

Functionalization of Low-Molecular-Weight Oligomer Dienes and Olefins with Sulfur- and Oxygen-Containing Compounds

R. Z. Biglova,¹ A. U. Galimzyanova,¹ V. A. Dokichev,² G. V. Konesev,³ G. E. Zaikov,⁴
R. F. Talipov¹

¹Bashkir State University, Ufa, Russia

²Institute of Organic Chemistry, Ufa Center of Science, Russian Academy of Science, Ufa, Russia

³Ufa State Petroleum Technical University, Ufa, Russia

⁴Russian Academy of Science, N. M. Emanuel'ja, Moscow, Russia

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ABSTRACT: The functionalization of piperylene fractions, oligodienes, and oligo-olefins by elemental sulfur and phenols was carried out. The functionalized compounds were shown to be multipurpose additives, exhibiting high antiwear and antiscuff properties and simultaneously antioxi-

dant and viscous properties. © 2006 Wiley Periodicals, Inc. *J Appl Polym Sci* 101: 3097–3105, 2006

Key words: additives; micelles; modification; oligomers

Multipurpose additives to polymers, as well as batch additives to various oils with a complex of necessary properties, are being used in an increasingly wider range of applications.^{1,2} This has resulted in the investigation of elemental sulfur reactions with dienes, oligodienes, and oligo-olefins. Cheap and available piperylene fractions (multitonage waste from obtaining isoprene in rubber manufacturing), their oligomerization products, and the oligomerization products of used butene–isobutane fractions of various molecular weights have been used as initial hydrocarbons.

Because the antiscuff and antiwear properties of additives depend on the content of covalently bonded sulfur, the selection of optimum conditions to effect sulfuring processes has been set with the introduction of the greatest possible amount of covalent sulfur. For this purpose, the functionalization of diene and olefin hydrocarbons was conducted in a wide time–temperature mode. Because the temperature of the boiling piperylene fraction was low (42–44°C), sulfuring was carried out in a constant-temperature-controlled autoclave in the presence of the catalyst cobalt phthalocyanine³ in media of nonpolar solvents (e.g., heptane). Under the synthesis conditions (130°C for 3 h, >C=C</sulfur molar ratio = 1 : 4), a product was obtained, with the sulfur concentration amounting to

54.95 mass % (yield = 10 mass %). The low yield of the target product was due to the experiment being conducted under static conditions, so the next series of experiments was carried out under dynamic conditions. When sulfuring was conducted in a constantly rotating autoclave with the same synthesis parameters but in an environment of aromatic hydrocarbons (toluene), the yield was 49.5 mass %, and the sulfur content increased to 70.35 mass %. Piperylene fraction interactions with elemental sulfur in the absence of the catalyst improved the results (Table I). At a >C=C</sulfur molar ratio of 1 : 2 in the *p*-xylene medium, sulfur reacted quantitatively.

The structure of the synthesized compounds was established by a set of methods: ¹H-NMR and ¹³C-NMR spectroscopy and elemental analysis. When the spectra were analyzed, ¹H-NMR signals in the range of 5.50–6.50 mD were detected, being characteristic of olefin protons. The correlation between the areas of the integral curves of the olefin and methyl protons enabled us to assume the presence of double bonds in the product molecule. This was confirmed by characteristic bands in the IR spectra, with absorption occurring at 1600–1660 cm⁻¹. A signal at 1.10 mD belonged to methyl group protons, and an intense multiplet in the region of 2.10–2.50 mD belonged to methine and methylene protons. In the ¹³C-NMR spectrum at 52.13 mD, a singlet due to the C–S group was found, with the absorption in the area of 600–450 cm⁻¹ (—S—S—) within the IR spectrum.

Correspondence to: G. E. Zaikov.

TABLE I
Sulfuring Piperylene Fraction with Elemental Sulfur in a Constantly Rotating Autoclave

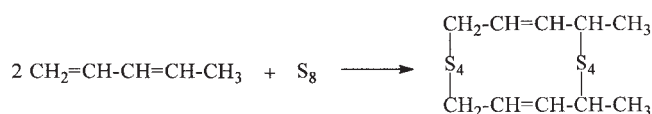
Reaction conditions	1	2	3	4	5	6
>C=C</S (mole)	1 : 2	1 : 1	1 : 2	1 : 1	1 : 2	1 : 1
Solvent	Toluene	Toluene	Kerosene	Kerosene	<i>p</i> -Xylene	<i>p</i> -Xylene
Yield of product (mass %)	48.0	24.9	43.0	19.5	100.0	78.6
Sulfur content in product (mass %)	69.86	48.23	66.11	47.01	68.24	50.71

Volume = 17 mL; temperature = 130°C; τ = 3 h.
 τ , duration of reaction (hour).

According to the results of the analyses, the reaction of piperylene with elemental sulfur proceeded according to Scheme 1, yielding piperylene bistetrasulfide. The scheme is in agreement with the data obtained by researchers^{3,4} who sulfured low-molecular-weight dienes, particularly divinyl.

