SYNTHESIS AND PROCESSING OF POLYMERS AND POLYMERIC COMPOSITES

СИНТЕЗ И ПЕРЕРАБОТКА ПОЛИМЕРОВ И КОМПОЗИТОВ НА ИХ ОСНОВЕ

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RESEARCH ARTICLE

Influence of iron ore concentrate (magnetite) on the kinetics of butadiene–styrene rubber-based blend curing in the presence of different accelerators

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Objectives. *To investigate the possibility of using a cheaper ingredient, such as magnetite, in the synthesis of rubber compounds based on butadiene–styrene rubber by examining its effect on the process of sulfuric vulcanization of butadiene–styrene rubber in the presence of various accelerators.*
Methods. *The influence of magnetite on the vulcanization kinetics was studied using an Alpha Technologies PRPA 2000 rotorless rheometer. Thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC) were performed using a Mettler Toledo TGA/DSC 2 device to evaluate the effect of magnetite on the butadiene–styrene rubber-based vulcanizates' oxidation.*
Results. *Magnetite was found to affect the kinetics of SBR-1500 butadiene–styrene rubber sulfuric vulcanization in the presence of thiazole-type accelerators (2-MBT, 2-MBS); in contrast, magnetite was inactive in the case of diphenylguanidine, sulfenamide T, and tetramethylthiuram disulfide. The obtained TGA/DSC data showed that magnetite has no significant effect on the butadiene–styrene rubber-based vulcanizates' oxidation and thermal destruction.*
Conclusions. *The obtained data confirmed magnetite's capability to act as a butadiene–styrene rubber sulfuric vulcanization activator in the presence of various accelerators. The most significant effect was observed in the presence of thiazole-type accelerators.*

Keywords: *iron ore concentrate, magnetite, butadiene–styrene rubber, vulcanization activator, kinetics of curing, thermogravimetric analysis, differential scanning calorimetry*

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НАУЧНАЯ СТАТЬЯ

Влияние железорудного концентрата (магнетита) на кинетику вулканизации резиновых смесей на основе бутадиен-стирольного каучука в присутствии различных ускорителей

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Цели. *Изучить возможность применения в рецептурах резиновых смесей на основе бутадиен-стирольного каучука более дешевого ингредиента – магнетита путем оценки его влияния на процесс серной вулканизации бутадиен-стирольного каучука в присутствии различных ускорителей.*

Методы. *Влияние магнетита на кинетику вулканизации исследовали с помощью безроторного реометра AlphaTechnologies PRPA 2000. Методами термогравиметрического анализа (ТГА) и дифференциально-сканирующей калориметрии (ДСК) оценили влияние магнетита на процесс окисления вулканизатов на основе бутадиен-стирольного каучука на приборе Mettler Toledo TGA/DSC 2.*

Результаты. *Показано, что магнетит влияет на кинетику серной вулканизации бутадиен-стирольного каучука SBR-1500 в присутствии ускорителей тиазолового ряда (дибензотиазолдисульфид, 2-меркаптобензотиазол), в то время как в случае с 1,3-дифенилгуанидином, сульфенамидом Т (N-трет-бутил-2-бензтиазолсульфенамид) и тиурамом (тетраметилтиурамдисульфид) магнетит малоактивен. Данные, полученные с ТГА/ДСК, демонстрируют, что магнетит незначительно влияет на окисление, а также на термодеструкцию вулканизатов на основе бутадиен-стирольного каучука.*

Выводы. *Исследовано влияние магнетита на кинетику процесса серной вулканизации бутадиен-стирольного каучука в присутствии различных ускорителей. Наибольший эффект наблюдается в присутствии ускорителей тиазолового ряда.*

Ключевые слова: *железорудный концентрат, магнетит, бутадиен-стирольный каучук, активатор вулканизации, кинетика вулканизации, термогравиметрический анализ, дифференциально-сканирующая калориметрия*

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INTRODUCTION

The choice of ingredients for synthesizing various rubber compounds is critical in the production of serial elastomeric products. When developing a new elastomeric compound, three main ingredient characteristics must be considered during compounding—the properties, manufacturability, and price of the elastomeric material [1]. These three characteristics are crucial in the manufacturing sector because certain ingredients can significantly enhance the vulcanization parameters and properties of the final product. However, such products are unaffordable in the market as their costs increase exponentially. Thus, the chemical analysts face the challenge of a) improving product quality, b) attributing it a set of new properties, c) reducing the concentration of expensive system ingredients, d) lowering the synthesis cost without compromising the technological characteristics of the mixtures, and e) reducing the operational properties of the finished product. Therefore, the search for new rubber compound ingredients is an urgent task [2–5].

