamic viscosity of two-component plastic lubricant produced from secondary raw materials, as well as lubricants obtained using classical petroleum raw materials and soap thickeners, significantly depends on the viscosity of the base oil. It was defined that type of polymer thickener has rather insignificant effect on the value of the effective dynamic viscosity comparing to viscosity of the base oil. High-density polyethylene (HDPE) has the biggest thickening effect on the base oil, and polypropylene (PP) has the least. A diagram of location of the viscosity measuring sensors in the middle of the apparatus for dispersing the polymer thickener in base oil as a dispersion area was proposed. It helps to optimize the process of dispersing the polymer thickener by controlling the duration technological process that results in reducing the consumption of energy resources.

***Keywords:*** *Rheology; Plastic lubricants; Viscosity; Waste; Polyethylene; Polypropylene; Product innovation; Process innovation.*

## 1. Introduction

Technological control in production of petroleum products and plastic lubricants in particular provides the required level of quality of the commodity product, which meets generally accepted world standards. This is primarily related to the optimization of the technological process according to such important technical and economic indicators of production as the consumption of energy resources, the duration of technological operations, ensuring the necessary production capacity. The specified indicators are based on the production design stage and significantly affect production costs, as well as the associated cost of production.

Technological control of production in petrochemical industry usually includes parameters that characterize specific chemical reactions of petroleum hydrocarbons that affect the properties of the final product. These parameters are pressure, temperature, the duration of the technological operation, or indicators of the quality of oil products that reflect their physical and chemical properties.

Nowadays technological control of production of plastic lubricants can be carried out both by controlling the duration of technological operations (for example, dispersion of the thickener) and by controlling the indicators of the quality of the intermediate/final product [1]. Hence, the first way usually means overspending of energy resources and does not always provides with objective information about product properties. The second way significantly increases the operational flexibility because it provides an operator of operational unit with reliable information that can be used for assessment of the technological process. But at the same time, there is a certain complexity with the choice of a quality indicator suitable for creating a system of operational control of production (OCS). In our opinion, from all the variety of indicators that are widely used today to determine the quality of plastic lubricants, the most sensible is operating with their rheological properties [2-3]: tangential shear stress ($\tau_r$, $P_a$ ), the rate of shear deformation or the shear gradient ($D_r$, $s^{-1}$ ), which occurs in the lubricant layer under the influence of $\tau_r$, and the effective dynamic viscosity ($\eta_m$, Pa·s ).

Rheological properties of plastic lubricants are important operational properties that significantly affect the reliable operation of equipment [4].

Recycling and processing of various petroleum wastes was considered a promising branch of industrial development [5-7]. Nowadays industrial and domestic wastes are a significant environmental problem. They have to be stored in special warehouses equipped with fire alarm sensors [8] or open landfill that produce environmental pollution [9].

Hodapp *et al.* [10] proposed to vary the viscosity of lubricants by adding thickeners. Basta *et al.* [11] described effect of polymer additives on organic lubricants by changing of kinematic viscosity. Baruah *et al.* [12] studied and improvement of lubricants quality by blending with organic additives of Methyl methacrylate (MMA). Quality of lubricants was assessed by the value of dynamic viscosity and shear stress. Authors of [13] and [14] described the behaviour of lubricants after blending with multifunctional additives. However, despite the numerous researches, development of technological scheme of sensors that provide a technological control of lubricants production is still an important and relevant task.

## 2. Materials and methods

Used lubricating oil (as a base oil) and used polymer products (dispersion phase) were used as the main materials for obtaining samples of two-component plastic lubricants, that actually provide the required thickness of the base oil.

The used oils were taken directly from the nodes and aggregates where they were operated during their replacement in accordance to the maintenance regulations. For research we selected 4 groups of oils (according to their viscosity), each included 10 oil samples. Group I – motor oils of viscosity classes SAE15W-40, SAE10W-40; Group II – SAE 85W-90 viscosity class transmission oils; Group III – HLP-46 brand hydraulic oils; Group IV – industrial oils of the brands BP Energol CS68, Mobil DTE Oil Medium Heavy.