Oligopiperylene (OPP) with a number-average molecular weight (M_n) of 910 was used to study the interaction of the oligomerization product of the piperylene fraction with elemental sulfur. ¹H- and ¹³C-NMR spectra of a test sample were compared with those given in ref. 5. The analysis of the chemical shifts and multiplicity signals testified to the fact that the sample of OPP synthetic rubber was mainly 1,4-trans oligomer. The total nonsaturation of the sample was 30%, corresponding to the presence of four active double bonds in a molecule. The results for determining the number of >C=C< bonds (by the bromine number) correlated with the spectral data.

The data on the interaction of OPP and oligoisobutylene (OIB; M_n = 390 or 880) with elemental sulfur are given in Tables II and III. According to the results of the elemental analysis, the contents of sulfur in the obtained products grew both with the rise in the synthesis temperature and with the increase in the process length. When the ratio of the initial substances exceeded the equivalent molar ratio of the oligomers and sulfur, no appreciable change in the values of the mass content of sulfur in the product was detected. The modification of OPP at temperatures greater than 140°C resulted in the crosslinking of the macromolecules. The reaction with elemental sulfur in a nonpolar solvent (octane and kerosene) medium did not affect the percentage of sulfur in the product, reduced the opportunity for



Scheme 1

resinification, and led to lower sulfur contents in the aromatic hydrocarbons.

Depending on the product molecular weights and sulfur contents, mono- and disulfide bridges could be found in the macromolecules. The latter corresponded to the data⁴ for sulfur interactions with isobutylene within the interval of 100–140°C. For the example of isobutylene oligomers and a link of OPP containing a double >C=C< bond, the processes can be illustrated as shown in Scheme 2.

Sulfur-containing compounds can acquire antioxidant and anticorrosive properties by the introduction of the appropriate functional groupings (a fragment of the shielded phenol) into their molecules. Therefore, the following stage of the work involved modifying oligomers with oxygen-containing compounds (phenol and 2,6-di-*tert*-butylphenol). The sequence of modification should be as follows: (1) alkylation and (2)

TABLE II
Functionalization of OPP with Elemental Sulfur at (a) temperature = 120°C^a (b) temperature = 130°C,^a and (c) temperature = 130°C, and τ = 8 h

	Conditions of functionalization: >C=C<S (mol)	τ (h)		
		4	6	8
a	1.00 : 0.50	0.67	1.11	1.69
	1.00 : 1.00	1.15	2.99	3.40
	1.00 : 2.00	0.95	2.87	3.29
	1.00 : 3.00	0.92	2.11	3.16
b	1.00 : 0.40	2.23	2.68	3.39
	1.00 : 0.50	3.57	4.02	4.48
	1.00 : 0.75	3.67	4.24	4.77
	1.00 : 1.00	4.19	5.97	6.57
	1.00 : 2.00	3.46	5.67	6.55
		Solvent		
		Octane	<i>p</i> -Xylene	Kerosene
c	1.00 : 0.05	1.93	1.17	2.57
	1.00 : 0.75	2.21	1.58	2.76
	1.00 : 1.00	3.59	2.21	6.10

^a A parameter was the sulfur content of the product (mass %).

^a Without solvent.

TABLE III
Functionalization of OIB with Elemental Sulfur

Conditions of functionalization	Sample							
	1	2	3	4	5	6	7	8
Solvent	Octane	Decane	<i>p</i> -Xylene	—	—	—	—	—
Temperature (°C)	120	160	160	140	140	140	160	160
τ (h)	6	6	8	3	6	6	6	6
$>C=C<S$ (mol)								
1.0 : 0.8	1.00	2.77	2.81	2.45	2.51	4.14	4.98	6.77
1.0 : 1.0	2.61	3.38	3.42	3.06	3.12	6.16	7.00	7.35

A parameter was the sulfur content in the product (mass %). For samples 1–5, $M_n = 880$, and for samples, 6–8, $M_n = 390$.

sulfuring. The change in the sequence is undesirable, as sulfide bridges do not have sufficient durability and can be broken during the subsequent alkylation. Because there are only terminal double bonds in oligoolefin macromolecules, the compositions of sulfur and alkyl oligomers as multipurpose additives have been investigated. Alkylation was carried out in nonpolar solvents (octane) in the presence of catalytic systems with reduced relative acidity [$Na(AlCl_4)$] within 8 h at 100–120°C at a $>C=C</math>/phenol/catalyst molar ratio of 1 : 1 : 0.2 according to Scheme 3 for the example of isobutylene oligomers and a link of OPP containing $>C=C<$.⁶ The sulfur concentration in the modified OPP was 4.92 mass % (1.58 mass % oxygen).$

