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## POSSIBILITY OF APPLICATION FOR ETHERS OF ACONITE ACID AS AW/EP ADDITIVE

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**Abstract:** *The possibility of using substances containing only carbon, hydrogen and oxygen as antiwear additives for lubricating oils has been investigated. Studies have been conducted on the example of ethers of aconite acid and hexadecyl alcohol. This compound was chosen as a promising AW/EP additive for biodegradable greases based on vegetable oils. In addition, the anti-wear mechanism of substances that do not contain phosphorus, sulfur, chlorine, metals and other heteroatoms is very interesting. Aconite acid hexadecyl ester was added to rapeseed oil, to petroleum oil without additives, and to standard gear oil. Antiwear properties of the obtained compounds were identified and presented as results. Measurements were carried out both in laboratories of SUSU and at Sofia University at several stands under various friction conditions. It was established that the test additive exhibits anti-wear properties in all three lubricating oils. With the introduction of additives in rapeseed oil, wear is reduced by 25 ... 50% depending on the test conditions. The wear reduction is more significant in steel-bronze contacts than in steel-steel and steel-iron contacts. With the introduction of additives in hydrocarbon oil without additives, anti-wear effect is only slightly lower than with the introduction of ZDDP. Anti-wear effect increases with increasing load (contact pressure), as well as for ZDDP. The most pronounced effect for both vegetable and petroleum oil, obtained by testing the method of four balls according to the technique of ASTM D 4172. When hexadecyl aconite acid is added to gear oil (GL-5 standard) containing ZDDP and EP additives, wear is reduced to the same extent as when it is added to oil without additives. The welding load increases slightly. This suggests that the studied additive does not compete with ZDDP and can be used in mixed composition. The absence of heteroatoms in the studied additive indicates an adsorption mechanism of anti-wear action. The study results indicate the prospects for the use of esters of polyatomic acids as AW/EP additives for biodegradable lubricating oils and oils for food production equipment.*

**Keywords:** *antiwear additives, extreme pressure additives, aconite acid, biodegradable lubricating oil, friction machine.*

### 1. INTRODUCTION

In connection with the increase in the negative impact of industrial production, electricity production and transport on the nature and environment of humans, considerable attention has recently been paid

to the environmental aspects of the functioning of technical devices. Major source of environmental danger and anthropogenic impact on natural systems are vehicles with internal combustion engines. Pollution is generated not only in the processes of extraction and processing of natural

hydrocarbons and fuel combustion. Lubricants and hydraulic fluids that enter the soil and water are also significant sources of pollution. This especially applies to agricultural machinery and water transport. This circumstance is the cause of attention to the development and use of biodegradable lubricants. Synthetic esters and products of chemical modification of vegetable oils are considered promising. But special attention is paid to vegetable oils, as environmentally friendly and renewable materials.

Vegetable oils have fundamental limitations on the scope of use except advantages. The most significant disadvantage is low oxidation resistance. Reduced resistance to oxidation of vegetable oils due to their chemical composition [1]. As a rule, to improve this parameter, oils with a low content of unsaturated acids are used, for example, castor oil. However, castor oil is inferior to rapeseed and other oils for antiwear properties. For long-term operation at high temperatures, resistant synthetic esters are more suitable. However, the ability of synthetic esters to biodegradation in the natural environment decreases with increasing thermal stability. The same dependence is observed for anti-wear properties. For applications such as the ships engines used in inland waters (rivers and lakes), vegetable oils are the preferred lubricants.

The second disadvantage is the relatively low anti-wear properties. Despite the fact that the antiwear and antifriction properties of vegetable oils are higher than those of petroleum and synthetic base oils without additives [2 - 6], they are significantly lower than those of lubricants containing additive packages. It should be noted that hydrocarbon-based lubricants have come a long way of improvement, including cleaning technologies from components that are not resistant to oxidation, and the development of effective multifunctional additives, including anti-wear, extreme pressure and antifriction.

