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RESEARCH ARTICLE

Using nitrogen-containing organosilicon compounds in the creation of heat- and fire-resistant filling compositions to seal high-voltage and high-frequency equipment

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Abstract

Objectives. To study the possibility of using nitrogen-containing organosilicon compounds in the creation of heat-resistant and fire-resistant casting compositions to seal high-voltage high-frequency equipment.

Methods. Nitrogen-containing organosilicon compounds were obtained using the *N*-siloxycarbonylation, formylation, and silylation methods. The methods used in the work were infrared spectroscopy, elemental analysis, viscometry, and differential scanning calorimetry. The mechanical and dielectric properties of the samples were determined.

Results. Previously unknown substances and compounds containing nitrogen-containing organosilicon products as components of the curing composition were obtained. Their physicomechanical and operational properties were examined, including the possibility of using them as filling heat-resistant and fire-resistant compositions for sealing high-voltage and high-frequency equipment.

Conclusions. It was shown that nitrogen-containing organosilicon compounds—3-(diethylamino)-2-[(triethoxysilyl)oxy]propyl-2-methacrylate and triethoxysilyl ester of γ -triethoxysilylpropyl-carbamic acid—can be used as part of a curing system together with bromine-containing fillers, in order to obtain compounds used for filling high-voltage high-frequency transformers, throttle valves, and other electronic equipment elements with non-combustible properties and increased heat resistance.

Keywords

nitrogen-containing organosilicon compounds, filling compositions, heat-resistant and fire-resistant filling compositions, sealing high-voltage and high-frequency equipment

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НАУЧНАЯ СТАТЬЯ

Использование азотсодержащих кремнийорганических соединений при создании термо- и огнестойких заливочных композиций для герметизации высоковольтной и высокочастотной аппаратуры

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Аннотация

Цели. Изучение возможности использования азотсодержащих кремнийорганических соединений при создании термо- и огнестойких заливочных композиций для герметизации высоковольтной и высокочастотной аппаратуры.

Методы. С помощью методов N-силоксикарбонилирования, формилирования и силилирования получены азотсодержащие кремнийорганические соединения. В работе использованы инфракрасная спектроскопия, элементный анализ, вискозиметрия, дифференциальная сканирующая калориметрия. Определены механические и диэлектрические свойства образцов.

Результаты. Получены ранее неизвестные соединения и компаунды, содержащие в составе отверждающей композиции азотсодержащие кремнийорганические продукты, изучены их физико-механические, эксплуатационные свойства, в том числе возможность использования в качестве заливочных термо- и огнестойких композиций для герметизации высоковольтной и высокочастотной аппаратуры.

Выводы. Показано, что впервые синтезированные азотсодержащие кремнийорганические соединения — 3-(диэтиламино)-2-[(триэтоксисилил)окси]пропил-2-метакрилат и триэтоксисилиловый эфир γ-триэтоксисилилпропилкарбаминовой кислоты — можно использовать в составе отверждающей системы совместно с бромсодержащими наполнителями для получения компаундов, применяемых для заливки высоковольтных и высокочастотных трансформаторов, дросселей и других элементов радиоэлектронной аппаратуры, обладающих негорючими свойствами и повышенной теплостойкостью.

Ключевые слова

азотсодержащие кремнийорганические соединения, заливочные композиции, термо- и огнестойкие заливочные композиции, герметизация высоковольтной и высокочастотной аппаратуры

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INTRODUCTION

Organosilicon sealants and compounds based on low-molecular silicone rubbers curable at room temperature in contact with air moisture began to be actively developed in the second half of the last century. These products are currently finding increased practical application in various fields of science and technology. They are used in construction, the chemical and aviation industries, in modern electronic and radio products and household chemicals [1–3].

Cold-curable sealant compositions consist of rubber, filler, vulcanizing agent, and curing catalyst. The catalyst can be present in the mixture (one-component compositions) or introduced into it immediately before vulcanization (two-component compositions). Depending on the purpose, these compositions are produced in different consistencies: paste-like, thixotropic or viscous-flowing [4, 5].

The most common cold-curable compositions are based on linear low-molecular polyorganosiloxane

rubbers with a molecular weight from 25000 to 100000, containing terminal silanol groups (Scheme 1) [6, 7]. Of greatest practical interest are: polydimethylsiloxanes **1**, polydimethylmethylphenylsiloxanes **2**, graft copolymers of α,ω -polydimethylsiloxanediol containing 1–3% of methylvinylsiloxane units and styrene in a ratio of 1 : 150–200, **3**, as well as polydimethylmethyltrifluoropropylsiloxanes **4**.