The functionalization of OIBs with phenols was also performed with an alternative technique in polar solvent media [dimethylformamide (DMF) and dimethyl sulfoxide (DMSO)], in which the reaction of sulfur with low-molecular-weight phenols was conducted.⁵ Pre-alkylated phenols (4-oligoisobutenylphenols) and elemental sulfur acted as the initial substances. The interaction was carried on in an inert nitrogen flow (with released hydrogen sulfide removed from the reaction zone) at 140–150°C. The experiment results are given in Table IV. *p*-Xylene and tetrahydrofuran (THF) were added to the reactant mixture for better solubility. The mass ratio of the solvents with respect

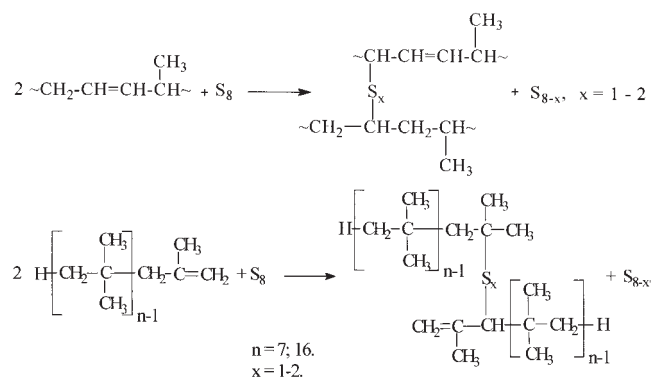
to the initial phenol weight was 1:4:1 phenol/DMF (DMSO)/*p*-xylene (THF).

It was experimentally found that in the DMF environment, the reaction proceeded with a greater yield than in DMSO. With the ratio of the initial substances lowered, the yield of the product grew; however, at ratios less than 1.0:0.5, the sulfur contents in the end compound decreased.

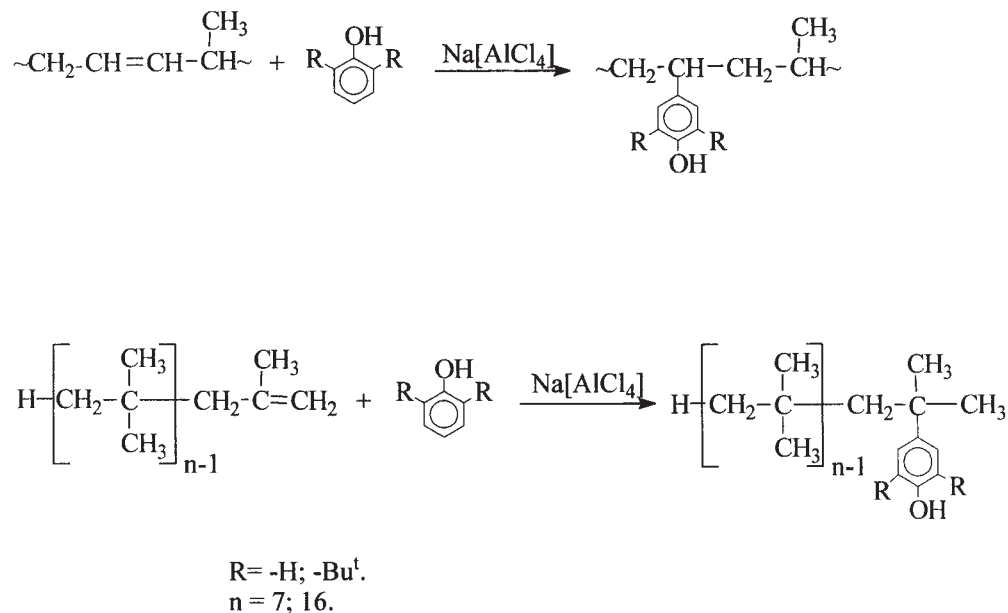
The relative viscosities of the modified oligomers, determined by viscosimetry, were somewhat higher than the values of the respective initial oligomers (Table V). The observed phenomenon allowed us to believe that in the interactions of the oligomers with phenols and elemental sulfur there occurred an increase in the molecular weight due to the functionalized product generated.

According to the elemental analysis results, the determination of the modified oligomer relative viscosities, and the spectral data and references,⁷ the interaction of OIB-alkylated phenols with elemental sulfur in our case can be written as shown in Scheme 4. The demand for multipurpose additives in the petrochemical industry is common knowledge. Functionalized sulfur- and oxygen-containing oligomers were investigated as additives. Motor oils contain up to 10% additives, which differ in their functional effects, to ensure long-term efficiency. These additives should protect oil-lubricated surfaces, improve the oil properties, and preserve the oil composition. According to the listed parameters, certain classes of chemical compounds providing necessary properties are applied.