In previous works [6, 7], the effect of magnetite on the kinetics of butadiene–styrene rubber SKS-30 ARK vulcanization in the absence of zinc oxide was studied. Therein, magnetite acted as an activator of sulfur vulcanization. However, the characteristics of the vulcanizates obtained with its use are slightly inferior to the properties of standard rubbers.

MATERIALS AND METHODS

The primary objective of this work is to study the kinetics of black-extended elastomeric material vulcanization based on SBR-1500 butadiene–styrene rubber in the presence of the classic vulcanizing group and with other sulfur vulcanization accelerators to investigate the potential use of magnetite in the composition of sulfuric vulcanizing systems as an active component that enhances vulcanization parameters of rubber compounds. In addition, the effect of different magnetite contents on the aging process of elastomeric material was examined by studying the nature of the variation in the thermogrvimetric analysis (TGA) curves and differential scanning calorimetry (DSC) of SBR-1500-based vulcanizates.

Herein, we used iron ore concentrate containing more than 69.5% mass fraction of iron, TU 0712-030-001186803-99 (*Lebedinsky Mining and Processing Plant*, Russia).

The dispersion of the iron ore concentrate powder was investigated using an Analysette 22 MicroTec Plus laser diffractometer manufactured by *Fritsch GmbH*, Germany.

RESULTS AND DISCUSSION

The particle size distribution (Fig. 1) is unimodal (from 0.1 to 100 µm). The maximum differential distribution *dQ* of the volume fraction of particles falls on ~20-µm-sized particles. The integral dependence of the volume fraction *Q* of particles on the size shows that 50% and 90% of the iron ore concentrate particles are less than 15 and 40 µm in size, respectively.

Figure 2 shows the X-ray diffractogram of the iron ore concentrate powder obtained on an HZG-4 X-ray diffractometer (*Helmholtz-Zentrum Geesthacht*, Germany) (Ni filter): CuKα = 1.54051 Å on a diffracted beam in the step-by-step mode with a pulse acquisition time of 10 s and a step value of 0.02° in the angular range of 2Θ = 2°–80°.

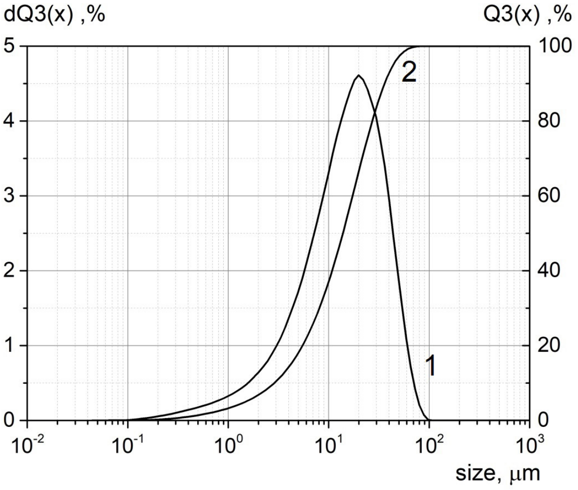

Fig. 1. Differential *dQ* (1) and integral *Q* (2) curves of iron ore concentrate powder particle distribution (obtained using Analysette 22 MicroTec Plus, *Fritsch GmbH*, Germany).

The iron ore concentrate used was magnetite, as evidenced by diffraction reflections at 2Θ values of ~36°, ~57°, and ~63°. The resulting spectrum is consistent with that obtained from the international database of diffraction standards, the Crystallography Open Database[1]. In the angular range of 2Θ = 16°–26°, an amorphous halo and diffraction reflections characteristic of quartz can be observed at 2Θ = ~27° and ~41° [8]. Silicon dioxide content determined from the diffraction patterns via semiquantitative analysis (using the Match! software package, *CRYSTAL IMPACT GbR*, Germany)[2] is about 5–6%.