As polymer products there were used solid household waste sorted by type: low-density polyethylene (LDPE), high-density polyethylene (HDPE) and atactic polypropylene (PP). The most valuable for our research is polyethylene with a low content of impurities with an atactic structure and a relative molecular weight of 80,000-200,000. This is primarily due to its prevalence among solid household waste, high viscosity and elasticity that have a positive effect on the rheological properties of two-component plastic lubricants. High molecular weight allows to provide thermo-mechanical-destructive processing with the formation of polymer chain fragments that can be used as a dispersed phase (thickener) of two-component plastic lubricants.

The study of the production two-component plastic lubricants from secondary raw materials was carried out in accordance with the Energy Strategy of Ukraine accepted for the period until 2035 «Safety, energy efficiency, competitiveness» (edition dated August 18, 2017) No. 605. According to the science project «Intensification of mass transfer processes of oil processing and production of purified oil distillates» No. 0118U003968, «Research of processes of processing industrial and household waste into construction and lubricants» No. 0120U100597, launched and carried out at the Department of oil, gas and of solid fuel refining technologies" in the National Technical University «Kharkov Polytechnic Institute», Ukraine.

The program of laboratory research aimed at obtaining plastic lubricants from secondary raw materials can be presented as the sequence of the following steps:

1) Study and choosing the feedstock materials.
2) Feedstock pretreatment.
3) Thermo-mechanical dispersion of feedstock.

4) Cooling the reaction mixture.
5) Homogenization of the reaction mixture (if necessary).
6) Determination of rheological and tribological properties of the obtained plastic lubricants.
7) Comparison of the properties of the obtained plastic lubricants with existing industrial analogues.

It was found that according to the composition and properties used lubricating oil can be considered an optimal base oil. Crushed and thermo-mechanically processed thermoplastic materials were chosen as a thickener for the base oil. The feedstock was pretreated by removing of mechanical impurities (less than 0,007 % mass.) and centrifugal dehydration (the moisture content should not exceed 0.1 % mass.). Removing of mechanical impurities was carried out by gravity settling at 60°C and dehydration was provided by a settling type centrifuge at 6000 rpm. During 2 hours. The dispersed phase was prepared by washing, drying (moisture content at the level of 0.3 %) and grinding (particle size up to 2 mm) of waste products represented by low density polyethylene (LDPE), high density polyethylene (HDPE) and polypropylene (PP).

Thermo-mechanical dispersion of the components were performed in a reactor at 130-170°C (depending on the melting point of the polymer) and constant stirring (speed of rotation at about 1000-2000 rpm) during 2 hours. The reaction mixture was cooled to the temperature in an air cooler. The reaction mixture was homogenized by pressing it under a pressure of 15-20 MPa through a nozzle with a hole size of 100-150 μm.

Rheological properties of the obtained plastic lubricants were determined using a modernized rotational rheometer Rheotest 2.1 rotational rheometer (VEB MLW Prüfgeräte, Medingen, Germany) with coaxial cylinders. The rheometer was connected to a computer for an effective data processing. A Minichiller 280 OLÉ thermostat (Peter Huber Kältemaschinenbau, Offenburg, Germany) was used to maintain a constant temperature of studied materials. Plastic lubricants preconditioned for 20 minutes according to the requirements of [15] at t=0°C and in a rather wide range of $D_r$ values that was equal to 0 to 80 $s^{-1}$.

## 3. Results and discussion

The type of polymer thickener, in comparison with the viscosity of the base oil $\eta_o$, has a rather insignificant effect on the value of $\eta_m$ (Fig. 1). This can be explained by the fact that the investigated polymers have the same nature, that is, they belong to the same class of synthetic polymers obtained by polymerization of olefins – polyolefins.