The anti-wear properties of vegetable oil-based lubricants can also be significantly enhanced by the addition of additives. However, the use of conventional anti-wear additives developed for hydrocarbon oils, such as ZDDP or triphenyl phosphorothionates, is either impossible or impractical. This is due to the fact that the effectiveness of additives varies significantly in base oils of different chemical composition. In addition, ZDDP can be destroyed by chemical interaction with the molecules of vegetable oils. And finally, the introduction of components containing sulfur, phosphorus and toxic metals, significantly reduces the environmental benefits of vegetable oils. The search for special additives for vegetable oils began only recently.

Three methods of enhancing anti-wear properties are used: introduction of solid lubricant components to lubricating oil, for example [7–11], chemical modification of vegetable oils, for example [12, 13], and introduction of oil-soluble antiwear components, for example [14–20]. The use of solid lubricant components is limited to greases. The use of nanosuspensions of metals and other inorganic components that are resistant to sedimentation affects the filterability of oils. This is an important parameter of oils for hydraulic systems and engines. In addition, operational impacts lead to a change in the composition of the oils. The oxidation of the oil and the presence of water can significantly affect the stability of the nanosuspensions. Chemical modification of vegetable oils, for example, the formation of thioesters, reduces the ability to biodegrade and can lead to the formation of corrosive products during operation.

From the standpoint of storing such advantages of vegetable oils as non-toxicity, no odor, ease of production, the authors consider the increase in the tribological parameters by adding fully soluble functional additives to be the most promising. This method gave excellent results for

hydrocarbon-based lubricants. Some examples of such studies are given in papers [14–20]. Despite the successful use of some AW / EP components developed for hydrocarbon oils [16–18], a number of papers note that the effectiveness of additives is different in hydrocarbon and vegetable oils. Increased adsorption of vegetable oil molecules and esters on the metal surface prevents the adsorption of molecules of traditional AW / EP components. In this regard, the most effective are more polar substances than those used in hydrocarbon oils.

The main fields of application of biodegradable oils are water transport engines used in closed reservoirs, hydraulic systems and transmissions of agricultural machinery, equipment for food processing. The principles for the development of anti-wear additives for these applications are associated primarily with the adsorption mechanism of action of the additives. Efficiency should be expected from oil-soluble surfactants containing a massive polar group, prone to adsorption on the metal surface, and quite long hydrocarbon radicals, providing intermolecular interaction with the base oil.

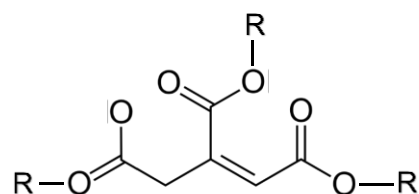
This paper considers the possibility of using esters as an AW additive. These esters have chemical and environmental properties that are as close as possible to vegetable and animal fats. At the same time, they are characterized by an increased tendency to adsorb on metal surfaces. Vegetable oils are esters of monobasic organic acids and glycerin triatomic alcohol. In this work, as an additive, a tribasic organic acid and a monohydric alcohol was tested. The presence of tribasic acid, forming stable complexes with metals, suggests an increased ability to adsorb. The similarity of the chemical nature of additives and fats, as well as the choice of non-toxic components, suggest a good compatibility of additives with vegetable oil and the absence of a negative impact on biodegradability and toxicity.

A test determination of the antiwear efficacy of aconitic acid ester and hexadecanol-1 in hydrocarbon oil without additives, automobile transmission oil and rapeseed oil was carried out. Testing was performed to verify the compatibility of the developed additive with standard anti-wear and extreme pressure additives. The focus was on determining the effectiveness of the additive in rapeseed oil.

## 2. EXPERIMENTAL RESEARCH

### 2.1 Synthesis of the additive

Citric acid was used as tribasic acid. Hexadecanol-1 was used to ensure the solubility of ether and the products of its partial decomposition in oils. A mixture of acid and alcohol, taken in a molar ratio of 1: 3, was heated to 160...190 °C. The reaction was carried out for 2...3 hours with occasional stirring. Under these conditions, the esterification takes place completely and does not require a catalyst. As a result of heating, citric acid separates water and turns into aconitic acid. The final product is a full ester of Hexadecanol and aconitic acid. The injected additive was called AAE-additive. The structural formula is presented in Figure 1. The resulting product has a wax consistency, the melting point is 60...65 °C. It is soluble in hydrocarbons, but insoluble in water.