Silicon dioxide (pyrogenic or precipitated), carbon black, quartz sand, or pigment titanium dioxide are usually used as fillers [8, 9]. Organic peroxides, diphenylguanidine, tetraethoxysilane, silicon or organometallic compounds consisting of at least three functional groups are used as vulcanizing agents [10]. Salts of metals (Sn, Pb, Ti, Cr, Zn, etc.) and carboxylic acids, most often diethyl dicaprylate, dibutyl dicaprylate and dibutyl tin dilaurate are usually used as curing catalysts [11–13]. In this case, the performance of organosiloxanes in air is determined by temperatures in the range of 250–300°C [14–16]. In order to increase this temperature range, polysiloxanes containing phenyl and trifluoropropyl radicals or, more often, so-called superstructural polymers—block copolymers with a ladder structure—are usually used [16, 17]. However, since polymer materials are flammable, in order to increase the heat resistance and fire safety of silicone compositions, mineral fillers such as oxides, hydroxides, halides, oxyhalides, and carbonates of metals—Ni, Co, Al, Sn, Fe, Cr, Ti—are used as a rule. Zirconium and calcium silicates, aluminosilicates, etc., or

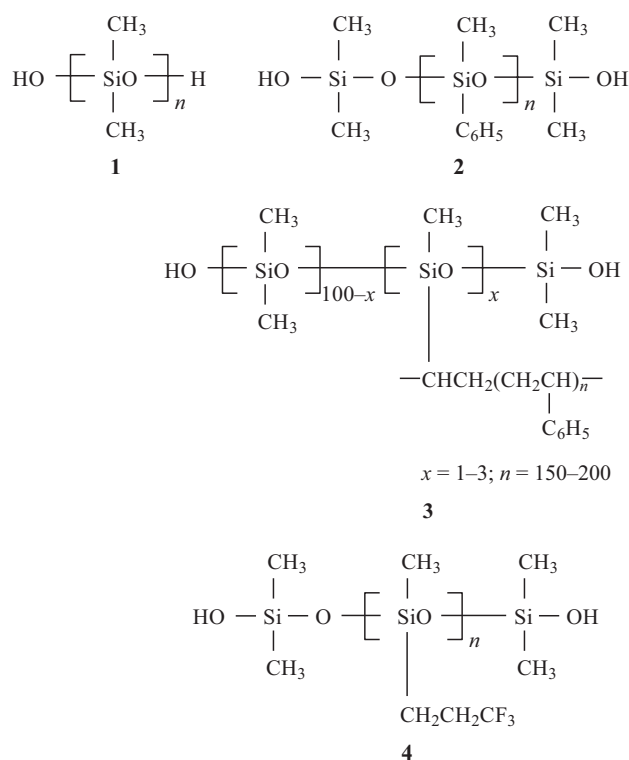
organic fillers, such as nitrogen-, boron-, phosphorus- and halogen-containing organic compounds, for example, melamine, phosphorus-containing diols and polyols can also be used [17, 18].

The use of azo compounds in addition to metal oxides is known to increase the thermal stability of silicone materials [19, 20]. However, the use of nitrogen-containing organosilicon compounds for these purposes is unknown. There is only a report on the use of γ -aminopropyltriethoxysilane as a modifier to increase the adhesion of silicone and natural rubbers to glass and plastics upon low-temperature vulcanization of rubber [21].

The simultaneous combination of heat-resistant and fire-resistant properties in one material is an important objective, so it seemed viable to study the possibility of using nitrogen-containing organosilicon compounds, in order to create heat- and fire-resistant filling compositions to seal high-voltage and high-frequency equipment.

EXPERIMENTAL

The basis for creating the compound was a linear low-molecular polyorganosiloxane rubber—styrosil—a graft copolymer of α,ω -polydimethylsiloxanediol **3** containing methylvinylsiloxane units, and styrene with a viscosity of 8–12 Pa·s. A mixture of ethyl silicate or tetraethoxysilane with dibutyltin oxide or with tin diethyl dicaprylate with the addition of a nitrogen-containing organosilicon compound was used as a vulcanizing agent (Table 1).