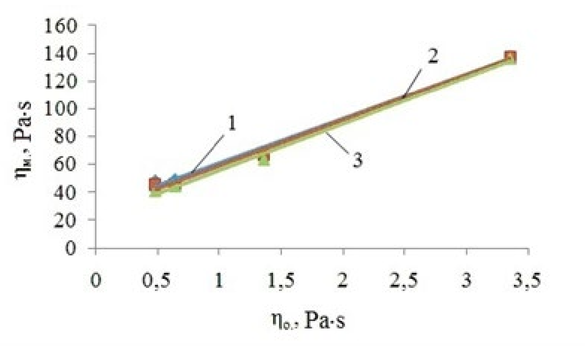


Figure 1. Effect of $\eta_o$ on the value of $\eta_m$ at 0°C and deformation velocity at 10$s^{-1}$: 1–HDPE; 2–LDPE; 3–PP.

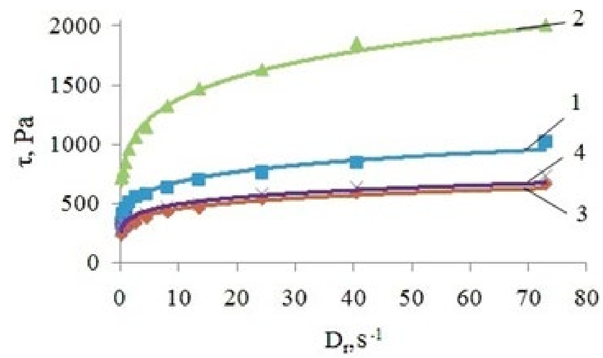


Figure2. Effect of $D_r$ on the value of $\tau_r$ for HDPE: 1 – group №1; 2 – group №2; 3 – group №3; 4 – group №4.

Considering quite gap difference in the obtained results we can use the following regression equation to predict the effect of $\eta_o$ of base oil on $\eta_м$ of plastic lube:

$$\eta_m = 32{,}767 \times \eta_o + 25{,}866;\ R^2 = 0{,}9819 \quad (1)$$

where $\eta_o$ – viscosity of the base oil; $\eta_m$ – dynamic viscosity of plastic lubricant.

This equation is relevant for values of $\eta_o$ for base oil at 0,45-3,5 Pa·s and can be applied for all groups of liquid lubricants from low-viscous hydraulic ones to high-viscous transmission oils that can be used as a dispersion medium in production of plastic lubes.

Results of rheology tests of plastic lubes, produced from secondary raw material, that contained certain amount of polymer thickener, are given at the Figs. 2-10.

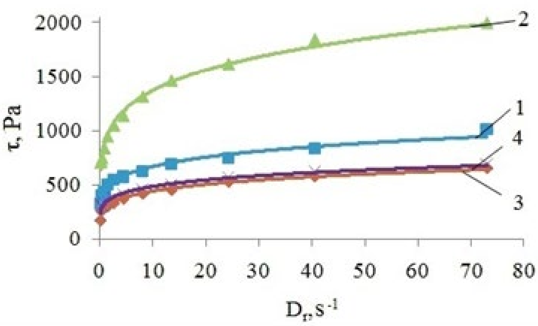


Figure 3. Effect of $D_r$ on the value of $\tau_r$ for LDPE: 1 – group №1; 2 – group №2; 3 – group №3; 4 – group №4.

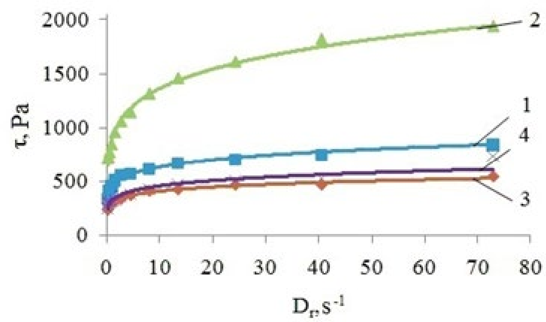


Figure 4. Effect of $D_r$ on the value of $\tau_r$ for PP: 1 – group №1; 2 – group №2; 3 – group №3; 4 – group №4.