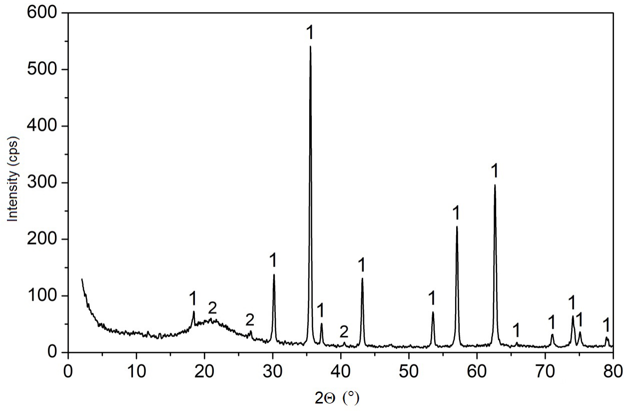

Fig. 2. Diffraction pattern of iron ore concentrate obtained from the *Lebedinsky Mining and Processing Plant*: (1) Fe_3O_4 phase; (2) SiO_2 phase.

The rubber compounds were prepared in a Brabender laboratory rubber mixer (*Brabender GmbH & Co KG*, Germany) at a chamber temperature of *T* = 60°C and an angular speed of rotor rotation, ω = 60–63 rpm, with the total mixing time being t < 10 min. The mixing was performed in two stages. In the first stage, SBR-1500 butadiene–styrene rubber, zinc oxide (ZnO), stearic acid, and fillers—magnetite, carbon black (CB) N339, or their mixtures having various compositions—were introduced; then, the mixture was cooled. In the second stage, the curing system was introduced, and the resulting mixtures were finalized on rollers.

The amount of magnetite in the mixtures based on SBR-1500 varied in the range of 0, 25, and 50 per hundreds of rubber (phr) at a constant concentration

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[1] Crystallography Open Database. URL: http://www.crystallography.net.

[2] URL: http://www.crystalimpact.com/match/Default.htm

of CB N339: 50 phr (see Table 1). In the case of altax (2-MBS) and sulfenamide T (TBBS), a formulation corresponding to GOST 15627-79[1] and GOST ISO 2322-2013[2] was used. For the remaining accelerators, TBBS accelerators were replaced with an equimolar amount of tetramethylthiuram disulfide (TMTD), diphenylguanidine (DPG), and captax (2-MBT) accelerators in accordance with ISO 2322:2009. In the case of mixtures containing DPG and 2-MBT as accelerators, the accelerator content was increased to 3.0 phr because at lower concentrations (vulcanization temperature T_V = 160°C, process duration τ = 60 min) the torque in the time curve did not reach the vulcanization plateau.

Vulcanization parameters were determined using an RPA 2000 rotorless rheometer (*Alpha Technologies*, USA). Rheometric curves were plotted at a temperature of T_V = 160°C for τ = 60 min.

TGA/DSC studies were performed on samples based on SBR-1500 manufactured according to ISO 2322:2009, with a constant total filler content of 50 phr. The dosage of magnetite was increased from 0 to 50 phr in increments of 10 phr, and the dosage of CB N339 was decreased in the same order. Measurements were conducted on a TGA/DSC 2 device (*Mettler Toledo*, USA) in an atmosphere of nitrogen and oxygen in the temperature range of 25–250°C, at a heating rate of 10 °C/min.

The following parameters were determined based on the kinetic curves—optimal vulcanization time (t'90), scorch time(t_S1), minimum (M_L), and maximum (M_H) torque. Table 2 shows the dependence of these parameters on magnetite content in rubber compounds based on SBR-1500.

Different magnetite contents influence the time taken to reach the vulcanization optimum and scorch time as well as the minimum and maximum torque in the presence of different accelerators. The time taken to reach the vulcanization optimum t′90 decreases at T_V = 160°C in the presence of the TBBS, 2-MBS, DPG, and 2-MBT accelerators as magnetite content increases. In addition, the maximum effect manifested in the case of 2-MBT (t'90 decreases from 36 to 19 min upon the introduction of 50 phr of magnetite). In the case of TMTD, variation in magnetite content has practically no effect on the time taken to achieve optimum vulcanization. The scorch time in the presence of the TBBS and 2-MBT accelerators decreases with increasing magnetite concentration. In the case of DPG and TMTD, as the magnetite content varies, the dependence of the vulcanization time passes through a maximum, and in the case of 2-MBS, through a minimum. Notably, the change in the absolute value of scorch time is not significant in all cases. The maximum torque in the presence of all accelerators increases linearly

