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BRIEF COMMUNICATIONS

Mono- and Diesters of *N*-Benzoylaminoacetic Acid as Antiwear Additives to Synthetic Oils

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Abstract—Mono- and diesters of *N*-benzoylaminoacetic acid, which exhibit high wear-preventive properties in a synthetic ester oil, were synthesized.

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In connection with development of synthetic high-temperature oils the need for antiwear additives efficient at high temperatures has increased for last years. Development of such additives is caused by the necessity to exclude from oils traditional additives containing reactive elements, sulfur, phosphorus, and halogens, causing corrosion of metal surfaces and formation of deposit at high temperatures [1, 2]. In this connection a necessity arises for the replacement of such additives by compounds, which do not enter into chemical reactions with a metal surface, but capable to form strong adsorption films [3].

The present work is devoted to the synthesis of mono- and diesters of *N*-benzoylaminoacetic acid and to the study of their influence on the wear-preventive properties of ester oils. The expediency of creating antiwear additives on the basis of these compounds is defined by their high adsorption capacity in combination with stability to thermal decomposition and oxidation at high temperatures [4, 5].

These compounds were obtained by the esterification reaction of *N*-benzoylaminoacetic acid with dihydric alcohols in the presence of the KU-2 catalyst according to the scheme

$$nC_6H_5CONHCH_2COOH \;+\; HOROH$$

$$\xrightarrow[-H_2O]{n=1} C_6H_5CONHCH_2COOROH \quad \textbf{I–III}$$

$$\xrightarrow[-2H_2O]{n=2} (C_6H_5CONHCH_2COO)_2R' \quad \textbf{IV–VI}$$

R = $(CH_2)_3$ (**I**), $(CH_2)_4$ (**II**), $(CH_2)_2O(CH_2)_2$ (**III**); R' = $(CH_2)_3$ (**IV**), $(CH_2)_4$ (**V**), and $(CH_2)_2O(CH_2)_2$ (**VI**).

The structure of the synthesized compounds was confirmed by IR spectroscopy data. Absorption bands characteristic of the carboxyl group COOH (960 cm^{-1}) are absent from the IR spectra of the synthesized mono- and diesters of *N*-benzoylaminoacetic acid. The physicochemical properties of the synthesized compounds are shown in Table 1.

Wear-preventive properties of compounds were estimated on a four-ball-type friction machine (FBM) according to GOST 9490-75 by the fixed-load method in the temperature interval of 20–200°C at a spindle speed of 1500 rpm, a trial duration of 1 h, and a load of 20 kgf (balls of ShKh-15 steel, diameter of 12.7 mm). The ester of pentaerythrite and C_5–C_9 synthetic fatty acids (PEE) was used as a synthetic oil. A sample of the oil with a known additive, tricresyl phosphate $(CH_3C_6H_4O)_3PO$ (TCP), was tested for comparison. Test data are presented in Table 2.

It is seen from Table 2 that the compounds under study noticeably raise wear-preventive properties of the pentaerythrite ester, the wear reduction to the values d_i =

Table 1. Characteristic of mono- and diesters of *N*-benzoylaminoacetic acid

Compound	Yield, %	mp, °C	Found, % Calculated, %			Gross-formula
			C	H	N	
I	72	142–143	60.64 60.75	6.28 6.33	5.80 5.90	$C_{12}H_{15}NO_4$
II	70	155–158	62.21 62.15	6.71 6.77	5.63 5.5	$C_{13}H_{17}NO_4$
III	70	38	58.50 58.43	6.48 6.37	5.22 5.24	$C_{13}H_{17}NO_5$
IV	64	138	63.41 63.2	5.45 5.53	7.1 7.0	$C_{21}H_{22}N_2O_6$
V	66	147	64.15 64.08	5.9 5.83	6.9 6.8	$C_{22}H_{24}N_2O_6$
VI	65	31	63.80 63.77	5.75 5.80	3.42 3.38	$C_{22}H_{24}N_2O_7$