The antiwear and antiscuff properties of sulfur-containing compounds on the basis of piperylene fractions were investigated. To characterize the designated properties of the additives, the following important parameters were employed: the rate of wearing (a_m) and the factor of friction (k_{fr}). The testing of sulfur-containing substances to withstand washing liquors (clay solution) and lubricants was performed on an MT3 experimental installation according to the technique described in ref. 8. Depending on the applied



Scheme 2



Scheme 3

loading, the a_m values of cores from chiseled steel in compositions based on mark 52 cylinder oil in the presence of the synthesized substances were determined. In comparison with VNIINP-354, an additive traditionally employed in the industry [zinc *o,o*-di(ocetylphenyl)dithiophosphate], the produced piperylene bistetrasulfide and sulfur-modified OPP displayed high results (Fig. 1).

Studying the antiwear properties of clay solutions in the presence of an insignificant amount (up to 1 mass %) of the sulfur-containing product and up to 3 mass % superficially active substance (SAS) (SDBUR) showed that an increase in the contact pressure resulted in the values of k_{fr} and a_m of cores from chiseled steel decreasing by 30–35% in comparison with the respective parameters for the clay solution without additives (Figs. 2 and 3). As the axial loading in-

creased, films were formed on the surface under friction, which led to greater efficiency of the drilling fluids at higher loadings. The operational parameters were affected by the order in which the components were mixed. The compositions of clay solutions with xylene solutions of the sulfured piperylene fraction to some extent increased the values of a_m and k_{fr} , in contrast to the product in the absence of the solvent.

Tests of the oil composition in the presence of synthesized piperylene bistetrasulfide were performed on 20 chisels of drills under the conditions of the Far North.

The antioxidant properties of oil with additives on the basis of OPP were determined by a stability measurement method during the induction period of deposition (IPD). The thermal-stabilizing ability of sulfured oligopiperylenylphenol added to mark

TABLE IV
Interaction of Sulfur with 4-Oligoisobutenylphenols in the Polar Solvent Medium

Conditions of interaction	Sample					
	1	2	3	4	5	6
Phenol S ₈ (mol)	1.0 : 1.5	1.0 : 1.0	1.0 : 0.5	1.0 : 0.5	1.0 : 0.5	1.0 : 0.3
Solvent	DMF	DMF + <i>p</i> -xylene	DMF + <i>p</i> -xylene	DMF + THF	DMSO + <i>p</i> -xylene	DMSO + <i>p</i> -xylene
Initial OIB ($M_n = 390$)						
Yield of product (mass %)	21.0	18.0	42.0	34.0	28.0	46.0
Sulfur content in product (mass %)	8.39	6.09	6.46	6.15	3.92	3.50
Initial OIB ($M_n = 880$)						
Yield of product (mass %)	29.0	30.7	96.0	41.0	65.0	72.6
Sulfur content in product (mass %)	1.24	1.86	3.76	0.63	1.40	0.73

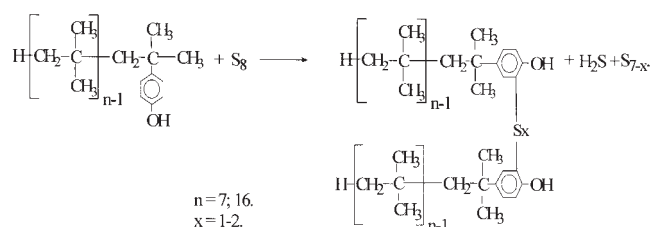
Temperature = 145–150°C; $\tau = 6$ h.

TABLE V
Viscosity Characteristics of the Initial and Functionalized Oligomers

Oligomer	Concentration (g/dl)	Relative viscosity
OIB ($M_n = 390$)	4.2	1.14
	1.0	1.03
Sulfured 4-oligoisobutenylphenol	4.2	1.41
	1.0	1.32
OIB ($M_n = 880$)	4.2	1.33
	1.0	1.19
Sulfured 4-oligoisobutenylphenol	4.2	1.92
	1.0	1.48
OPP ($M_n = 910$)	4.2	1.42
	1.0	1.26
Sulfured OPP	4.2	2.29
	1.0	1.67

I-20 industrial oil was within the limits satisfying the All-Union standard (the contents of deposit content in oxidized oil (X_0) precipitation were less than 0.5 mass %). The given compositions were also tested for the viscosity properties. The index of the viscosity should be no less than 90 to produce thickening action; this was confirmed by the data given in Table VI.

Detergent-dispersive additives are indispensable components of batch additives. Imides of amber acid, whose washing action is considered with respect to their solubilizing capacity, that is, the transfer of micelles into a solution of SAS products, insoluble in the given medium, are most common in this quality. The influence of oxidation inhibitors (oligo-olefinylphenol and oligodienylphenol), antiwear additives (sulfuring oligomers of isobutylene and piperylene), and chemical additives of multifunctional action (oligo-olefin and oligodiene-alkylated phenol products and products of subsequent sulfuring of the obtained compounds in both nonpolar and polar solvents) on the detergent-dispersive efficiency of cuccinimide was investigated to find the optimum concentration of the compounds within a batch. According to ref. 9, the concentration dependence of the antiwear efficiency of sulfur-containing compounds is of an extremely complex char-



