DEVELOPMENT OF MULTIPURPOSE PLASTIC LUBRICANTS AND STUDY OF THEIR RHEOLOGIC AND TRIBOLOGIC PROPERTIES

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1. INTRODUCTION

Vegetable oil (**VO$_S$**) used both in food industry and in technology acquired great practical consequence in all branches of economy. Some sorts of **VO$_S$** are used in lubricants, for example, the lubricant based on castor oil is indispensable in special situations with heavy duty conditions.

VO$_S$ have a great molecular mass which stipulates their low volatility even under high vacuum [1]. Their use as a disperse medium for efficient plastic lubricants (**PL$_S$)** intended for operation under high vacuum may be prospective.

Due to it anti-wear, anti-score and anti-friction properties of some vegetable oils have been investigated. Some oils were extracted from fruits stones of plums, cherries, apricots and from tomato and grape seeds as well, i.e. from the raw agricultural waste. Besides, olive, corn, sunflower oils and also peanut and rape seed oils have been investigated.

Basic parameters of **VO$_S$** are given in literature (table 1) [1-3].

Multipurpose plastic lubricants can be applied virtually to any friction units of mechanisms. As a rule these lubricants are operative in a wide range of temperatures loads, velocities and are water-resistant. They can replace many antifriction and special lubricants. In case the lubricant features low evaporability in vacuum, the range of its applicability becomes even wider.

Nowadays some **PL$_S$** for operation in vacuum are commercially available: **ЦИАТИМ-221** (GOST 9433-80) with polyethil-siloxane fluid as a disperse medium. It has a very low evaporability even at 423 K. It is used in friction units operating in high vacuum (10^{-1} – 10^{-10} Pa) but has poor anti-wear quality at sliding friction [4]. Besides, in high vacuum and in a wide range of temperatures in space devices (external equipment, space suits) are used **PL$_S$: ВНИИ НП–274н;–74ф;–293;–257;–270;–258** [5]. The polychlorine-syloxane fluid is used as a disperse medium for lubricants, **ВНИИ НП–274н and–274ф.** The fluid ensursefluids satisfactory antiwear properties and these **PL$_S$** recommended for in low loaded friction units. Lubricant, **ВНИИ НП–293** with a polymethyl-siloxane fluids as the disperse medium features even worse antiwear properties in comparison with lubricant, **ВНИИ НП–274н.** Besides, **PL$_S$ ЦИАТИМ-221** and **ВНИИ НП** mentioned above are very expensive.

2. EXPERIMENTAL PARTY

The investigation objective was to determine most effective **VO$_S$** which can be recommended as a disperse medium for plastic lubricant.

VO$_S$ lubricating properties was investigated with the use of a four ball friction machine (**FBFM**) in compliance with the method provided by GOST 9590-75. **RPM** of the upper ball was 1420 stepwise increase for axial load till the balls welded together, duration of each test was 10 seconds; the load of seizing was identified by a sharp increase of the worn spot diameter on the lower balls. For each axial load there were carried out three tests with a turn of balls but without their removing. Prior to each test a new portion of oil was added. The balls were made of steel **ШХ 9 of 60…62 HRC.** The oil temperature under test was 292...294 K. Worn spot diameter after the test was used for estimating the index of oil anti-friction properties. In the end each test the value of friction force in the ball contact was measured (with the help of a strain-gauge dynamometer coupled with the friction machine lower cup, an amplifier **TA-5** and an indicating micro-amperometer **M266M**). This value was converted into the friction coefficient value which was used for estimating the oil anti-friction properties. Using the results of tests and in compliance with GOST 9490-75 the values for score index, critical load and also load of welding together were determined.

All **VO$_S$** showed (table 2, 3) comparatively high anti-wear, anti-score and anti-friction properties.

Table 1. Lubricating properties of the VO_S

Parameters	Castor	From seeds				From fruits stones			From		
		Grape	Tomato	Rape	Peanut	Apri-cots	Plums	Cher-ries	Corn	Olive	Sun-flower
Density at the 15ºC, g/cm³	*0,962*	*0,91… 0,96*	*0,92… 0,93*	*0,91…0,92*	*0,91… 0,93*	*0,91… 0,92*	*0,91… 0,92*	*0,92… 0,93*	*0,924*	*0,917*	*0,924*
Temperature of solidification, ºC	*18… -10*	*-20… -10*	*-12… -7*	*-10… -4*	*-3… +3*	*-22… -12*	*-8… -5*	*-20… +16*	*-15… -10*	*-6… -2*	*-19… -16*
Tempera-ture of des-truction, ºC	*240…250*										
Molecular mass	*850….940*										
Iodine number	*84… 88*	-	-	*94… 106*	*83… 108*	-	-	-	-	*75… 88*	*127… 136*
Distillation number	*33,5*	-	-	*36,5*	*53*	-	-	-	-	*54*	*25*