Table 1. Formulations of elastomeric mixtures based on butadiene–styrene rubber SBR-1500 and magnetite with different accelerators

Component	Component content, phr														
	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15
SBR-1500	100	100	100	100	100	100	100	100	100	100	100	100	100	100	100
ZnO	3.00	3.00	3.00	5.00	5.00	5.00	3.00	3.00	3.00	3.00	3.00	3.00	3.00	3.00	3.00
Sulfur	1.75	1.75	1.75	2.00	2.00	2.00	1.75	1.75	1.75	1.75	1.75	1.75	1.75	1.75	1.75
Stearic acid	1.00	1.00	1.00	1.50	1.50	1.50	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00
TBBS	1.00	1.00	1.00	–	–	–	–	–	–	–	–	–	–	–	–
2-MBS	–	–	–	3.00	3.00	3.00	–	–	–	–	–	–	–	–	–
TMTD	–	–	–	–	–	–	1.00	1.00	1.00	–	–	–	–	–	–
DPG	–	–	–	–	–	–	–	–	–	3.00	3.00	3.00	–	–	–
2-MBT	–	–	–	–	–	–	–	–	–	–	–	–	3.00	3.00	3.00
CBN339	50.0	50.0	50.0	40.0	40.0	40.0	50.0	50.0	50.0	50.0	50.0	50.0	50.0	50.0	50.0
Magnetite	0.0	25.0	50.0	0.0	25.0	50.0	0.0	25.0	50.0	0.0	25.0	50.0	0.0	25.0	50.0

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[3] GOST 15927-79. State Standard. Synthetic rubbers butadiene-methylstyrene СКМС-30 АРК and butadiene-styrene СКС-30 АРК. Specifications. Moscow: IPK Izd. Stand.; 2003.

[4] GOST ISO 2322-2013. State Standard. Styrene-butadiene rubber (SBR) of emulsion- and solution-polymerized types. Evaluation methods. Moscow: Standartinform; 2014.

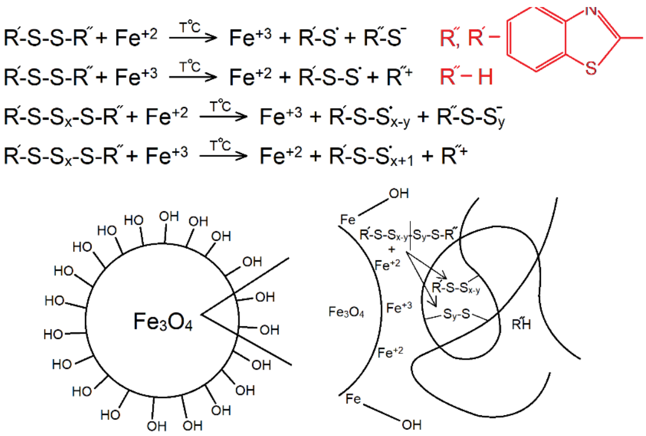
Table 2. Dependence of vulcanization characteristics on magnetite concentration in black-extended mixtures based on SBR-1500 in the presence of various accelerators

Accelerator type	Accelerator content, phr	CB N339 content, phr	Magnetite content, phr	Vulcanization characteristics			
				$t'90$, min	t_S1, min	M_L, dyn·m	M_H, dyn·m
TBBS	1.00	50.0	0.0	15.84	5.32	2.8	22.41
	1.00	50.0	25.0	14.88	4.81	2.74	24.63
	1.00	50.0	50.0	12.54	4.55	3.42	26.31
2-MBS	3.00	40.0	0.0	14.62	2.5	1.87	19.65
	3.00	40.0	25.0	12.54	2.06	2.06	21.22
	3.00	40.0	50.0	11.59	2.41	2.4	23.41
TMTD	1.00	50.0	0.0	6.85	1.1	2.95	29.63
	1.00	50.0	25.0	6.26	1.26	3.16	31.67
	1.00	50.0	50.0	6.57	1.28	3.58	34.69
DPG	3.00	50.0	0.0	32.86	2.45	2.58	18.73
	3.00	50.0	25.0	31.93	2.71	2.64	19.48
	3.00	50.0	50.0	31.23	2.49	2.82	20.07
2-MBT	3.00	50.0	0.0	36.29	1.83	2.7	16.99
	3.00	50.0	25.0	23.97	1.33	2.93	20.69
	3.00	50.0	50.0	19.22	1.3	2.98	23.09

(at different speeds) with increasing magnetite content, with a constant content of CB (see Table 2).