Scheme 4

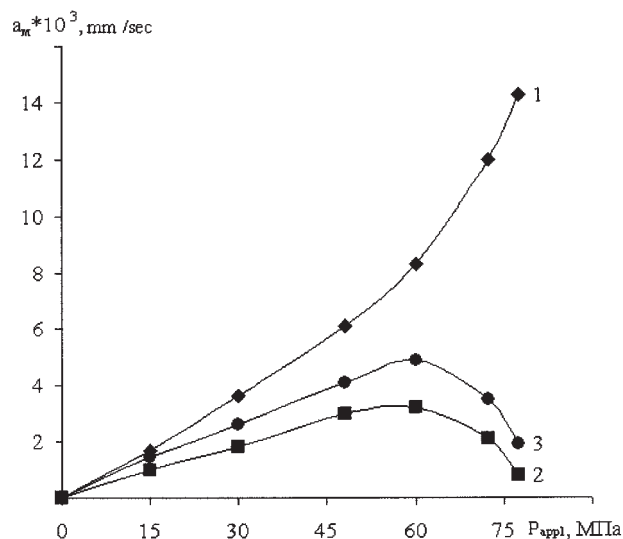


Figure 1 Dependence of a_m on the applied loading (P_{appl}): (1) mark 52 industrial oil/VNIINP-354, (2) mark 52 industrial oil/piperylene bistetrasulfide, and (3) mark 52 industrial oil/sulfured OPP.

acter explained by micelle formation in the additive oil solutions. From this point of view, it is interesting to investigate the concentration dependence of the antiwear and detergent-dispersive additive compositions.

Cuccinimide, necessary for studying solubilization, was synthesized on the basis of OIB ($M_n = 880$) by a known technique,¹⁰ and the nitrogen concentration was about 3.98 mass % (according to TU-38101146-77, the concentration should be no less than 1.4 mass %). The research of the solubilization abilities of the addi-

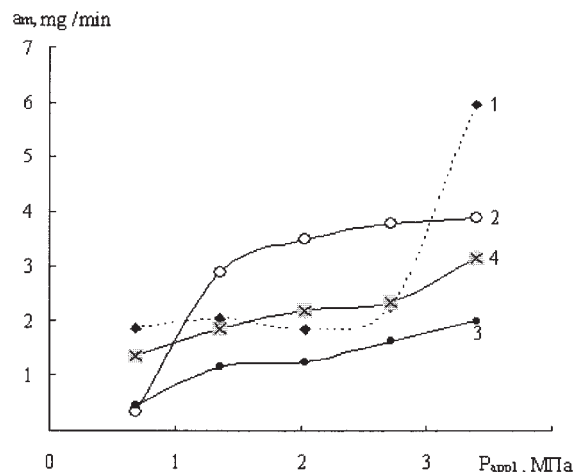


Figure 2 Dependence of a_m on the applied loading (P_{appl}): (1) clay solution, (2) clay solution/SDBUR (1.0 mass %), (3) clay solution/SDBUR (1.0 mass %)/piperylene bistetrasulfide (0.1 mass %), and (4) clay solution/SDBUR (1.0 mass %)/piperylene bistetrasulfide in a solvent (0.1 mass %).

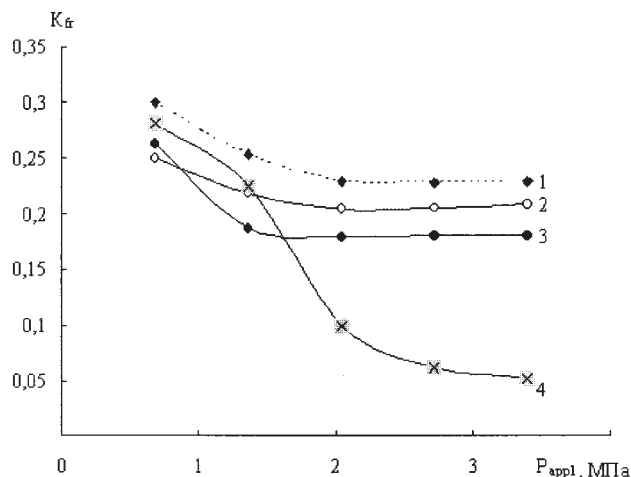


Figure 3 Dependence of k_{fr} on the applied loading (P_{app1}): (1) a clay solution, (2) a clay solution/SDBUR (1.0 mass %), (3) a clay solution/SDBUR (1.0 mass %)/piperylene bistetrasulfide (0.1 mass %), and (4) a clay solution/SDBUR (1.0 mass %)/piperylene bistetrasulfide in a solvent (0.1 mass %).

tives was carried out with the technique described in ref. 11. With respect to the dye, the solubilizing action was to a greater or lesser extent displayed by all the types of the investigated compounds. The curves of the composition solubilization effect dependence on the additive concentrations were of an extremely complex character; also, they differed in their location in relation to the axes of the coordinates. For the majority of the substances, the dependence curves had three strongly pronounced areas. The first covered an interval of concentrations with no solubilization effect, the second promoted the growth of the solubilization ability because of the change in the additive content, and the third was an area of higher concentration with the maximum values of the effect. This was because in each specified area, the additive solutions exhibited various colloidal properties.

