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USE OF PURIFIED ADSORBENT DERIVED FROM SUNFLOWER OIL PRODUCTION AS A BIOFILLER FOR ELASTOMERIC COMPOSITIONS FOR THE MANUFACTURE OF RUBBER PRODUCTS

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The article presents the results of a study on the use of a two-stage purified diatomite-based adsorbent derived from sunflower oil production (AS-SOpur$_2$) as a biofiller for elastomeric compositions based on carbon-chain diene rubbers intended for the manufacture of rubber products. It has been established that the introduction of a biofiller increases the plasticity of rubber compounds, i.e., improves their processability compared to compositions filled with kaolin, which contributes to a reduction in energy costs at the mixing and processing stages. It has been shown that AS-SOpur$_2$ has an intensifying and anti-reversion effect on the sulfur vulcanization process: the time to reach the vulcanization optimum is reduced, the cross-linking rate and the apparent rate constant increase, and the vulcanization plateau expands. It has been established that rubbers with AS-SOpur$_2$ are characterized by a more stable complex of physical and mechanical properties both under normal conditions and after various types of aging. The results obtained indicate that the diatomaceous adsorbent purified using a two-stage scheme can effectively replace kaolin as a functional biofiller. The use of AS-SOpur$_2$ is promising in view of the environmental, resource-saving, and economic feasibility of using secondary raw materials in elastomeric compositions.

Keywords: elastomeric composition, biofiller, sunflower oil production waste, properties of rubber compounds and vulcanizates, renewable bio-raw materials, environmental safety.

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Introduction

Modern scientific and technical developments in the field of elastomeric materials technology are characterized by a trend toward expanding the use of light-colored dispersed mineral fillers. This trend is primarily due to the established toxicological and environmental hazards associated with the use of carbon black [1,2]. At the same time, the high material costs of importing silicon dioxide, as well as the significant energy and resource intensity of the technological processes of its synthesis, are driving active research into the development of alternative sources of natural mineral biofillers [3,4]. In this context, the strategy of expanding the range of fillers for elastomeric compositions by utilizing secondary resources (waste) from domestic industries is particularly relevant, which is promising from the standpoint of closed-loop economy principles and

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environmental safety.

In the process of sunflower oil production, after the winterization stage, a significant amount of spent filter adsorbent is produced in the form of a powdery, finely dispersed material, on the surface and in the pores of which there are residues of vegetable oil, waxes, etc. [5]. The presence of organic components makes this waste prone to oxidation by atmospheric oxygen (exothermic reaction), which creates a risk associated with self-heating and potential spontaneous combustion during storage and transportation. According to the Ukrainian state classifier «Waste Classifier DK 005-96,» the used filter powder from sunflower oil production is classified as hazard class IV. In view of the principles of sustainable development and the circular economy, it is important to find technological solutions aimed at using this waste in other industries. The use of products obtained from used filter powder in elastomeric compositions will simultaneously solve the environmental problem of reducing industrial waste and reduce the consumption of traditional mineral fillers for rubber.

The single-stage purification method we propose for used filter powder allows mineral and organic components to be separated [6,7]. The use of the organic component [6], for example, as a plasticizer-softener in the composition of an elastomeric compound for the manufacture of the tread part of tires, compared to the industrial petrochemical lubricant Nytex 4700, allows to increase the cohesive strength of the rubber mixture by 1.5 times, while maintaining vulcanization parameters, ensuring 30–40% higher resistance to reversion and relaxation processes at 100^0C, lower dynamic losses, and high physical and mechanical characteristics. Studies of the fat- and wax-containing fraction obtained by heat treatment at temperatures of 100^0C and 160^0C (FWF-100 and FWF-160, respectively) in the composition of filled and unfilled model elastomeric compositions based on butadiene-α-methylstyrene rubber [7] showed the superiority of the FWF-100 sample. The studied additive FWF-100 allows, in filled compositions, to ensure high activity and degree of cross-linking at low activation energy of the sulfur vulcanization process, to form a general complex of physical and mechanical characteristics of rubbers at the level of stearic acid, and to improve fatigue endurance by 1.3 times.

In the composition of elastomeric compounds for the manufacture of tire elements, it was found that FWF-100 can be recommended as a multifunctional additive that has three main positive effects: 1) a technological additive that increases the tackiness and cohesive strength of rubber compounds by up to 20%; 2) a thermal destruction stabilizer that preserves a quarter of the elastic and strength properties of rubber; 3) an anti-fatigue agent with 30% efficiency [7].