Scheme 1. Some linear low molecular weight organopolysiloxane rubbers with molecular weight from 25000 to 100000 containing end silanol groups

Table 1. Used nitrogen-containing organosilicon compounds

Substance	Manufacturer, country of origin	Reference to production procedure
Styrosil (low molecular weight polymer styrosil)	NIISK ¹ , St. Petersburg, Russia	—
Ethyl silicate (ethyl silicate-40, grade A, premium grade) (ethyl silicate-32)	EKOTEK Chemical Components Plant ² , Russia	—
Tetraethoxysilane (tetraethoxysilane, grade A, 98.5%)	EKOTEK Chemical Components Plant, Russia	—
Dibutyltin oxide (dibutyltin oxide, 98%)	Acros Organics ³ , Belgium	—
Tin diethyl dicaprylate (catalyst 230-15—solution of tin diethyl dicaprylate in ethyl silicate-32)	EKOTEK Chemical Components Plant, Russia	—
γ -Aminopropyltriethoxysilane (3-aminopropyltriethoxysilane, 99%)	Acros Organics, Belgium	—
Diethylcarbamic acid trimethylsilyl ester	—	[22]
Glycidyl methacrylate (glycidyl methacrylate, 97%, stable)	Acros Organics, Belgium	—
Triethoxysilane (triethoxysilane, 95%)	Acros Organics, Belgium	—
Diethylcarbamic acid triethoxysilyl ester	—	Diss. Dr. Sci. (Chem.) of L.O. Belova ⁴
Methyl formate (methyl formate, 97%)	Acros Organics, Belgium	—

Infrared (IR) spectra were recorded on a Nicolet 7600 spectrometer (*Thermo Fisher Scientific*, USA) in petroleum jelly or in a thin layer. Elemental analysis was carried out using a FLASH EA 1112 instrument (Italy). Differential scanning calorimetry was carried out on a differential scanning calorimeter DSC-822e (*Mettler Toledo*, Switzerland) at a heating rate of 10 deg/min. The dielectric properties were measured using a Novocontrol Alpha-A impedance analyzer (*Novocontrol Technologies*, Germany) and a ZGS Alpha Active Sample Cell (*Novocontrol Technologies*, Germany) with gold disk electrodes with a diameter of 20 mm. The measurements were performed in the frequency range of 10^{-1} – 10^6 Hz. The mechanical properties of the samples

were determined using a universal testing machine AGS-10kNG (*Shimadzu Corporation*, Japan). The samples used to determine the tensile strength and elongation at break were strips with a working part size of 3×20 mm. The stretching speed was 10 mm/min. The dynamic viscosity was determined using a Haake Viscotester C rotational viscometer (*Thermo Electron Corporation*, USA).

All starting compounds were thoroughly dried and purified by distillation before use. Synthetic operations, isolation and sampling for the analysis of compounds were carried out in a dry nitrogen atmosphere. Table 2 shows chemical formulas, names, and codes for used nitrogen-containing organosilicon compounds.

¹ <https://fgupniisk.ru/>. Accessed April 03, 2024.

² <https://www.eko-tec.ru/>. Accessed April 03, 2024.

³ <https://www.thermofisher.com/ru/ru/home/chemicals/acros-organics.html>. Accessed April 03, 2024.

⁴ Belova L.O. *New approaches to the synthesis and application of diazot-containing organosilicon compounds*: Diss. Dr. Sci. (Chem.). Moscow: 2011. 283 p. (in Russ.).

Table 2. Used nitrogen-containing organosilicon compounds

Chemical formula	Name	Code
$(\text{EtO})_2\text{Si}(\text{CH}_2)_3\text{NHC}(\text{O})\text{O}$	2,2-Diethoxy-1,6,2-oxaazasilepan-7-one	Product 111-269
$(\text{EtO})_3\text{Si}(\text{CH}_2)_3\text{NHC}(\text{O})\text{H}$	γ -(Triethoxysilylpropyl)-formamide	Product 111-300
$(\text{EtO})_3\text{SiOCH}_2\text{CH}(\text{NEt}_2)\text{CH}_2\text{OC}(\text{O})\text{C}(\text{Me})=\text{CH}_2$	2-(Diethylamino)-3-[(triethoxysilyl)oxy]propyl-2-methacrylate	ESAM
$(\text{EtO})_3\text{Si}(\text{CH}_2)_3\text{NHC}(\text{O})\text{OSi}(\text{OEt})_3$	Triethoxysilyl ester of γ -triethoxysilylpropyl-carbamic acid	OSU

2,2-Diethoxy-1,6,2-oxaazasilepan-7-one (product 111-269). A mixture of 15 g (0.07 mol) of γ -aminopropyltriethoxysilane and 12.83 g (0.07 mol) of diethylcarbamic acid trimethylsilyl ester was heated using a total refluxing head until the release of diethylamine and ethoxytrimethylsilane ceased. Product 111-269, 14.9 g (97%) was isolated by means of vacuuming in the form of a viscous oily substance, refractive index n_D^{20} 1.4459. IR spectrum (ν , cm^{-1}): 1690 (C=O); 3310 (N–H). Found, %: C 43.81; H 7.82; N 6.39. $\text{C}_8\text{H}_{17}\text{O}_4\text{NSi}$. Calculated C 47.96; H 9.77; N 7.99. In the dissertation of O.V. Belova (Footnote 4): n_D^{20} 1.4459. IR spectrum (ν , cm^{-1}): 1680 (C=O); 3310 (N–H).

