FOREIGN TECHNOLOGY DIVISION

INCREASING THE QUALITY OF LUBRICATING GREASE BY APPLICATION OF COMPOSITIONS OF CERTAIN ADDITIVES CONTAINING SULFUR AND PHOSPHORUS

by

R. K. Veliyeva, Z. A. Ali-Zade, et al.

EDITED TRANSLATION

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R. K. Veliyeva, Z. A. Ali-Zade, T. B. Aliyeva, and A. G. Aliyev

A number of very effective methods and materials for protecting metallic equipment operating in various conditions – in the atmosphere, in water, in solutions of salt, acids and alkalies – have been applied. These include enamel, bitumen, metallic, and rubber coatings, lubricating greases, etc. Recently lubricating greases have found ever wider use to protect metallic equipment. However, they have a number of deficiencies, e.g., weak adhesion to the metal surface, creeping at temperatures above 40°C, and near-wall syneresis. These deficiencies are eliminated by adding surface-active substances (SAS) to the greases.

Our study was concerned with research on the influence of mixtures of certain salts of diesterdithiophosphoric acid and triesters of dithiophosphoric acid obtained from technical alkylphenol and its methylene-bis derivative on the anticorrosion properties of lubricating greases.

In order to obtain the indicated mixture of additives, a toluene solution of technical alkylphenol or its methylene-bis derivative was treated at 110-115°C with a calculated quantity of phosphorus pentasulfide. Upon completion of the reaction the obtained diesterdithiophosphoric acids were treated at 60-70°C with

vinyl acetate in one experiment and with methylmethacrylate in another. Then the reaction mixture was divided into three equal parts; in the first the unreacted portion of the diesterdithiophosphoric acid was neutralized with heating by magnesium carbonate, in the second part – with zinc oxide, and in the third part, with barium hydroxide.

In all experiments the insoluble impurities were separated by filtration or centrifuging. After distilling off the solvent and other volatile products, we tested the prepared additives.

As the result of these experiments 12 additives were prepared; their compositions are given in Table 1.

Table 1.

Diesterdithiophosphoric acid	From technical alkylphenol		From methylene-bis technical alkylphenol	
With the compound	Vinyl acetate	Methyl-methacrylate	Vinyl acetate	Methyl-methacrylate
With neutralization of the acid residue in experiments No. 1-12 these salts were formed:	1 barium	7 barium	4 barium	10 barium
	2 magnesium	8 magnesium	5 magnesium	11 magnesium
	3 zinc	9 zinc	6 zinc	12 zinc

The inhibiting effect of the obtained SAS in plastic greases on corrosion in neutral electrolytes was determined according to the retardation of cathode and anode processes on the metal under a film of grease. The cathodic and anodic polarization curves were taken for metallic specimens coated with grease films 10 μm thick and stabilized in still sea water for 2 h. With this coating thickness corrosion was determined basically by polarization resistance [1]. A uniform layer 10 μm thick was achieved by smearing the grease onto the polished surface of specimen plates by means of a sliding roller wrapped with polyamide film. This film was changed after application of each grease composition. The working surface

of the polarizing specimens equaled 3 cm^2 in all cases. Plate specimens of st. 3, 25 × 35 mm in size, were ground to a surface finish of class 7 and treated in benzene, acetone, and alcohol before application of the tested grease. After application of the grease film the specimens were framed [mounted] in a melt of grease made up of a mixture of petrolatum and 10% ceresin at 80°C. At 1 h after framing the specimens were immersed in sea water (volume 0.4 *l* for one specimen). The electrode potential was measured every 5 min for 2 h. When the magnitudes of specimen electrode potential coincide within the limits of 10 mV the polarization characteristic is taken according to the generally accepted galvonostatic procedure.

Each of the products shown in Table 1 was added to the melted grease in a quantity of 3%. Total dissolution, with preparation of a homogeneous grease, was achieved after 30 min at 100°C with agitation.

The characteristics of the obtained lubricants are shown in Table 2, from which it is evident that addition of many of the tested SAS to petrolatum leads to an improvement in protective properties and to an insignificant increase in the creep temperature, i.e., the maximum operating temperature of the grease. Determination of adhesion according to the tendency to be thrown off a rotating disk indicated that it is not improved in all cases. The porosity of the grease film was determined by immersing a steel plate with an applied film 50 μm thick in a solution of copper sulfate. The porosity of the grease film was determined with respect to the area of copper deposition in 3 h.

Comparison of the porosity of investigated compositions with corrosion leads to the conclusion that the increase in continuity of the grease film does not lead in all cases to a reduction in corrosion of the steel. An increase in protective effect is apparently connected also with the formation of a protective layer directly on the steel, i.e., with the inhibiting action of SAS on the corrosion process.

Table 2.