In experiments with rhodamine C, mainly two types of curves [Fig. 4(a–d)] were constructed: the first was characterized by fast colloidal dissolution of the dye and solution stability (e.g., curves 2b and 3b), and the

other was characterized by a longer stabilization period of the solutions (curves 2a and 3a) but had a sharp growth of the solubilization effect for compositions in the area of the higher concentration (curve 9a). For the compositions of the specified additives with succinimide, the solubilization effect was more evident and the values of the critical micelle concentration (cmc) were lower in comparison with the features of individual substances in isooctane (Table VII). When sulfured piperylene fractions were characterized by the cmc interval of 0.05–0.45 mass %, in the presence of 0.5 and 1.0 mass % succinimide, the cmc areas corresponded to the following values: 0.02–0.50 and 0.01–0.50 mass %.

The influence of the size and structure of a hydrocarbon radical of antioxidant and antiwear additives introduced into an oil composition on the succinimide detergent-dispersion action was observed. The maximum solubilization effect took place for the ramified and smaller radicals. Such an effect was discovered in substances synthesized on the basis of OPP ($M_n = 910$) and OIB ($M_n = 390$). Apparently, the phenomenon was explained by the fact that in inverse emulsions such as water in tar, the increase in the length of the hydrocarbon substituted by one $-\text{CH}_2-$ group in the given homologous row leads to a cmc shift into an area of larger values.

The sulfur concentration increase in functionalized samples contributed to a narrower cmc interval and its shift into an area of smaller values [Fig. 4(d): a sulfured piperylene fraction containing 47.01 mass % sulfur].

The experimental data (Table VII) demonstrated that the introduction of the synthesized substances into an oil composition with succinimide allowed us to considerably cut the dosage of the latter and maintain the operational characteristics at a sufficiently high level.

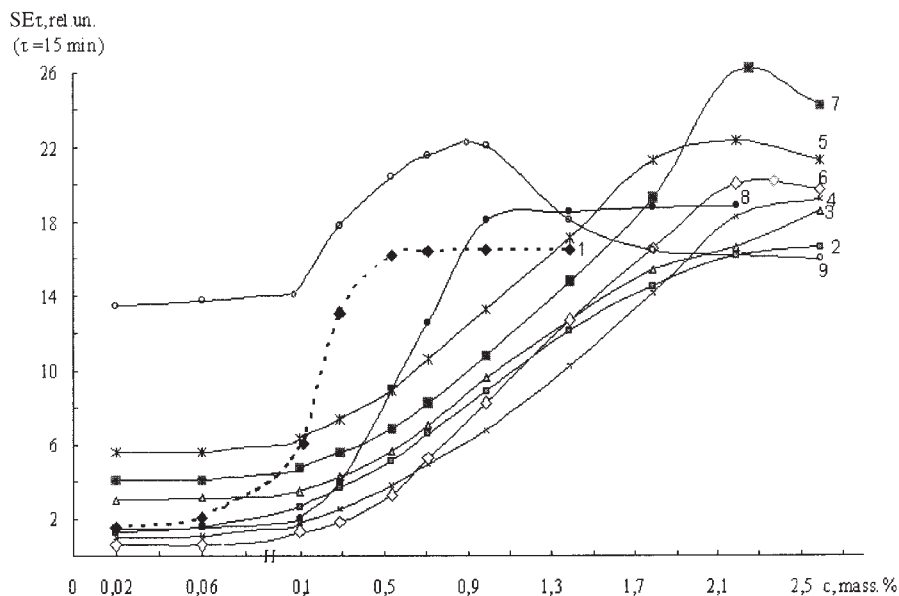
CONCLUSIONS

The functionalization of piperylene fractions, oligodienes, and oligo-olefins by elemental sulfur was car-