3-(Diethylamino)-2-[(triethoxysilyl)oxy]propyl-2-methacrylate (ESAM). Diethylcarbamic acid triethoxysilyl ester, 15.9 g (0.057 mol) was slowly added to 8.1 g (0.057 mol) of glycidyl methacrylate. The reaction mixture was stirred at 55°C for 6 h. ESAM, 17.22 g (80%) was isolated by means of vacuuming at 1 mm Hg within 1 h in the form of a non-distillable liquid, n_D^{20} 1.4255. IR spectrum (ν , cm^{-1}): 1690 (C=O); 1640 (C=C). Found, %: C 54.15; H 9.45; N 3.91. $\text{C}_{17}\text{H}_{35}\text{O}_6\text{NSi}$. Calculated C 54.08; H 9.34; N 3.70.

Triethoxysilyl ester of γ -triethoxysilylpropyl-carbamic acid (OSU). A mixture of 141.6 g (0.64 mol) of γ -aminopropyltriethoxysilane and 105 g (0.64 mol) of triethoxysilane was heated to 45°C, and carbon dioxide was introduced with stirring for a period of 12 h. OSU, 245.5 g (96%) was obtained by evacuation at 1 mm Hg within 1 h in the form of a non-distillable liquid, n_D^{20} 1.4244. IR spectrum (ν , cm^{-1}): 1690 (C=O); 3340 (N–H). Found, %: C 44.85; H 8.85; N 3.31. $\text{C}_{16}\text{H}_{37}\text{O}_8\text{NSi}_2$. Calculated C 44.94; H 8.7; N 3.27.

γ -(Triethoxysilylpropyl) formamide (product 111-300). 23.4 g (0.11 mol) of γ -aminopropyltriethoxysilane and 6.6 g (0.11 mol) of methyl formate were placed in

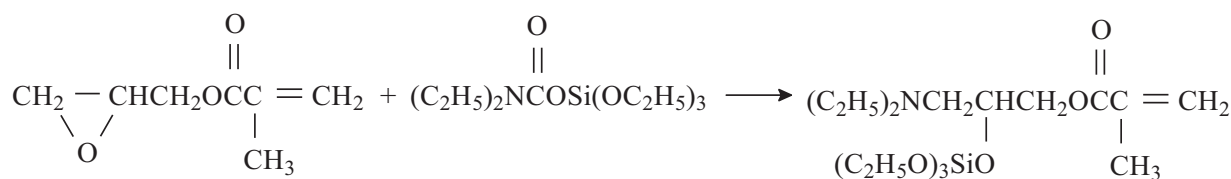
a flask equipped with a thermometer and a distillation column head. The reaction mixture was heated. After separating the released methyl alcohol, distillation of the still bottom gave 23.84 g (86.9%) of product 111-300, boiling point 146–147°C (2 mm Hg), n_D^{20} 1.4390. IR spectrum (ν , cm^{-1}): 1680 (C=O); 3310 (N–H). Found, %: C 48.07; N 9.30; N 5.40. $\text{C}_{10}\text{H}_{23}\text{O}_4\text{NSi}$. Calculated C 48.16; H 9.31; N 5.61.

RESULTS AND DISCUSSION

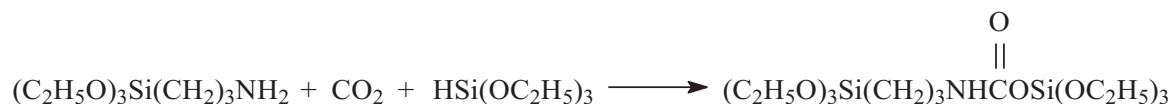
We had previously conducted research on the possibility of using nitrogen-containing organosilicon compounds as catalysts for the curing of silicone rubbers [24, 25]. The resulting coatings possessed frost resistance, high dielectric properties, increased mechanical and electrical strength, and adhesion. However, they turned out to be flammable which did not always make it possible for them to be used, especially in the electrical insulation of power and radio equipment.

Continuing these studies, first of all product 111-269 (2,2-diethoxy-1,6,2-oxaazasilepan-7-one) and ethyl silicate-40, which had worked well previously, were used as a hardener. Then a number of other first synthesized nitrogen-containing organosilicon compounds were used (Table 1). ESAM was synthesized using diethylcarbamic acid triethoxysilyl ester (Scheme 2). OSU was obtained by *N*-siloxycarbonylation (Scheme 3), and product 111-300 was obtained by formylation (Scheme 4).