**Figure 1.** Formula aconitic acid trihexadecyl ester, where R is the residue of alcohol C<sub>16</sub>H<sub>33</sub>

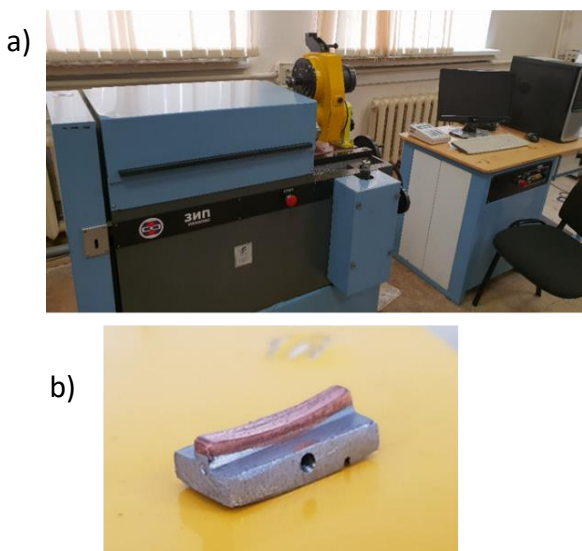
### 2.2 Determination of the anti-wear properties of the AAE additive in petroleum lubricating oil without additives

Industrial oil without additives (viscosity grade ISO 32) was used as the base oil. The AAE-additive was administered in an amount

of 3% by weight. For comparison, zinc dihexadecyl dithiophosphate (ZDDP) was used, which included the same hydrocarbon radicals as the additive under study. ZDDP was introduced into the base oil in an amount of 2% by weight.

The tests were carried out on the machine friction II-5018. The tests were carried out on the machine friction II-5018. Unlike the universal friction machine MTU-1 [21], the friction machine II-5018 allows to carry out tests under the conditions of contact "roller block". The diameter of the roller is 90 mm, the material is steel. The block is made of the liner of the tractor bearing, the material is bronze, the dimensions of the working surface are  $24 \times 2$  mm. Test conditions: roller rotation speed  $500 \text{ min}^{-1}$ , load range 1000 N. Oil supply is drop, at the entrance to the friction contact, 0,2 ml/s.

The values of the friction coefficient  $\mu$  were calculated based on the measured friction moment  $M$  at each load value  $F$ . The contact pressure  $P$  was calculated based on the load value and the contact area. Temperature  $T$  was measured by a thermoelectric transducer inserted into the side opening of the block. A general view of the friction machine II-5018, block image and contact assembly, is shown at Figure 2.

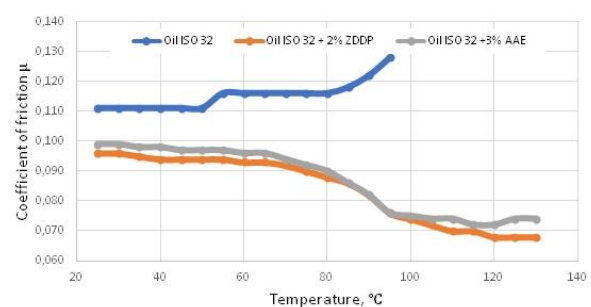


**Figure 2.** General view of the friction machine II 5018 (a), block (b)

The measurement results are presented in Table 1 and in Figure 3.