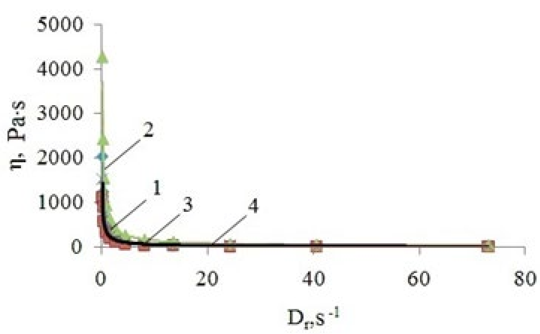


Figure 5. Effect of $D_r$ on the value of $\eta_m$ for HDPE: 1 – group №1; 2 – group №2; 3 – group №3; 4 – group №4.

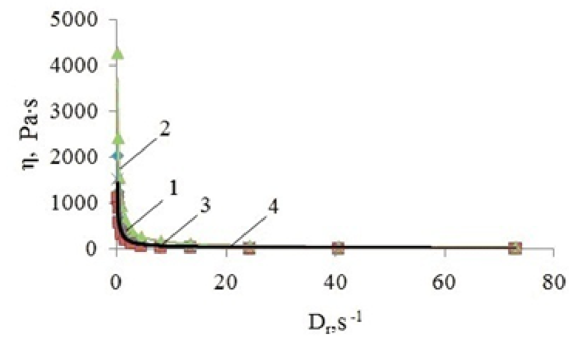


Figure 6. Effect of $D_r$ on the value of $\eta_m$ for LDPE: 1 – group №1; 2 – group №2; 3 – group №3; 4 – group №4.

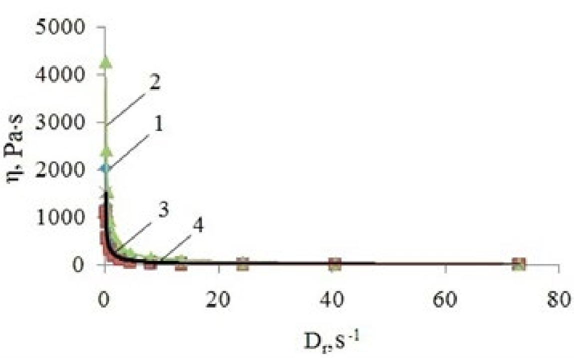


Figure 7. Effect of $D_r$ on the value of $\eta_m$ for PP: 1 – group №1; 2 – group №2; 3 – group №3; 4 – group №4.

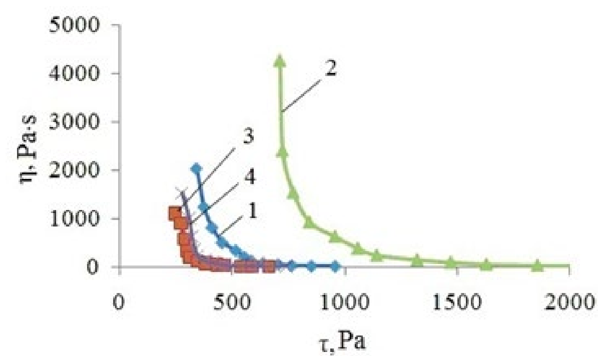


Figure 8. Effect of $\tau_r$ on the value of $\eta_m$ for HDPE: 1 – group №1; 2 – group №2; 3 – group №3; 4 – group №4.

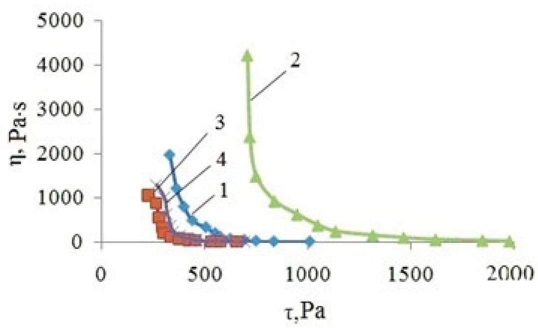


Figure 9. Effect of $\tau_r$ on the value of $\eta_m$ for LDPE: 1 – group №1; 2 – group №2; 3 – group №3; 4 – group №4