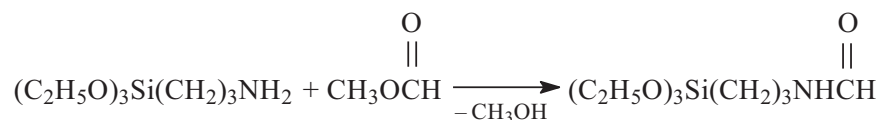
Two compositions were used as fillers: a mixture containing Aerosil (filler based on hexabromobenzene (GB filler), see composition in Table 3) and a mixture without Aerosil (filler based on hexabromobenzene (DB filler), see composition in Table 4). The results of testing the compositions using a range of vulcanizing agents and fillers are presented in Tables 5–11.



Scheme 2. Preparation of 3-(diethylamino)-2-[(triethoxysilyl)oxy]propyl-2-methacrylate (ESAM) using triethoxysidyl ester of diethylcarbamic acid



Scheme 3. Preparation of triethoxysilyl ester of γ -triethoxysilylpropylcarbamic acid (OSU) by *N*-siloxy carbonylation



Scheme 4. Preparation of γ -(triethoxysilylpropyl) formamide (product 111-300) by formylation

Table 3. Composition of the filler based on hexabromobenzene (GB filler)

Substance	Weight parts
Hexabromobenzene	49.5
Melamine	30.0
Aluminum hydroxide (pure)	14.0
Calcium carbonate	6.0
Aerosil 200	0.5

Table 4. Composition of the filler based on decabromodiphenyl oxide (DB filler)

Substance	Weight parts
Decabromodiphenyl oxide	47.8
Melamine	31.4
Aluminum hydroxide (pure)	14.8
Calcium carbonate	6.0

Table 5. Influence of the type of filler and the degree of its filling on the viscosity of rubber, the physical and mechanical properties of the vulcanizate, and the ability to self-extinguish. Hardener, weight parts (wt.p.): ethyl silicate-40, 2.6; dibutyltin oxide, 0.025

Parameter	Value								
Stirosil (9.0 Pa·s), wt.p.	100	100	100	100	100	100	100	100	100
DB filler, wt.p.	–	30	–	40	–	50	–	60	–
GB filler, wt.p.	–	–	30	–	40	–	50	–	60
Dynamic viscosity, Pa·s	9.0	13.5	16.6	18.3	21.5	28.3	33.0	35.0	38.0
Viability at 20°C, min	>120	40	40	40	40	40	40	30	30
Tensile strength, MPa	2.45	2.55	1.86	2.45	2.06	1.18	0.98	2.45	2.06
Relative elongation, %	120	110	100	85	120	85	110	105	80
Compression module at 20°C, MPa	2.94	–	–	2.45	–	2.84	–	3.14	–
Crystallization temperature, °C	–60	–	–	–60	–	–60	–	–60	–
Dielectric constant at 10 ³ Hz, 20°C	3.2	–	–	3.2	–	3.4	–	3.5	–
tg δ* at 10 ³ Hz, 20°C	0.002	–	–	0.0051	–	0.0087	–	0.0073	–
Self-extinguishing, s	–	2	2	1.6	1.5	1.5	1.5	1.5	2
Electrical strength, kV/mm	35	–	–	40	–	38	–	33	–

*tg δ (dielectric loss tangent) is defined as the ratio of the active component of the leakage current through the insulation to its reactive component.

Table 6. Influence of the curing system on the technological and physicomachanical properties of the compound. Hardener (2 wt.p. — 100%): tetraethoxysilane — 80%; product 111-269 — 20%; tin diethyldicaprylate — 0.02%

Parameter	Value
Stirosil (3.3–9.0 Pa·s), wt.p.	100
DB filler, wt.p.	–
GB filler, wt.p.	–
Dynamic viscosity, Pa·s	8–9
Viability at 20°C, min	120
Tensile strength, MPa	1–2.7
Relative elongation, %	70–210
Compression module at 20°C, MPa	1.96–2.94
Curing time at 20°C, day	1–2
Crystallization temperature, °C	–60

Table 6. Continued

Parameter	Value
Dielectric constant at 10^3 Hz, 20°C	3.6
tg δ at 10^3 Hz, 20°C	0.002
Self-extinguishing, s	–
Electrical strength, kV/mm	24–30
Heat resistance, °C	150

Table 7. Influence of the curing system on the technological and physicomachanical properties of the compound. Hardener, 2 wt.p.: tetraethoxysilane — 80%; ESAM — 20%; tin diethyldicaprylate — 0.02%