**Table 1.** Measurement results. The dependence of the coefficient of friction  $\mu$  on the temperature when lubricated with oil without additives, the same oil with the addition of 3% aconitic acid trihexadecyl ester and 2% ZDDP

Friction unit temp., °C	Friction coefficient $\mu$		
	Industrial Oil ISO 32	ZDDP	AAE
25	0,111	0,096	0,099
30	0,111	0,096	0,099
35	0,111	0,095	0,098
40	0,111	0,094	0,098
45	0,111	0,094	0,097
50	0,111	0,094	0,097
55	0,116	0,094	0,097
60	0,116	0,093	0,096
65	0,116	0,093	0,096
70	0,116	0,092	0,094
75	0,116	0,09	0,092
80	0,116	0,088	0,09
85	0,118	0,086	0,086
90	0,122	0,082	0,082
95	0,128	0,076	0,076
100	contact	0,074	0,075
105	-	0,072	0,074
110	-	0,07	0,074
115	-	0,070	0,072
120	-	0,068	0,072
125	-	0,068	0,074
130	-	0,068	0,074



**Figure 3.** Effect of ZDDP and AAE on the dependence of the friction coefficient on temperature in a conformal slip contact

Due to the dependence of the friction coefficient on the temperature and the complexity of contact temperature control, the measurement of the dependence of the friction coefficient on the load was carried out at three load values. The load was constant during each measurement. The moment value

was recorded when the temperature reached 80 °C. Before performing each measurement, the surface of the roller was polished to achieve a roughness of  $R_a = 0,02$ . The lubricant used was the same oil and the same additive concentrations as in the measurements described above. The measurement results are shown in Table 2.

**Table 2.** Measurement results. The dependence of the friction coefficient  $\mu$  on the load when lubricating with oil without additives, the same oil with additives of 3% aconitic acid trihexadecyl ester and 2% ZDDP

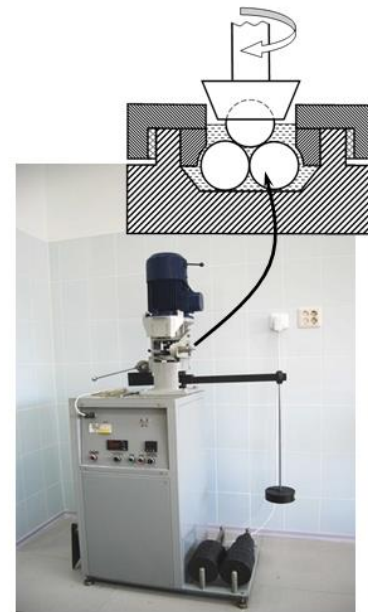
Load, N	Friction coefficient $\mu$		
	Industrial Oil ISO 32	ZDDP	AAE
200	0,054	0,046	0,052
500	0,070	0,062	0,065
1000	contact	0,084	0,085

### 2.3 Determination of the anti-wear properties of the AAE additive in gear oil

Transmission oil LUKOIL API GL-5, SAE 75W-90 was used as the base oil. This oil contains antiwear and extreme pressure additives. The additive ZDDP was administered in an amount of 2% by weight, the additive AAE was introduced in an amount of 3% by weight.

Antiwear properties were determined by the four ball method on a standard ChMT-1 friction machine complying with the requirements of ASTM D 4172 [21] according to the ASTM D 4172 method at a rotation speed of 1200 rpm (0,46 m/s). The normal load is 392 N, the temperature of the oil bath is  $40 \pm 5$  °C. The balls were made of steel, the hardness is HRC 64-66, the diameter is 12,7 mm. An optical microscope with an accuracy of 0,01 mm was used to measure wear spots. A general view of the friction machine is presented in Figure 4.

For the original gear oil made 6 measurements. For oil with additives ZDDP and AAE, 3 measurements were carried out. The measurement results were averaged. The measurement results are shown in tables 3 - 6. The type of wear marks is shown in Figure 5.