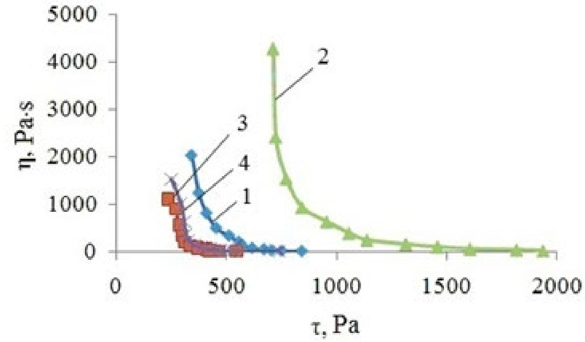


Figure 10. Effect of $\tau_r$ on the value of $\eta_m$ for PP: 1 – group №1; 2 – group №2; 3 – group №3; 4 – group №4

According to the requirements for plastic lubes their rheologic properties are defined at 0°C and speed of deformation at 10 $s^{-1}$. Therefore the value of $\tau_r$ can be calculated at $D_r$=10 $s^{-1}$ using the regression equation that consider effect of $D_r$ on $\tau_r$ (see Table 1). The obtained results and values of $\eta_m$ are given in the Table 2.

Table 1. Regression equations for polymer thickener.

| Group of base oil | Regression equation that consider effect of $D_r$ on $\tau_r$ at p=0,95 for polymer thickener | | | $R^2$ |
|---|---|---|---|---|
| | HDPE | LDPE | PP | |
| №1 | $\tau_r = 461,28 \times D_r^{0,1648}$ | $\tau_r = 449,54 \times D_r^{0,173}$ | $\tau_r = 456,39 \times D_r^{0,1433}$ | 0,97 |
| №2 | $\tau_r = 903,02 \times D_r^{0,184}$ | $\tau_r = 893,32 \times D_r^{0,1859}$ | $\tau_r = 900,93 \times D_r^{0,1792}$ | 0,98 |
| №3 | $\tau_r = 330,42 \times D_r^{0,1676}$ | $\tau_r = 313,47 \times D_r^{0,1622}$ | $\tau_r = 304,3 \times D_r^{0,1287}$ | 0,96 |
| №4 | $\tau_r = 346,55 \times D_r^{0,1547}$ | $\tau_r = 324,5 \times D_r^{0,1487}$ | $\tau_r = 289,36 \times D_r^{0,1861}$ | 0,98 |

Table 2. Results of measuring of $\tau_r$ and $\eta_M$ at 0°C and $D_r$ = 10$c^{-1}$.

| Group of base oil | Values of parameters for polymer thickener | | | | | |
|---|---|---|---|---|---|---|
| | HDPE | | LDPE | | PP | |
| | $\tau_r$, (Pa) | $\eta_M$(Pa·s) | $\tau_r$, (Pa) | $\eta_M$(Pa·s) | $\tau_r$, (Pa) | $\eta_M$(Pa·s) |
| №1 | 674 | 67,4 | 669 | 66,9 | 635 | 63,5 |
| №2 | 1380 | 138,0 | 1370 | 137,0 | 1361 | 136,1 |
| №3 | 486 | 48,6 | 455 | 45,5 | 409 | 40,9 |
| №4 | 495 | 49,5 | 467 | 46,7 | 444 | 44,4 |

The results of current research confirmed that production of plastic lubricants has to be controlled not by duration of process but by their quality characteristics that indicate the actual structure of lubricants and their physical and chemical properties in any moment of production stage. Analyzing the long-term practical experience in production and using of plastic lubricants in nodes and mechanisms, described in numerous scientific articles all around the world, we came to the following conclusion – all the principles and regularities of production of plastic lubricants from petroleum feedstock and soap thickener are completely suitable for production of lubricants from secondary raw materials. This statement was confirmed by the analysis of functions $\tau=f(D_r)$ and $\eta_m=f(D_r)$.