Parameter	Value				
Stirosil (3.3–9.0 Pa·s), wt.p.	100	100	100	100	100
DB filler, wt.p.	–	–	40	50	60
GB filler, wt.p.	–	30	–	–	–
Dynamic viscosity, Pa·s	9.0	16.6	18.3	28.3	35.0
Viability at 20°C, min	120	120	120	90	90
Tensile strength, MPa	2.16	1.96	2.26	2.35	2.06
Relative elongation, %	113	100	120	90	90
Compression module at 20°C, MPa	2.75	3.14	2.75	2.65	3.04
Curing time at 20°C, day	1	1	1	1	1
Crystallization temperature, °C	–60	–60	–60	–60	–60
Dielectric constant at 10^3 Hz, 20°C	3.2	3.2	3.3	3.4	3.5
tg δ at 10^3 Hz, 20°	0.0018	0.0039	0.0047	0.0104	0.0072
Self-extinguishing, s	–	2	2	1	1.6
Electrical strength, kV/mm	27	30	30	–	40
Heat resistance, °C	150	–	–	–	–

Table 8. Influence of the curing system on the technological and physicomechanical properties of the compound. Hardener, 2 wt.p.: ESAM — 20%; tin diethyldicaprylate — 0.03%

Parameter	Value				
Stirosil (3.3–9.0 Pa·s), wt.p.	100	100	100	100	100
DB filler, wt.p.	–	–	40	50	60
GB filler, wt.p.	–	30	–	–	–
Dynamic viscosity, Pa·s	9.0	16.6	18.3	28.3	35.0
Viability at 20°C, min	120	90	90	90	60
Tensile strength, MPa	1.27	1.37	1.47	1.47	1.47
Relative elongation, %	250	150	180	170	130
Compression module at 20°C, MPa	2.16	2.16	2.45	2.75	2.84
Curing time at 20°C, day	1–2	1	1	1	1
Crystallization temperature, °C	–60	–60	–60	–60	–60
Dielectric constant at 10 ³ Hz, 20°C	–	3.2	3.3	3.4	3.4
tg δ at 10 ³ Hz, 20°C	–	0.0052	0.0053	0.0074	0.0083
Self-extinguishing, s	–	–	2	–	–
Electrical strength, kV/mm	–	–	36	–	–

Table 9. Influence of the curing system on the technological and physicomechanical properties of the compound. Hardener, 2–3 wt.p.: product 111-300 — 100%; tin diethyldicaprylate — 0.02%

Parameter	Value		
Stirosil (3.3–9.0 Pa·s), wt.p.	100	100	100
DB filler, wt.p.	40	50	60
Dynamic viscosity, Pa·s	18.3	28.3	35.0
Viability at 20°C, min	40	40	30
Tensile strength, MPa	2.55	–	2.35
Relative elongation, %	90	–	80

Table 9. Continued

Parameter	Value		
Compression module at 20°C, MPa	2.45–3.43	2.45–2.94	2.55–2.84
Curing time at 20°C, day	1	1	1
Crystallization temperature, °C	–60	–60	–60
Dielectric constant at 10 ³ Hz, 20°C	3.4	–	3.5
tg δ at 10 ³ Hz, 20°C	0.0050	–	0.0184
Self-extinguishing, s	1	2	2
Electrical strength, kV/mm	36	–	–
Heat resistance, °C	180	–	–

Table 10. Influence of the curing system on the technological and physicochemical properties of the compound. Hardener, 2 wt.p: tetraethoxysilane — 80%; ESAM — 20%; tin diethyldicaprylate — 0.02%

Parameter	Value		
Stirosil (3.3–9.0 Pa·s), wt.p.	100	100	100*
DB filler, wt.p.	–	40	40
GB filler, wt.p.	30	–	–
Dynamic viscosity, Pa·s	16.6	18.3	18.3
Viability at 20°C, min	120	120	0.5
Tensile strength, MPa	2.35	2.35	–
Relative elongation, %	100	120	
Compression module at 20°C, MPa	2.84	3.24	2.55–2.84
Curing time at 20°C, day	1	1	1
Crystallization temperature, °C	–60	–60	–60
Dielectric at 10 ³ Hz, 20°C	–	3.2	–

Table 10. Continued

Parameter	Value		
tg δ at 10 ³ Hz, 20°C	—	0.006	—
Self-extinguishing, s	2	1–2	—
Electrical strength, kV/mm	—	32	—
Heat resistance, °C	170	176	130

*3–4 wt.p. — 100%; tetraethoxysilane — 80%; ESAM — 20%; tin diethyldicaprylate — 0.02%.