**Figure 4.** The view of the four-ball friction machine and the scheme of the friction unit

**Table 3.** The wear trace diameter for LUKOIL GL-5 SAE 75W-90 oil

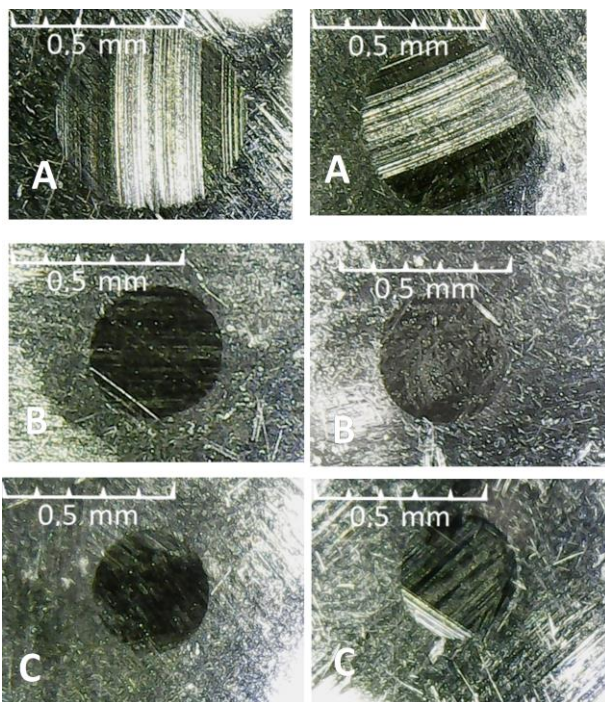
No	1	2	3	4	5	6
The wear trace diameter, mm	0,44	0,46	0,46	0,45	0,46	0,43
	0,44	0,47	0,46	0,45	0,46	0,43
	0,45	0,46	0,46	0,45	0,46	0,44
	0,44	0,46	0,45	0,45	0,46	0,44
	0,43	0,42	0,45	0,45	0,44	0,41
	0,43	0,42	0,45	0,45	0,47	0,42
	0,44	0,43	0,45	0,45	0,44	0,43
	0,44	0,43	0,45	0,45	0,45	0,45
	0,43	0,44	0,45	0,45	0,45	0,43
Average value mm	0,452					

**Table 4.** The diameter of the wear trace for oil LUKOIL GL-5 SAE 75W-90, containing 2% ZDDP

No	1	2	3
The wear spot diameter, mm	0,335	0,346	0,335
	0,342	0,344	0,325
	0,348	0,345	0,329
	0,35	0,345	0,331
	0,345	0,34	0,339
	0,347	0,332	0,353
	0,348	0,332	0,357
	0,356	0,333	0,353
	0,347	0,339	0,338
Average value, mm	0,344		

**Table 5.** The diameter of the wear trace for oil LUKOIL GL-5 SAE 75W-90, containing 2% ZDDP

No	1	2	3
The diameter of the wear trace, mm	0,328	0,335	0,322
	0,332	0,334	0,328
	0,327	0,325	0,328
	0,326	0,328	0,332
	0,332	0,332	0,33
	0,333	0,332	0,323
	0,324	0,326	0,326
	0,328	0,334	0,328
Average value, mm	0,328		



**Figure 5.** Type of wear spots when tested according to the four ball method: A - when lubricated with LUKOIL GL-5 SAE 75W-90 oil, B - the same oil with 3% AAE added, C - the same oil with 2% ZDDP added

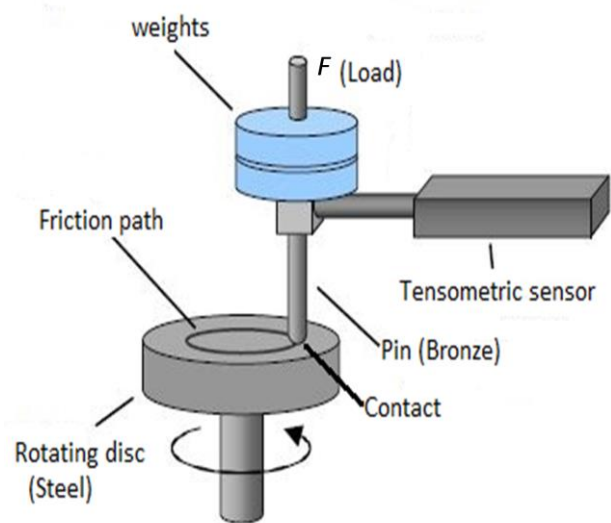
#### 2.4 Determination of the anti-wear properties of the AAE additive in gear oil

Tests were carried out at the Sofia Technical University (Bulgaria). To determine the presence of antifriction action of aconitic acid trihexadecyl ester (AAE) in a flat contact, technical rapeseed oil was used. The AAE addition was added in an amount of 4% by weight.