Table. 2. Base parameters of VO_S

Vegetable Oil	Index of wear, I_w	Critical load, P_{cr}, N	Welding load toaster, P_w, N
From rape seeds	*43,5*	*790*	*2000*
From fruits stones cherries	*39,5*	*790*	*1580*
From fruits stones plums	*36,0*	*890*	*1410*
From sun flower	*35,4*	*790*	*1580*
From seeds tomato	*35,0*	*790*	*1410*
Castor	*34,8*	*790*	*1410*
From seeds grape	*33,2*	*790*	*1410*
Olive	*33,1*	*790*	*1410*
From fruits stones apricots	*32,5*	*630*	*1410*
From seeds peanut	*32,0*	*790*	*1410*

Probably, it accounts for the fact all those lubricants are based on treacil-glycerols which in the process of friction under high temperatures in the contact zone and being oxidized by air oxygen form peroxide compounds, oxy-acids and products of polymerization.

The values of iodine and distillation numbers indicate that the rape seeds oil has a satisfactory tendency to solidification and polymerization.

The analysis of test causes a conclusion that the best anti-wears, anti-score and anti-friction properties were manifested by the rape seeds oil which is the cheapest and thus can be recommended to be used as a disperse medium for preparing multipurpose plastic lubricant.

We developed a formulation where the rape seeds oil (GOST 8988-77) was used as a disperse medium and the lithium soap of the **12-oxystearic acid** as a stiffener (disperse phase) with obligatory introduction of the **antioxidant "Naftam -2"** (GOST 3079) and a solidifying additive – **polyisobutylene П-20** (ТУ 38 103-257-80) of the lubricant [6]. There were prepared five compositions with different proportions of four above mentioned components (weight %).

The technology of preparing the above mentioned compounds included the use of a high temperature method – the maximal temperature was 478 K, operations ensuring complete saponification of 12-oxystearic acid; neutralization of the rape seeds oil; formation of a slightly alkaline lubricant (**0,1% NaOH**) and a homogenized product.

Rheologic properties of lubricant with different compositions were determined with the use of methods in compliance with certain state standards: the temperature of drop falling

Table 3. Tribologic properties (worn spot diameter, mm/friction coefficient value) of VO_S

Axial load. P_{ax}, N	*Castor*	*From seeds peanut*	*From fruits stones apricots*	*From fruits stones plums*	*From seeds tomato*	*From seeds grape*	*From seeds rape*	*From fruits stones cherries*	*Sunflower*	*Olive*	*Corn*
200	0,39 / 0,070	0,38 / 0,099	0,39 / 0,099	0,39 / 0,099	0,36 / 0,099	0,44 / 0,110	0,31 / 0,099	0,34 / 0,099	0,37 / 0,100	0,38 / 0,099	0,37 / 0,099
250	0,40 / 0,070	0,41 / 0,087	0,41 / 0,095	0,39 / 0,095	0,36 / 0,095	0,46 / 0,100	0,35 / 0,095	0,40 / 0,120	0,39 / 0,100	0,42 / 0,088	0,39 / 0,088
320	0,43 / 0,070	0,45 / 0,093	0,44 / 0,120	0,42 / 0,093	0,38 / 0,098	0,46 / 0,090	0,35 / 0,093	0,43 / 0,120	0,40 / 0,110	0,43 / 0,082	0,42 / 0,083
400	0,45 / 0,070	0,46 / 0,099	0,46 / 0,110	0,44 / 0,990	0,38 / 0,099	0,47 / 0,090	0,40 / 0,084	0,43 / 0,110	0,43 / 0,099	0,45 / 0,075	0,43 / 0,075
500	0,47 / 0,060	0,46 / 0,079	0,56 / 0,120	0,47 / 0,087	0,57 / 0,130	0,48 / 0,120	0,43 / 0,075	0,47 / 0,099	0,45 / 0,099	0,45 / 0,080	0,44 / 0,099
630	0,61 / 0,090	0,47 / 0,079	0,63 / 0,130	0,55 / 0,095	0,85 / 0,140	0,51 / 0,140	0,50 / 0,074	0,50 / 0,098	0,59 / 0,130	0,70 / 0,110	0,64 / 0,130
790	0,66 / 0,110	0,77 / 0,109	0,74 / 0,110	0,57 / 0,083	0,86 / 0,130	0,76 / 0,120	0,77 / 0,073	0,67 / 0,100	0,73 / 0,110	0,74 / 0,100	0,75 / 0,105
1000	0,83 / 0,110	0,84 / 0,099	0,96 / 0,110	0,82 / 0,099	0,92 / 0,120	0,82 / 0,120	0,78 / 0,074	0,79 / 0,120	0,89 / 0,099	0,86 / 0,090	0,82 / 0,100
1120	0,98 / 0,110	0,89 / 0,110	0,89 / 0,120	0,93 / 0,110	0,92 / 0,110	0,85 / 0,110	0,84 / 0,079	0,89 / 0,120	0,90 / 0,110	0,97 / 0,090	0,89 / 0,090
1260	0,98 / 0,110	*Welding*	1,01 / 0,130	0,96 / 0,100	0,93 / 0,110	1,10 / 0,099	0,87 / 0,780	1,00 / 0,130	1,07 / 0,100	1,09 / 0,110	0,9 / 0,090
1410	*Welding*		*Welding*	*Welding*			1,0 / 0,085	1,10 / 0,130	1,3 / 0,09	*Welding*	
1580							1,10 / 0,110	1,15 / 0,130	*Welding*		
1780							1,41 / 0,12	*Welding*			
2000							*Welding*				