Table 11. Influence of the curing system on the technological and physicochemical properties of the compound. Hardener, 2–3 wt.p.: OSU — 100%; tin diethyldicaprylate — 0.02%

Parameter	Value		
Stirosil (3.3–9.0 Pa·s), wt.p.	100	100	100
DB filler, wt.p.	—	40	40
GB filler, wt.p.	30	—	—
Dynamic viscosity, Pa·s	17.5	18.3	18.3
Viability at 20°C, min	90	90	90
Tensile strength, MPa	2.45	2.75	2.75
Relative elongation, %	170	210	210
Compression module at 20°C, MPa	3.63	3.92	3.92
Curing time at 20°C, day	1	1	1
Crystallization temperature, °C	–60	–60	–60
Dielectric constant at 10 ³ Hz, 20°C	3.2	3.2	3.2
tg δ at 10 ³ Hz, 20°C	0.0006	0.0007	0.0007
Self-extinguishing, s	0.7	0.5	0.5
Electrical strength, kV/mm	31	35	35
Heat resistance, °C	180	190	190

Table 12. Effect of the curing system [ESAM + tin diethyldicaprylate] on the properties of rubber vulcanizates

Parameter	Value						
ESAM	3 wt.p.						
Tin diethyldicaprylate, wt.p.	0.164	0.02	0.016	0.005	0.004	0.003	0.002
Stirosil (9.0 Pa·s), wt.p.	60	60	60	60	60	60	60
DB filler, wt.p.	40	40	40	40	40	40	40
Viability at 20°C, min	No	<10	30	60	90	90	120
Curing time at 20°C, day	0.33	0.5	1	<24	<24	24	24

Table 13. Influence of the curing system [ESAM + tin diethyldicaprylate on the properties of rubber vulcanizates]

Parameter	Value			
Tin diethyldicaprylate, wt.p.	0.003			
ESAM, wt.p.	2	3	5	8
Stirosil (9.0 Pa·s), wt.p.	60	60	60	60
DB filler, wt.p.	40	40	40	40
Viability at 20°C, min	120	90	90	60
Tensile strength, MPa	1.67	1.47	1.47	–
Relative elongation, %	160	185	180	–
Curing time at 20°C, day	48	24	24	48

The results presented (Tables 5–11) clearly indicate that self-extinguishing, i.e., decay time after removal from the flame, s, of the filled compositions, regardless of the amount of filler used (30–60 wt.p.), attained value 1–2 s, while their heat resistance increased at the same time from 150 to 180°C.

The data obtained (Tables 5–11) on tensile strength, relative elongation and compression modulus shows

that the introduction of filler has virtually no effect on the mechanical properties of the polymer, as well as on the crystallization temperature of the compositions. In addition, it is clearly shown that GB filler, compared to DB filler, increases the polymer system viscosity to a greater extent (apparently due to the presence of aerosil), while the degree of filling significantly affects the increase in viscosity. For example, with a viscosity

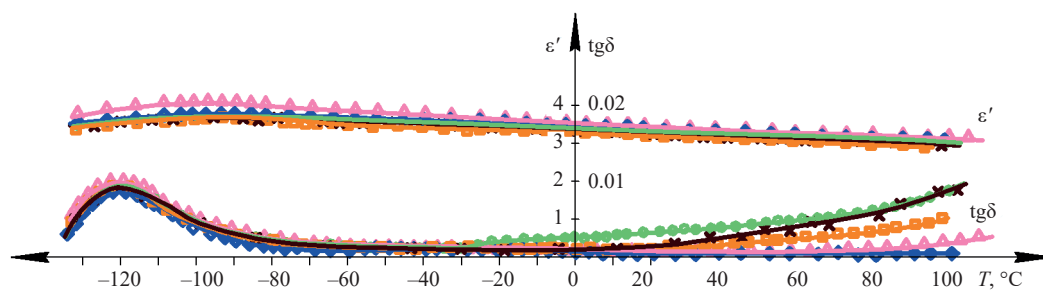


Fig. 1. Temperature dependence of $\text{tg}\delta$ and ϵ' on the type of hardener at a frequency of 1000 Hz. Hardener: ●●● ethyl silicate 40 + dibutyltin oxide; xxx product 111-300; ▲▲▲ tetraethoxysilane + ESAM; ○○○ OSU; *** ESAM