According to the above data, magnetite activates the sulfur vulcanization process. The maximum effect of magnetite introduction is observed in the presence of thiazole-type accelerators; this suggests that Fe^{+2} and Fe^{+3} ions on the surface of magnetite particles catalytically accelerate disulfide bond decomposition (scheme) through a mechanism similar to radical polymerization initiation by redox systems based on iron(II) and iron(III) salts [9, 10]. The radicals formed by the decomposition of accelerator disulfide bonds subsequently activate the sulfur vulcanization process.

Thermograms of TGA vulcanizates (Figs. 3 and 4) under nitrogen atmosphere show that when the magnetite is introduced into the rubber mixture based on SBR-1500 in an amount equivalent to the concentration of CB N339 in the standard mixture (50 phr), less weight loss occurred than in the case of samples containing only CB N339, in the entire temperature range. A similar effect occurred in an oxygen atmosphere, wherein the curves are characterized by an increase in mass in the temperature range up to 100°C. This is most likely due to the accumulation of oxidation products.



Scheme. Supposed interaction between magnetite particles and butadiene–styrene rubber macromolecules in the presence of accelerators containing disulfide bonds.

The DSC data presented in Fig. 5 (in an inert medium of N_2) indicates that magnetite practically does not accelerate the thermal decomposition (endothermic process) of the vulcanizates. Slight differences were observed only in the temperature range above 150°C.

Similar results were obtained when studying the processes of thermal oxidative degradation in an O_2

medium (Fig. 6). In this case, the thermal oxidation of the vulcanizates begins at 190–220°C. An exothermic peak appears in the temperature range of 210–230°C, and shifts to lower temperatures as the magnetite concentration increases. No significant activating effect of magnetite is observed in these processes.

However, as stated in [11], iron cations of variable valency in the polymer phase accelerate oxidative processes several times. This is true only in cases wherein iron salts are at least partially dissolved in the polymer phase. In the case of magnetite, iron cations of variable valency capable of causing oxidation are available only on the particle surfaces. This considerably complicates the oxidative process' catalysis.

CONCLUSIONS

The above data confirm the capability of magnetite to act as an active agent in the process of sulfuric vulcanization of butadiene–styrene rubber in the presence of various accelerators. Herein, the most significant effect of magnetite, as an active component of the vulcanizing system, manifested in the presence of thiazole-type accelerators (2-MBS and 2-MBT). In addition, the presence of magnetite in SBR-1500-based vulcanizates practically did not affect the oxidation process in the operating temperature range of most elastomeric products.

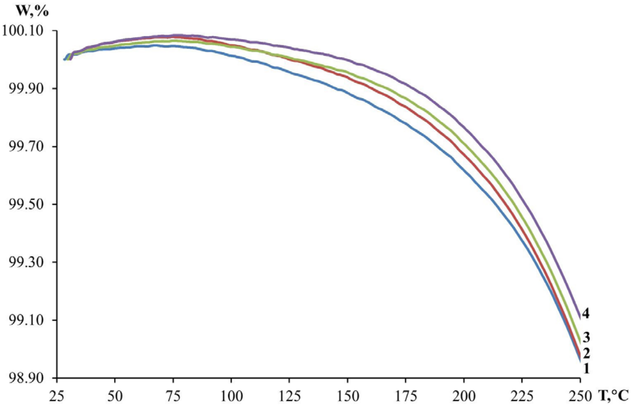


Fig. 3. TGA patterns for vulcanizates based on SBR-1500 with different magnetite and CB N339 (in nitrogen atmosphere) contents: (1) 50 phr of N339 + 0 phr of magnetite; (2) 30 phr of N339 + 20 phr of magnetite; (3) 10 phr of N339 + 40 phr of magnetite; and (4) 0 phr of N339 + 50 phr of magnetite.