to GOST 6793-74; penetration at 25ºC under stirring to GOST 5346-78; viscosity to GOST 7163-84 at the average gradient of deformation rate 10 s^{-1} at temperatures –30ºC, –20ºC, 0ºC, +20ºC; the lubricant ultimate strength at temperatures 20ºC, 50ºC, and 80ºC to GOST 7143-73 with the use of a plastometer **K-2.** The colloid stability was determined with the use of the method for squeezing oil in the **TGA** instrument at the room temperature to GOST 7142-74.

Evaporability was determined by measuring the lubricant mass loss under rated conditions in compliance with GOST 9566-60; different compositions of lubricant were applied into evaporating cups as a layer 1 mm thick (weight about 0,3 g), the cups were put on a heating plate and heated at 150ºC during one hour.

The mass fraction of free organic acids was determined with the use of methods to GOST 6707-76. The lubricant corrosion effect on copper plates was determined to GOST 9080-77.

Mechanical stability of the lubricants with different composition was determined with the use of the **"SHELL"** instrument (ASTM-D-1831) in the curse of their destruction during 2 hours at 4ºC with the following determination of the lubricant ultimate strength at 50ºC.

Anti-score and anti-wear properties of the lubricants with different compositions were determined with the use of the four-ball friction machine in compliance with GOST 9490-75. There were critical load, welding together load and score index determined.

Vaporability of PL_S with different composition, **Литол-24** and **ЦИАТИМ-221** and special lubricant [7, 8] was determined with the use of vacuum station **ВУП-4**, where there was created a vacuum of $4 \cdot 10^{-5}$ mm Hg at 298 K during 30 minutes.

The lubricants were applied into evaporating cups as layers 2,5 mm thick. Vaporability was determined by the lubricant mass loss with the use of an analytical balance, model ВЛО-200М.

Basic parameters of received PL_S are presented in table 4, which also includes data on well-known lubricants: **Литол-24, ШРУС-4, ЦИАТИМ-222, ВНИИ НП-274ф** given in [4].

3. DISCUSSION OF RESULTS

The prototype for their lubricant worked out was **Литол-24**. In comparison with it the lubricant worked out has an advantage: it has a very good viscosity and temperature characteristic with allows it to be operable in a wide range of temperatures.

Obviously, the low limit of its use is about –40ºC as the lubricant is considered operable up to viscosity 20 000 Pa [4] at the deformation rate gradient 10 s^{-1}. The upper temperature limit is 120 (130ºC). Another advantage of the PL worked out is also an easy process of its preparation; only a slight foaming was registered at the stage of saponification.

The mass fraction of free organic acids for this lubricant meets the norm to mg КОН/I g approved for the lubricant **ШРБ-4** manufactured industrially [4]. As for the colloid stability, it does not exceed in three compositions of lubricant the rated value of 12% for the majority of lubricants.

Lubricating properties of the worked out lubricant are much better than for **Литол-24** and **ЦИАТИМ -221.** The comparison of the lubricant ultimate strength at 50ºC prior to test in the **"SHELL"** instrument and after it shows amount of destruction, i.e. the lubricant features a good mechanical stability. The lubricant vaporability at 150ºC during one hour is very low in comparison with a whole set of multipurpose lubricants produced industrially and **ЦИАТИМ-221** as well.

A comparative test under vacuum ($4 \cdot 10^{-5}$ mm Hg) shows an advantage of the worked out PL over **Литол-24** and **ЦИАТИМ-221**. PL_S of optimal compositions manifest a more lower vapor ability than **PL_S** under use nowadays.

The lubricant can be used in heavy duty friction units and also in the units where it comes into contact with rubber parts.

On the base of plastic lubricant [6] has been developed lubricant for cold stamping [7] has high lubricant characteristics and allows to increase 1,5…2,0 times the firmness of the working details of the press tools, especially on the separation operations.

This type lubricant consists of product of interaction of kaprolaktam with copper hydrate, kerosene, sulfur as disperse medium the plastic lubricant [6].