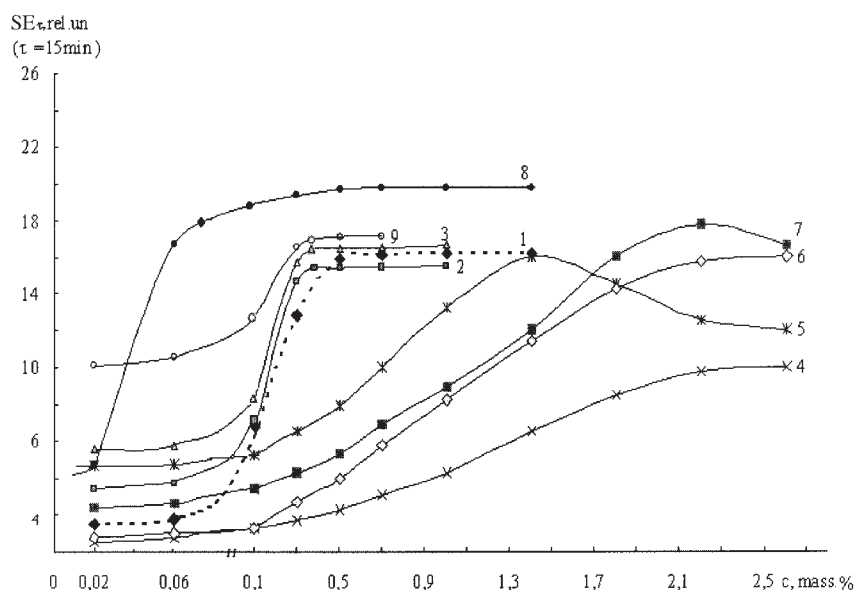
TABLE VI
Characteristics of I-20 Oil in the Presence of Additives

Additive	Additive concentration in oil (mass %)	Viscosity (cc) at		IV	Deposit (mass % IPD)
		40°C	100°C		
OPP ($M_n = 910$)	2	74.2	9.1	95	—
	5	84.7	10.3	98	—
Sulfuring 2,6-di-tert-butyl-4-oligopiperylenyl phenol	2	81.0	10.1	96	0.22
	5	103.3	11.4	100	—
I-20 oil	—	66.7	8.3	91	0.85

IV, the index of viscosity characterizes ability of the additives to produce thickening action to oil.



(a)



(b)

Figure 4 Dependence of the solubilizing power on the additive concentration (c) in solution: (a) modified OIB with $M_n = 880$ [(1) succinimide, (2) sulfured OIB, (3) sulfured OIB/succinimide (0.3 mass %), (4) 2,6-di-*tert*-butyl-4-oligoisobutenylphenol, (5) 2,6-di-*tert*-butyl-4-oligoisobutenylphenol/succinimide (0.3 mass %), (6) 2,6-di-*tert*-butyl-4-oligoisobutenylphenol/sulfured OIB (mass ratio = 1 : 1), (7) 2,6-di-*tert*-butyl-4-oligoisobutenylphenol/sulfured OIB (mass ratio = 1 : 1)/succinimide (0.3 mass %), (8) sulfur-containing bis-4,4'-oligoisobutenylphenol, and (9) sulfur-containing bis-4,4'-oligoisobutenylphenol/succinimide (0.3 mass %)], (b) modified OIB with $M_n = 390$ [(1) succinimide, (2) sulfured OIB, (3) sulfured OIB/succinimide (0.3 mass %), (4) 2,6-di-*tert*-butyl-4-oligoisobutenylphenol, (5) 2,6-di-*tert*-butyl-4-oligoisobutenylphenol/succinimide (0.3 mass %), (6) 2,6-di-*tert*-butyl-4-oligoisobutenylphenol/sulfured OIB (mass ratio = 1 : 1)/succinimide (0.3 mass %), (7) 2,6-di-*tert*-butyl-4-oligoisobutenylphenol/sulfured OIB (mass ratio = 1 : 1)/succinimide (0.3 mass %), (8) sulfur-containing bis-4,4'-oligoisobutenylphenol, and (9) sulfur-containing bis-4,4'-oligoisobutenylphenol/succinimide (0.3 mass %)], (c) modified OPP [(1) succinimide, (2) sulfured OPP, (3) sulfured OPP/succinimide (0.3 mass %), (4) 2,6-di-*tert*-butyl-4-oligopiperilynylphenol, (5) 2,6-di-*tert*-butyl-4-oligopiperilynylphenol/succinimide (0.3 mass %), (6) sulfured 2,6-di-*tert*-butyl-4-oligopiperilynylphenol, and (7) sulfured 2,6-di-*tert*-butyl-4-oligopiperilynylphenol/succinimide (0.3 mass %)], and (d) sulfured piperylene fraction [(1) succinimide, (2) piperylene bistetrasulfide, (3) piperylene bistetrasulfide/succinimide (0.5 mass %), and (4) piperylene bistetrasulfide/succinimide (1.0 mass %)].