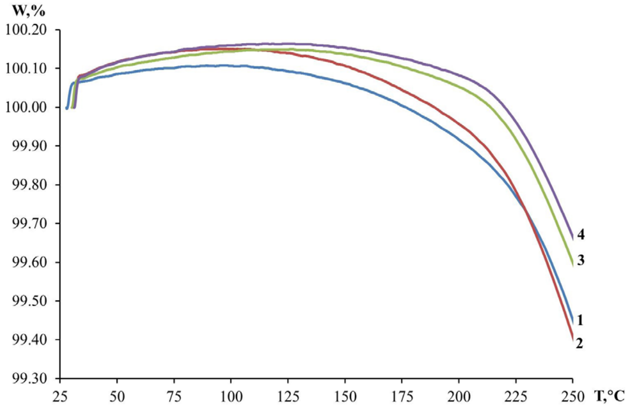


Fig. 4. TGA patterns for vulcanizates based on SBR-1500 with different magnetite and CB N339 (in oxygen atmosphere) contents: (1) 50 phr of N339 + 0 phr of magnetite; (2) 30 phr of N339 + 20 phr of magnetite; (3) 10 phr of N339 + 40 phr of magnetite; and (4) 0 phr of N339 + 50 phr of magnetite.

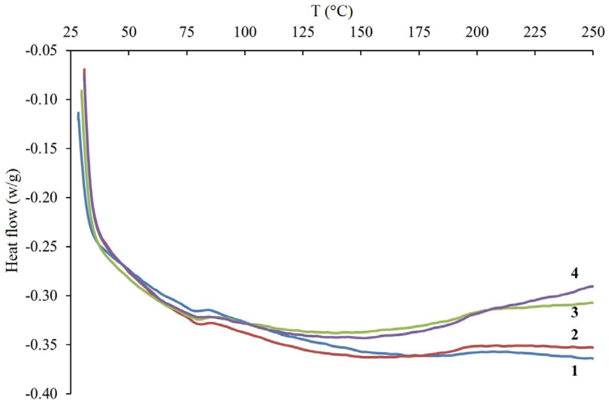


Fig. 5. DSC pattern for vulcanizates based on SBR-1500 with different magnetite and CB N339 (in nitrogen atmosphere) contents: (1) 50 phr of N339 + 0 phr of magnetite; (2) 30 phr of N339 + 20 phr of magnetite; (3) 10 phr of N339 + 40 phr of magnetite; and (4) 0 phr of N339 + 50 phr of magnetite.

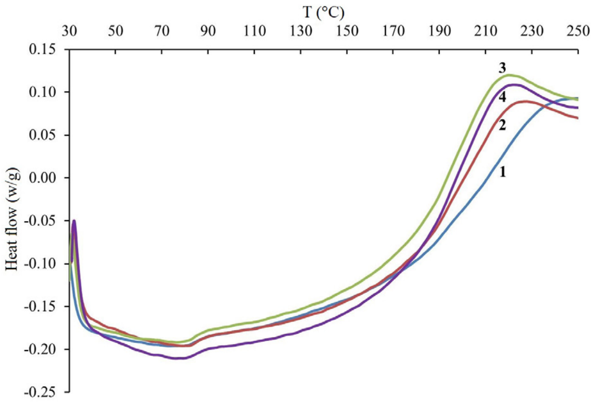


Fig. 6. DSC pattern for vulcanizates based on SBR-1500 with different magnetite and CB N339 (in oxygen atmosphere) contents: (1) 50 phr of N339 + 0 phr of magnetite; (2) 30 phr of N339 + 20 phr of magnetite; (3) 10 phr of N339 + 40 phr of magnetite; and (4) 0 phr of N339 + 50 phr of magnetite.

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Authors' contribution

A.A. Khachaturov – literature analysis, article writing, research planning; carrying out all the stages of the study, formalization of the list of references;

E.E. Potapov – idea, development of the research design, consultation on the problems of carrying out all the stages of the study;

S.V. Reznichenko – consultation on the problems of planning, methodology and implementation of the study;

A.N. Kovaleva – scientific editing, technical editing, formalizing the bibliography.

The authors declare no conflicts of interest.

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