Besides, on the base of plastic lubricant has been developed lubricant for hinges of equal angle velocities [8], used in the cars and others mechanics, including vacuum, consists of: rape seed oil, lithium soap of the 12-oxystearic acid; antioxidant and viscous dopants and also the antifriction and antiscore additives of disulfide of molybdenum (**"Motimol Pulver").**

This composition provides: high level of strenght, colloidal stability, low vaporability, low vaporability at the 150ºC and under the vacuum on the level of $4 \cdot 10^{-5}$ mm Hg, high mechanical stability, low index of destruction and high critical load in comparison with its prototype **ШРУС-4** wide used in the cars.

Besides, we a electrically conducting lubricant [9] was developed, which has high electro conductivity and very good rheological and lubricating properties.

The lubricant represented a composition of plastic lubricant [6] and powder copper **ПМС-20** in quantity 8…12 weight %.

Taking into account the high rheologic a tribologic properties, the developed plastic lubricants can be extensively applied in the construction of machinery, electropower engineering, motor transport, tractors and agriculture machines. Moreover, the lubricants have low volatility in vacuum conditions , therefore it can be used in friction units of altitude aviation.

Rubber stuffing elements used in combination with proposed lubricants will be characterized by higher durability due to the fact that vegetable oils, in comparison to the mineral ones, do not affect to the same extent the rubber.

The industrial production of proposed oils can be organized at the production facilities of Moldova Republic, taking into account fact that during next 1-2 years in Moldova high yields of oil from rape seeds are expected. Correspondingly, Republic of Moldova will be able to satisfy its necessities of lubricants without importing similar types of materials from abroad.

Table 4. Rheologic and tribologic properties of lubricants

Parameters	*Lubricants*							
	Литол-24 (Russia)	*Patent Nr.778 (MD)*	*ШРУС-4 (Russia)*	*Patent Nr.1065 (MD)*	*Patent Nr.907059 (USSR)*	*Patent Nr.1029 (MD)*	*ЦИАТИМ -221 (Russia)*	*ВНИИНП -274Ф (Russia)*
1. Temperature of drop failing, ºC	*185*	*180…185*	*186*	*184*			*200*	*175*
2. Viscosity, Pa·s, at the average gradient of deformation 10 s⁻¹ at temperatures, **– 30ºC** **– 20ºC** **0ºC** **+ 20ºC**	 *800-1500* *300-600* *200-250* *80-120*	 *683-1631* *362-673* *231-382* *53-250*	 *<1800* *720* *<250* *200*	 *730-1190* *402-545* *262-273* *94-310*			 *–50ºC to 400-750* *-150ºC to 120-250* *80-200* *40-100*	 *– 50ºC to 300* *80-100* *50-65*
3. The ultimate strength Pa, at temperatures, **+ 20ºC** **+ 50ºC** **+ 80ºC**	 *500-1000* *400-600* *200-600*	 *340-900* *260-800* *175-600*	 *300-700* *≈630* *>150*	 *520-830* *320-450* *210-280*			 *250-450* *120-250* *60-150*	 *270-370* *70-300* *140-150*
4. Colloidal stability, %	*8-12*	*10-11*	*<16*	*10-11*			*3-7 (3N)*	*18 (3N)*
5. Vaporability under 4·10⁻⁵ mm Hg in during 30 min and 20ºC, %	*0,31*	*0,05-0,07*	*0,3-0,4*	*0,05-0,07*			*0,136*	*–*
6. Vaporability at 150ºC during 1 hour, %	*2-3*	*0,06-2,0*	*0,3-0,4*	*0,05-0,07*			*2,0*	*0,5*
7. Mechanical stability, the ultimate strength, at 50ºC prior to test in the „SHELL" instrument (ASTM-D-1831), Pa	*–*	*290…420*	*–*	*310…340*			*–*	*–*
8. Index of destruction, %	*10...60*	*15…25*	*60*	*10…25*			*75…85*	*25…40*
9. Lubricating properties: **Critical load, P_{cr}, N** **Welding load together, P_w, N** **Index of wear, I_w**	 *630* *1410* *22-28*	 *940* *1780* *33-38*	 *1410* *3500* *30-36*	 *1780* *3500* *35-40*	 *1280* *2000* *24-30*	 *1610* *3180* *36-40*	 *280-340* *1100-1780* *–*	 *–* *–* *–*
10. Temperature range	*From –40ºC to 120 (130ºC)*						*– 60ºC to 150ºC*	*– 60ºC to 150ºC*

4. CONCLUSIONS

1. The use of the worked out **PLs** in different branches of industry can be commercially efficient as it will cause a reduction of wear in friction parts, an increase of intervals between lubricant changes and thus a sharp reduction of material and labor expenditures.

2. Due to the using of rape seeds oil as a disperse medium in plastic lubricant the last are ecologically pure.

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