It was proved that effective dynamic viscosity of such lubricants directly depends on their structure and is characterized by the function $\eta_m=f(\tau)$, in which the value of $\tau$ varied in the range from 250 Pa to 2000 Pa. Therefore, it can be considered an important indicator which provides an effective technological control of production.

Effective dynamic viscosity of two-component plastic lubricants obtained both from secondary raw materials and from petroleum feedstock significantly depends on the viscosity of base oil. For instance, a lubricant produced from transmission oil SAE 85W-90 has a rather high value of 136-138 Pa·s and lubricant produced from low-viscous hydraulic oil HLP-46 has low values of ηm from 40.9 Pa·s to 48.6 Pa·s.

Type of polymer thickener has less significant effect comparing to the viscosity of the feedstock. Thus, with the content of 5 % polymer thickener in each composition, the value of the effective dynamic viscosity of the compositions varies within the following ranges: for HDPE thickener – from 48.6 Pa·s (for SAE15W-40, SAE10W-40 oils) to 138.0 Pa·s with (SAE 85W-90); for LDPE thickener – from 45.5 Pa·s (for SAE15W-40, SAE10W-40 oils) to 137.0 Pa·s (SAE 85W-90); for RP thickener – from 40.9 Pa·s (for SAE15W-40, SAE10W-40 oils) to 136.1 Pa·s (SAE 85W-90). High-density polyethylene (HDPE) has the biggest thickening effect on the base oil, and polypropylene (PP) has the least.

Considering directly the production of plastic lubricants from secondary raw materials it should be mentioned that the main technological operation of their production is dispersion of polymer thickener (HDPE, LDPE and PP) in a dispersion medium – base oil. At the same time, dispersion occurs at temperatures higher than the melting temperature of polymer and with continuous mechanical stirring [16].

Macromolecules of linear polymers have a shape of static ball in their balanced state but having been affected by external factors they turn into chain of macromolecules and get a spherical shape when return to the steady state. The mechanism of affect any external forces is the following. Any constant directed force applied to a polymer amorphous material (especially heated to a viscous state) should cause the formation of fragments of an ordered macromolecular structure due to the orientation of polymer macromolecules parallel to the vector of the applied force. According to the Le Chatelier-Brown principle, a thermodynamically balanced system reacts to any external disturbance in a way that minimizes the consequences of this disturbance [17]. Orientation of polymer macromolecules along the direction of interlayer friction reduces the coefficient of this friction (for liquid substances – of viscosity) and, therefore, minimizes mechanical impact on the dispersed system.

Limited factors of this process are elasticity of macromolecules, and chaotic thermal motion of heated macromolecules. When an external force affecting the balanced system (in our case it is energy of stirring $E_{st}$) exceeds the total elastic energy of macromolecules $E_{el}$ and thermal motion of macromolecular segments $E_{T.s.}$, a macromolecular structure gets arranged. When $E_{st} < E_{el.} + E_{T.s.}$ and moreover if $E_{el} = 0$, the system returns to the initial balanced state (Fig. 11).

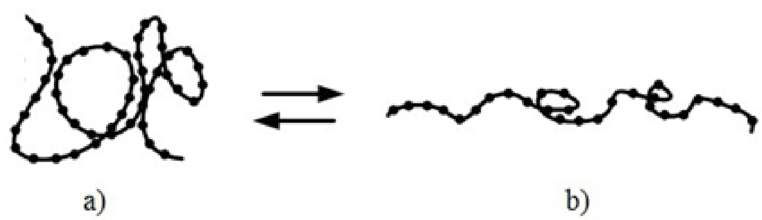


Figure 11. The model of influence of an external effect on a polymer macromolecule:
a) without any external affect; b) with an external affect.

If a polymer melts, thermal motion involves atoms and groups of atoms that are characterized by oscillating movements and segments that are characterized by translational and rotational movements [15]. In case if $E_{st}$ exceeds the strength threshold of macromolecules $E_s$, macromolecular associates get destructed and then the macromolecules themselves, which is followed by the dispersion of the polymer filler in the dispersion medium (Fig. 12).