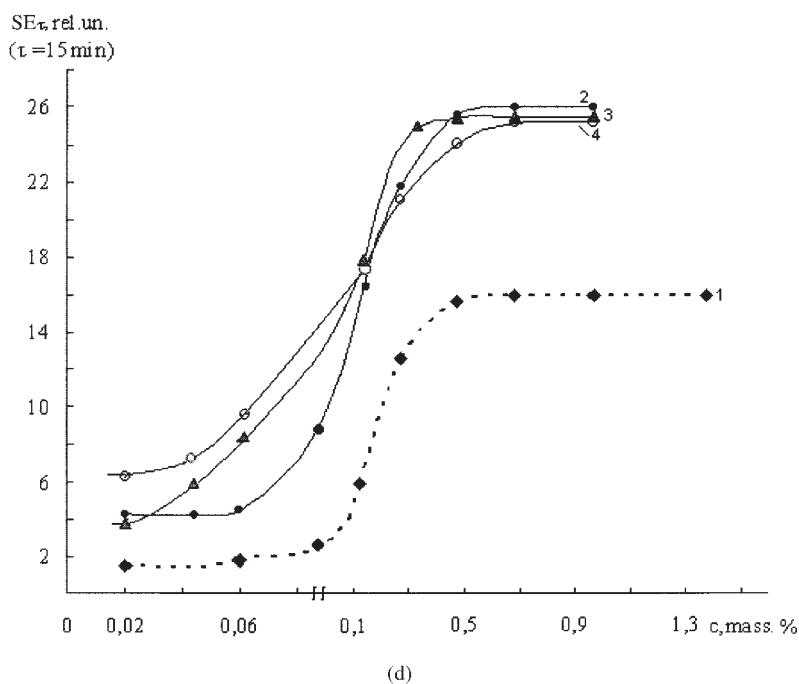
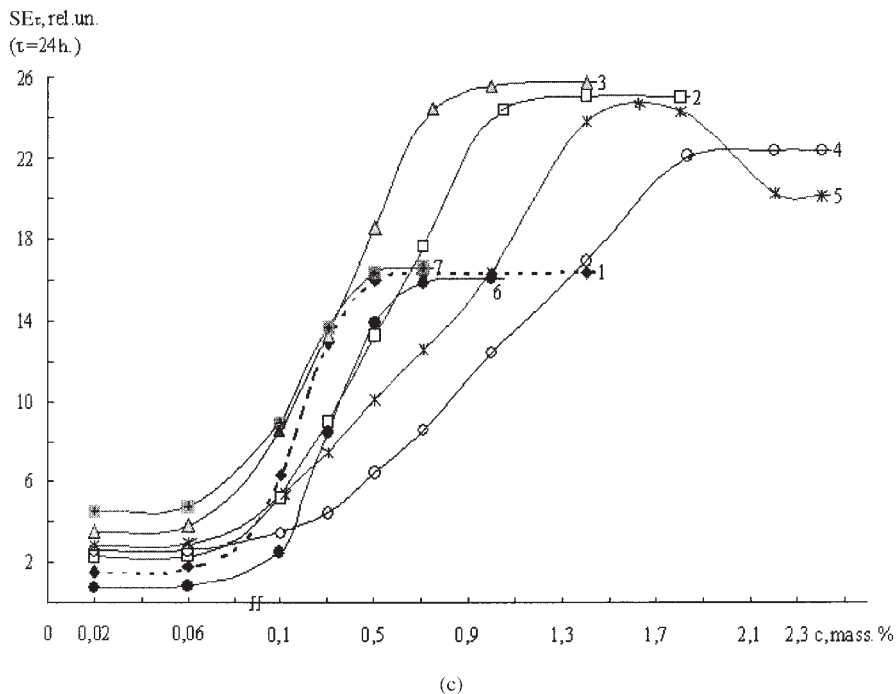


Figure 4 (Continued from the previous page)

ried out, and optimum conditions to effect their interaction with the greatest possible quantity of sulfur were selected.

Functionalized compounds were shown to be multipurpose additives exhibiting high antiwear and anticuff properties and simultaneously antioxidant and viscous properties.

The influence of the obtained sulfur-containing substances and shielded phenols on the detergent-dispersive action of succinimide was investigated. It was established that combinations of antiwear and antioxidant additives in an oil composition with succinimide could appreciably reduce the contents of the latter to maintain sufficiently high operational characteristics of lubricants.

TABLE VII
CMC (mass %) of the Functionalized Products

Additive	OPP ($M_n = 910$)	OIB	
		$M_n = 880$	$M_n = 390$
Cuccinimide	0.08–0.42	0.08–0.42	0.08–0.42
Sulfured oligomer	0.09–1.30	0.10–1.90	0.09–0.30
Sulfured oligomer cuccinimide (0.3 mass %)	0.08–0.70	0.08–1.70	0.08–0.20
Alkylated 2,6-di-tert-butyl-phenol	0.20–1.80	0.30–3.00	0.20–2.20
Alkylated 2,6-di-tert-butyl-phenol cuccinimide (0.3 mass %)	0.08–1.60	0.10–2.40	0.10–1.40
Product of successive alkylation and sulfuring in nonpolar solvents	0.09–0.56	0.10–2.40	0.16–2.20
Product of successive alkylation and sulfuring in nonpolar solvents cuccinimide (0.3 mass %)	0.09–0.30	0.10–2.20	0.08–2.10
Product of successive alkylation and sulfuring in polar solvents	—	0.15–0.95	0.07–1.00
Product of successive alkylation and sulfuring in polar solvents cuccinimide (0.3 mass %)	—	0.03–0.09	0.04–0.09

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