**Table 6.** The results of measurements of wear marks by the four-ball method (axial load is 392 N) for LUKOIL GL-5 SAE 75W-90 oil; and the same oil containing additional additives ZDDP and AAE

No	Lubricant	Average wear diameter, mm	Average contact patch area, mm <sup>2</sup>	Minimum contact pressure reached during testing, MPa
1	LUKOIL GL-5	0,452	0,204	640
2	LUKOIL GL-5 + 3% AAE	0,344	0,118	1110
3	LUKOIL GL-5 + 2% ZDDP	0,328	0,108	1210

The measurements were performed on a laboratory device "Fixed Pin - Rotating Disk." The device is shown in Figure 6.



**Figure 6.** Scheme of the device for measuring the friction coefficient when lubricated with rapeseed oil with or without additive

A pin is a cylindrical specimen made of BrO10F1 tin bronze. The diameter is 19 mm. The rotating disk is made of alloy steel with a hardness of HRC56,9. The pin is fixed in the device, which can move freely in a plane parallel to the disk plane and along the normal to the surface. The pressing force  $F$  to the disk is created by placing weights on the device for

the pin fastening. A tensometric sensor attached to the fastening of the pin.

Using the device shown in Figure 6, the friction force  $F_t$  acting on the pin from the disk was measured. The friction force was measured with an accuracy of 0,1 N. Each experiment at each load value was performed at the same values of time (friction path), disk rotation speed, and ambient temperature. The lubricant was applied to the contact by drip method (drip lubrication) at a rate of 30 drops/min. The experiment parameters are shown in Table 7.

**Table 7.** Parameters of the experiment

No	Parameters	Value
1	Load	$F_1 = 60 \text{ N}, F_2 = 100 \text{ N}, F_3 = 140 \text{ N}, F_4 = 220 \text{ N}$
2	Nominal contact area	$A_a = 283,3 \text{ mm}^2$
3	Nominal contact pressure	$P_{a1} = 21,2 \text{ N/cm}^2, P_{a2} = 35,3 \text{ N/cm}^2, P_{a3} = 49,5 \text{ N/cm}^2, P_{a4} = 77,7 \text{ N/cm}^2$
4	Speed of rotation	$n = 95 \text{ min}^{-1}$
5	Sliding speed of the center contact	$V_c = 0,89 \text{ m/s}$
6	Initial rape oil temperature	$T = 21^\circ \text{C}$
7	Ambient temperature	$T = 21^\circ \text{C}$

The friction coefficient was calculated as the ratio of the friction force to the normal load (the force of pressing the pin to the disk). Rapeseed oil and rapeseed oil containing 4% aconitic acid trihexadecyl ester (AAE-additive) were used as a lubricant. The temperature of the lubricant supplied to the friction contact is equal to the ambient temperature 21 °C.

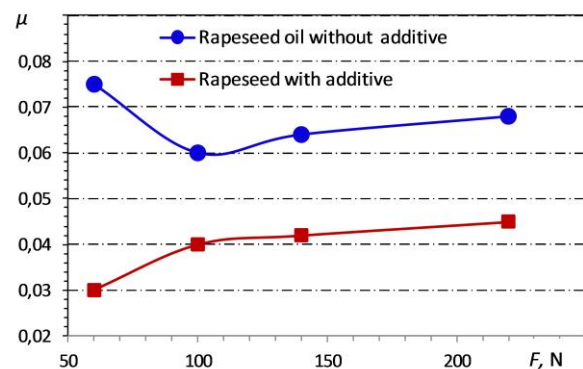
Table 8 presents the results of measurements of friction coefficients at four load values. Figure 7 shows the dependence of friction coefficients on the load.