Figure 12. Model of the effect of external mechanical forces on a polymer macromolecule: a) with an effect of external force; b) mechanical destruction.

It also becomes possible to produce components of thickener of anisometric fiber-like shape, which provides the formation of the spatial structure of lubricant and increase its stability. In other words, by regulating the power and rotation speed the mechanical stirring device, it is possible to achieve the formation of an arranged structure of plastic lubricant.

As the polymer gets destructed and distributes in the base oil, the viscosity of such system will continuously increase. This phenomenon can be used to control the dispersion of the thickener by volume of the dispersion medium. It is necessary to apply viscosity measuring sensors that work according to the principle given in [18] in the reactor-type apparatus. The proper placing of sensors in the apparatus for dispersing the polymer thickener is presented on Fig. 13.

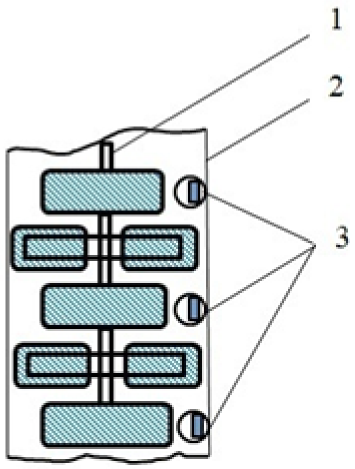

Figure 13. Scheme of placing of sensors in the apparatus: 1 – stirring device; 2 – apparatus casing; 3 – viscosity sensors.

According to the Fig. 13, the sensors are placed at different levels along the length of the device and are protected from mechanical damage by pipes. Flows of the mixture passing through the pipes are controlled by sensors that continuously measure the viscosity value. When a certain viscosity value of the mixture is reached, the operator of the device receives a signal (light, sound) about the end of the dispersing stage of the thickener. At the same time, the operator empties the device (the mixture is fed to the next stage of production) and pumps new components into the device.

## 4. Conclusions

The relevance of technological control in production of plastic lubricants based on their quality indicators has been substantiated. It has been shown that rheological parameters, and dynamic viscosity of plastic lubricants in particular, directly depends on their structure and is the indicator that mostly provides the technological control of their production.

Our research allowed to confirm the fact that the effective dynamic viscosity of two-component plastic lubricants produced from secondary raw materials, as well as conventional lubricants soap, significantly depends on the viscosity of the base oil. Effective dynamic viscosity of two-component plastic lubricants obtained both from secondary raw materials and from petroleum feedstock significantly depends on the viscosity of base oil. A lubricant produced from high-viscous transmission oil has a rather high value of 136-138 Pa·s and lubricant produced from low-viscous hydraulic oil HLP-46 has low values of ηm from 40.9 Pa·s to 48.6 Pa·s. Besides, the value of tangential shear stress from 250 Pa to 2000 Pa is also considered an important indicator of effective technological control of production.

Type of polymer thickener has an insignificant effect on the value of dynamic viscosity. It can be explained by the fact that the investigated polymers have the same origin and belong to the same class of synthetic polymers obtained by polymerization of olefins – polyolefins. But high-density polyethylene (HDPE) has the biggest thickening effect on the base oil, and polypropylene (PP) has the smallest.

A scheme of viscosity sensors location has been proposed. The presented scheme will allow to optimize the dispersing of polymer thickener by reducing the consumption of energy resources by monitoring the duration of technological process.

Effective dynamic viscosity can be used to control the technological process of production of plastic lubricants at refineries or petrochemical plants. Therefore, we developed a scheme of viscosity measurement sensors inside the main reactor for dispersing the polymer thickener in the dispersion medium – the base oil. This scheme is applicable for any petrochemical facility. It will provide a significant increase the efficiency of production of plastic lubricants and decrease of energy consumption and, as a result, reduce the cost of commercial lubricants and increase their competitive ability on the world market of petroleum products. According to that our further research will be focused on developing of technological schemes and processes that involve the described sensors and analyzers.

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