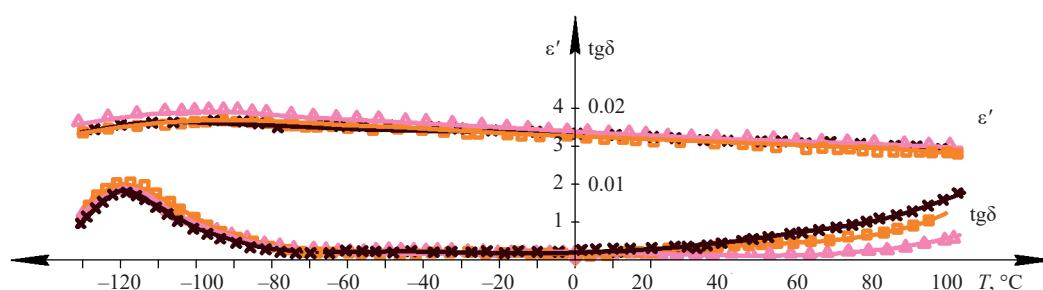


Fig. 2. Temperature dependence of $\text{tg}\delta$ and ϵ' on the amount of hardener ESAM at a frequency of 1000 Hz: ●●● 2; xxx 3; and ▲▲▲ 5 wt.p. hardener per 100 wt.p. polymer

of the original rubber of 9.0 Pa·s, the introduction of 40 wt.p. of a filler increases its value approximately by a factor of 2, while using 60 wt.p. of a filler, viscosity becomes equal to 35.0 Pa·s. The constant value of the compositions crystallization temperature can be explained by the inertness of the fillers with respect to styrosil.

When studying the dielectric properties of the resulting compositions, which characterize the behavior of the material in an electric field, the dielectric constant (ϵ'), dielectric losses ($\text{tg}\delta$) and electrical strength (E_s) were considered in detail. They depend on the compound operating conditions and are closely related to the macrochain chemical composition and structure. From the results obtained it is clear that a wide asymmetric maximum of dipole-segmental losses can be observed in the temperature range from -90 to -130°C (at a frequency of 1000 Hz) (Fig. 1). The asymmetry of the $\text{tg}\delta$ maximum appears to be due to the influence of the supramolecular organization on the polymer electrical properties. As a rule, this effect is especially pronounced for crystallizing polymers: something which we observed for this composition. In contrast, in the temperature

range from -90 to $+100^\circ\text{C}$, the value of the dielectric loss tangent remains practically unchanged and is about 0.005, which allows us to recommend its use at high frequencies in this temperature range.

The influence of the brand and amount of hardener on the value of the dielectric loss tangent (Fig. 2) is manifested both in the region of positive temperatures (as the amount of hardener increases, losses increase slightly, since the polar component amount increases) and in the region of subzero temperatures (fluctuations in $\text{tg}\delta_{\text{max}}$ values and relaxation time spectrum are observed, apparently due to the influence of functional groups on molecular mobility).

However, the differences observed in the dependence of the dielectric loss tangent $\text{tg}\delta$ on temperature are so small that they can be neglected for the practical use of the compound. At the same time, the dielectric properties of the filled compositions change somewhat with an increase in the $\text{tg}\delta$ value. For compositions containing up to 40 wt.p. of filler, the $\text{tg}\delta$ value does not exceed 0.005, and at a higher degree of filling, it increases to 0.01, exceeding the permissible value for a high-voltage, high-frequency dielectric.

Thus, after the introduction of fillers, a compound using nitrogen-containing organosilicon compounds as part of the curing system retains high dielectric properties, increased electrical and mechanical strength and frost resistance. This is characteristic of high-voltage and high-frequency compounds. At the same time, the compound acquires non-flammable properties with a simultaneous increase in heat resistance.

CONCLUSIONS

The study shows that the use of synthetically available nitrogen-containing organosilicon compounds—3-(diethylamino)-2-[(triethoxysilyl)oxy]propyl-2-methacrylate and triethoxysilyl ester of γ -triethoxysilylpropylcarbamic acid—as part of a curing system together with bromine-containing fillers makes it possible for compounds to be obtained to fill high-voltage and high-frequency transformers, throttles and other elements of radio-electronic equipment with non-flammable

properties, while simultaneously increasing heat resistance.

The physicochemical and dielectric properties of a self-extinguishing organosilicon compound were studied, showing that it is manufacturable, has a high fire resistance (no more than 10 s) and increased heat resistance (up to 180°C). The compound retains high dielectric properties ($\tan\delta$ no more than 0.005) and electrical strength (not less than 25 kV/mm), good mechanical properties (tensile strength not less than 1.5 MPa) with high elasticity (relative elongation not less than 140%).

Authors' contributions

A.D. Kirilin—idea of the study and general management.

L.O. Belova, N.I. Kirilina—the analysis of the obtained results and writing the text of the article.

N.A. Golub, M.V. Pletneva—conducting the experiments, scientific editing.

D.E. Mironov—conducting the experiments.

The authors declare that there is no conflict of interest.

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