Table 2. Effect of mono- and diesters of *N*-benzoylaminoacetic acid on wear-preventive properties of pentaerythrite ester

Compound	Concentration, %	Wear scar diameter d_i, mm	
		1 h	4 h
PEE without additive	–	0.90	0.95
Tricresyl phosphate	1 3 4	0.80 0.70 0.55	0.75 0.70 0.50
$C_6H_5CONHCH_2COO(CH_2)_3OH$ (**I**)	1 2	0.65 0.60	0.70 0.65
$C_6H_5CONHCH_2COO(CH_2)_4OH$ (**II**)	1 2	0.70 0.55	0.75 0.60
$C_6H_5CONHCH_2COO(CH_2)_2O(CH_2)_2OH$ (**III**)	1 2	0.60 0.50	0.55 0.50
$(C_6H_5CONHCH_2COO)_2(CH_2)_3$ (**IV**)	0.5 1	0.60 0.55	0.60 0.50
$(C_6H_5CONHCH_2COO)_2(CH_2)_4$ (**V**)	0.5 1	0.55 0.45	0.60 0.50
$(C_6H_5CONHCH2COO)_2(CH_2)_2O(CH_2)_2$ (**VI**)	1 2	0.55 0.45	0.50 0.40

0.5–0.55 in the presence of the compounds under study being reached at lower concentrations than for tricresyl phosphate (compounds **I, IV**, and **V**).

The lubricity effectiveness of esters increases in the presence of hydroxyl groups in molecules, and also with increasing number of methylene groups and molecular weight of the esters.

High wear-preventive properties of the studied compounds are probably caused by the presence of –COOR, –N<, and –C=O groups characterized by intensive adsorption on metal surfaces. In this case charged boundary layers bound with the metal basically by physical adsorption forces are formed on the metal surface.

Thus, the possibility in principle of creating antiwear additives to pentaerythrite ester on the basis of compounds belonging to the mono- and diesters of *N*-benzoylaminoacetic acid has been established.

EXPERIMENTAL

3-Hydroxypropyl ester of benzoylaminoacetic acid. To a solution of *N*-benzoylaminoacetic acid (8.95 g, 0.05 mol) in 50 ml of dehydrated toluene propanediol-1,3 (7.6 g, 0.05 mol) and KU-2 cation-exchange resin (2.4 g) were added. The mixture was heated up at 105°C within 5 h with stirring. Water in the form of azeotrope was collected in a Dyne–Stark trap. After termination of water evolution the reaction product was filtrated, the solvent was distilled off, and the residual was recrystallized from isopropanol. White crystals with melting point of 148–150°C were obtained. Yield 72%. Other monoesters of this series were obtained similarly.

1,4-Bis(*N*-benzoylaminoacetoxy)butane. Ground crystals of *N*-benzoylaminoacetic acid (34.0 g, 0.2 mol) were dissolved in 75 ml of *p*-xylene, and ethanediol-1,4 (9 g, 0.1 mol) and KU-2 (5 g) were added. The mixture was heated at 140°C with stirring up to the full release of water. Water in the form of azeotrope was collected in a Dyne–Stark trap. After termination of water evolution the reaction product was filtrated, the solvent was distilled off, and the residual was recrystallized from benzene. White crystals with melting point of 147°C were obtained. Yield 66%. Other esters of this series were obtained similarly.

The IR spectra of the specified compounds were taken on a Specord 75-IR instrument.

CONCLUSIONS

(1) Mono- and diesters of *N*-benzoylaminoacetic acid were synthesized by reactions of this acid with dihydric alcohols.

(2) Wear-preventive properties of the synthesized compounds in the temperature range of 20–200°C were determined.

(3) It was found that the lubricity effectiveness of esters increases in the presence of hydroxyl groups as the number of methylene groups and the molecular weight of the esters increase.

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