Composition of grease	Drop point, °C	Creep temp., °C	Adhesion at 6000-7000 r/min	Porosity, % of total surface	Corrosion on 10-mark scale during prolonged testing, cycles		
					10	30	60
Petrolatum	53	40	94,3	7	1	3	4
" + 3% ПАВ 1	53	42	91,5	0,1—0,3	1	2	2
" + 3% ПАВ 2	53	42	96,0	0	1	2	2
" + 3% ПАВ 3	57	42	93,1	0	—	—	—
" + 3% ПАВ 4	51	42	96,5	0,1	0	1	2
" + 3% ПАВ 5	56	43—44	97,0	0,3—0,5	0	1	2
" + 3% ПАВ 6	56	42	94,5	0,1	1	2	3
" + 3% ПАВ 7	55	42	93,5	0,1—0,3	0	1	2
" + 3% ПАВ 8	55	41	97,5	0	1	2	2
" + 3% ПАВ 9	54	41	89,3	0,1	1	2	2
" + 3% ПАВ 10	55	42	92,3	0	1	2	4
" + 3% ПАВ 11	56	42	92,0	0,1—0,3	0	1	2
" + 3% ПАВ 12	57	42	94,5	0,1	0	1	2

[ПАВ = SAS]

Figures 1 and 2 show the displacement of electrode potential during cathodic polarization of steel specimens coated with tested grease compositions.

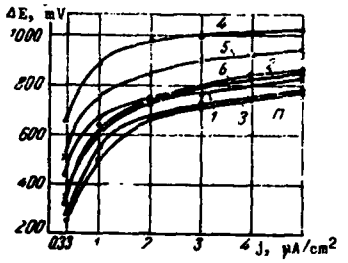

Fig. 1.

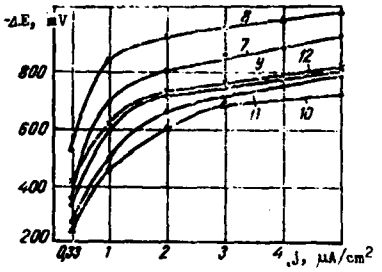

Fig. 2.

As is evident from the figures, only one of the tested products (10) accelerates the reaction of oxygen and hydrogen depolarization, while the remaining specimens vary in the degree of influence on the cathode reaction. The greatest inhibition of the cathode process occurs when product 4 is present in petrolatum; the series of other products in descending order of inhibiting action is 8, 5, 6, 7, 2, 12, 9, 1, and 3.

Consequently, among the SAS obtained which inhibit the cathode process, the greatest inhibiting action is found basically in products from interaction of vinyl acetate with diesterdithiophosphoric acid obtained from methylene-bis technical alkylphenol. For these compositions the presence of a methylene bridge between two

alkylphenols leads to an increase in inhibiting action. The presence of the barium salt of diesterdithiophosphoric acid in the two-component mixture has a greater effect than the presence of the magnesium salt.

Figure 3 shows anode polarization curves, designating principally the same products, which displayed an inhibiting effect on the cathode process in sea water.

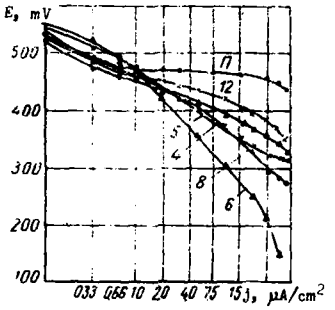

Fig. 3.

As is evident from Fig. 3, the greatest inhibiting action on the anode process is manifested by products 6 and 8. Comparison of the inhibiting action of products 6 and 8 on the cathode and the anode processes leads to the conclusion that they are simultaneously inhibitors of both the cathode and the anode processes.

Conclusions

1. Technical alkylphenol and its methylene-bis derivative were used to obtain effective SAS of the type of triesters of dithiophosphoric acid in combination with barium, magnesium, and zinc salts of diesterdithiophosphoric acid; these SAS, possessing a minor thickening action on petrolatum, increase the continuity of the grease film.

2. It was established that products 4, 8, 5, 6, and 7 are effective inhibitors of cathode corrosion of grease in sea water.

Also, products 6 and 8, inhibitors of the cathode process, simultaneously manifest a significant inhibiting action on the anode process.

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2. Yagubovich, S. V. Issledovaniye lakokrasocheykh materialov i pokrytiy (Investigation of paint and varnish materials and coatings), Goskhimizdat, 1952.

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13. ABSTRACT

Additives were prepd. from mixts. of diaryl dithiophosphates and triaryl dithiophosphates, obtained by reaction of P(Subscript 2)S(Subscript 5) with tech. alkylphenols (I) or methylenebis (alkylphenols) (II). Each of the di- and triester mixts. obtained from I and II was reacted with vinyl acetate and with CH(Subscript 2):CMeCO(Subscript 2)Me in toluene, sepd. into 3 equal parts, and neutralized with MgCO(Subscript 3), ZnO, and Ba(OH)(Subscript 2), to give a total of 12 additives with surface-active properties. The inhibiting effect of 3% of each additive in greases on corrosion in neutral electrolytes was detd. by the inhibition of the cathodic or anodic processes on metal. The metal was covered with a 10-μ film of grease contg. one of the additives. The film was stabilized 2 hours in sea water, and the corrosion was detd. by the polarization resistance. None of the additives had a significant thickening effect on the grease (petrolatum), but increased the continuity of the grease film. The additives were efficient inhibitors of cathodic corrosion for the grease in sea water. Some of the additives also had an important inhibiting effect on the anode process.

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14. KEY WORDS	LINK A		LINK B		LINK C	
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Grease						
Chemical compositon						
Corrosion inhibitor						
Polyvinyl acetate						
Thiol						
Petroleum product						
Electrolyte						
Surface active agent						
Phosphate ester						
Phenol						
Lubricant additive						

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