The diagram of the relative change of the friction coefficient  $\bar{\mu}$  under different normal loads is shown in Figure 8. The value  $\bar{\mu}$  is

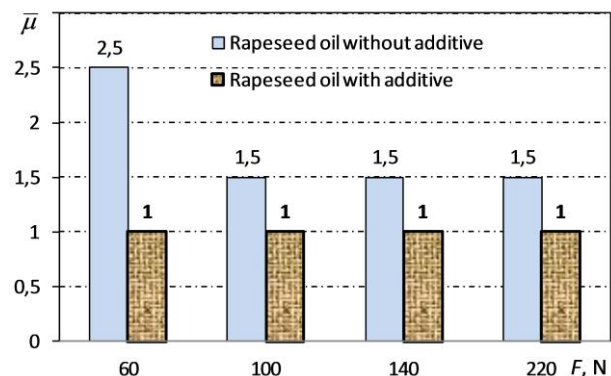
equal to the ratio of the friction coefficient for the oil with the additive to the friction coefficient for the original oil.

**Table 8.** The results of measurements of friction force  $F_t$  and friction coefficient  $\mu$

№	lubricating oil	Load, $F$			
		$F_1 = 60 \text{ N}$		$F_2 = 100 \text{ N}$	
		$P_{a1} = 21,2 \text{ N/cm}^2$	$\mu$	$P_{a2} = 35,3 \text{ N/cm}^2$	$\mu$
1	Rapeseed oil	4,5	0,075	6	0,06
2	Rapeseed oil+ AAE-Additive	2	0,03	4	0,04
№	lubricating oil	$F_3 = 140 \text{ N}$		$F_4 = 220 \text{ N}$	
		$P_{a3} = 49,5 \text{ N/cm}^2$	$\mu$	$P_{a4} = 77,7 \text{ N/cm}^2$	$\mu$
		$F_{t3}, \text{ N}$	$\mu$	$F_{t4}, \text{ N}$	$\mu$
1	Rapeseed oil	9	0,064	15	0,068
2	Rapeseed oil+ AAE-Additive	6	0,042	10	0,045



**Figure 7.** Dependence of friction coefficient on the normal load



**Figure 8.** The diagram of the relative change of the friction coefficient  $\bar{\mu}$  under different normal loads

### 3. DISCUSSION

The results of the experiments showed that the introduction of aconitic acid trihexadecyl ester (AAE-additive) into the composition of vegetable oil significantly improves the antiwear properties (paragraph 2.2). In the conformal contact of sliding friction, imitating the bearing of the crankshaft of the tractor engine, the results of the introduction of the investigated additive are close to the results of the introduction of ZDDP with alkyl radicals of the same length and structure as in AAE-additive. This can be explained by the adsorption mechanism of action of the additives. The effect of the adsorbed surfactant layer extends to an extremely thin layer of liquid. Under conditions of simulating a radial friction bearing with low surface roughness, hydrodynamic pressures provide the separating layer of lubricant even at high contact pressures.

With the introduction of ZDDP and AAE in gear oil also obtained similar results. Both types of additives significantly increase the anti-wear properties of gear oil class API GL-5, which is quite unexpected. The results obtained in tests of paragraph 2.3 showed that AAE is compatible with ZDDP and can be used in conjunction with them, for example, to reduce the ash content of lubricating oils.

Tests (paragraph 2.4) indicate the presence of noticeable AW/EP action. However, the results suggest a slightly lower efficacy of AAE in rapeseed oil compared to the efficiency in hydrocarbon oils. Perhaps this is due to the higher anti-wear properties of vegetable oil compared to pure hydrocarbon oils. It should be noted that AAE is an ester and is chemically similar to vegetable oil. The main difference is the higher ability of AAE to adsorb onto metal surfaces. This confirms the assumption about the predominantly adsorption mechanism of action of this additive.

### 4. CONCLUSION

The results show that aconitic acid trihexadecyl ester exhibits anti-wear

properties in both hydrocarbon and vegetable oils at high contact pressures. Aconitic acid ester can be used when it is necessary to enhance the AW/EP properties of lubricants based on hydrocarbon and vegetable oils in cases where low ash content, non-toxicity and biodegradability are particularly important. For example, in food processing equipment.

This additive can be used as a prototype for the synthesis of more effective additives with low toxicity.

### ACKNOWLEDGEMENT

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