The two-stage purification method for used filter powder allows separating mineral-containing powder, fat- and wax-containing fractions, and wax fraction (OS Wax) separately. According to the results of experimental studies [8], it has been established that 1.0–2.0 phr of OS Wax in the composition of elastomeric compounds for the manufacture of tire elements acts as a technological additive. Thus, it reduces the effective activation energy of sulfur vulcanization, initiates and deepens the process of sulfur vulcanization, reduces heat generation, and has a positive effect on Payne effect indicators while maintaining the dynamic and physical-mechanical properties characteristic of rubbers with petrochemical microwax of the SVOZ-75 U brand.

The mineral component (AS-SOpur product) obtained as a result of single-stage purification of filter powder was studied in model elastomeric compositions based on isoprene rubber [9]. A positive effect of AS-SOpur was established when kaolin (20.0 phr) was replaced in equal weight on the formation of elastic and strength characteristics of rubbers under the influence of elevated temperatures. A comparative analysis of the AS-SOpur product and the AS-SOpr sample obtained after additional annealing of AS-SOpur was carried out in the composition of tread-type elastomeric compositions based on general-purpose rubbers SKD and SKMS-30 ARKM-15 with an equal replacement (10.0 phr) of Ultrasil VN 2 GR silicon dioxide. It has been shown that the AS-SOpr product is more effective in the processing of rubber compounds, allowing to maintain balanced parameters of the sulfur vulcanization kinetics and a high level of cross-linking. However, against the background of the optimal dosage of reinforcing technical carbon in tread rubber and in relation to vulcanizates with Ultrasil VN 2 GR, the products AS-SOpur and AS-SOpr do not have a reinforcing effect.

For the reduction of the AS-SOpur product's organic content, but not for its complete dehydrophobization, a two-stage purification of the spent filter powder with various solvents (AS-SOpur$_2$ product) was carried out. A comparative analysis of diatomaceous products of single-stage and two-stage purification as fillers was carried out for model elastomeric compositions based on butadiene-a-methylstyrene rubber [10]. It is shown that the hydrophobized products under study significantly affect the processibility of rubber compounds, the course of sulfur vulcanization at different temperatures, the level of rheokinetic characteristics, and the density of crosslinks of elastomeric compositions. It has been established that in terms of the rubber reinforcement factor, the spent adsorbent with 37 wt.% of the organic component (AS-SOpur$_2$ product), regenerated by two-stage washing with dichloroethane and petroleum ether, is superior to a product with 57 wt.% organic component (AS-SOpur product), as well as chalk, but inferior to kaolin. Product AS-SOpur$_2$ can be recommended for use in industrial rubber formulations.

Therefore, this work aimed to experimentally evaluate the possibility of using a diatomaceous adsorbent produced from sunflower oil (AS-SOpur$_2$ product), purified using a two-stage scheme, as a biofiller for industrial-type elastomeric compositions for the manufacture of rubber products.

Experimental

Materials

The AS-SOpur$_2$ product samples for research were prepared using a two-stage purification method described earlier [8], which involves the selective separation of the used sunflower oil winterization adsorbent with various solvents. In the first stage, the fat- and wax-containing fractions were separated using dichlorethane, and in the second stage, waxes were removed using petroleum ether. The mineral adsorbent was dried at room temperature under suction to a constant weight, resulting in a purified filter powder, the AS-SOpur$_2$ product.

The AS-SOpur$_2$ product was a finely dispersed, light beige, odorless powder. The physico-chemical characteristics of AS-SOpur$_2$ were studied in comparison with traditional mineral rubber fillers (Table 1) used in the work.

The AS-SOpur$_2$ product has the highest residue on a 014 sieve and the lowest bulk density compared to traditional fillers, which, based on the dibutyl phthalate (DBF) absorption index, indicates that the purified filter powder is a porous material with a developed specific surface area. Increased weight loss at 105^{0}C for 2 hours and upon further heating may indicate the presence of organic residue on the surface of the AS-SOpur$_2$ product, in addition to the existing moisture, as the samples were dried to a constant weight at room temperature.

Research on the complete replacement (17.0 phr) of P-2 kaolin with AS-SOpur$_2$ was conducted in elastomeric compositions for the production of beating fingers for feather removal devices from poultry carcasses. The elastomeric base was a combination of natural rubber of SVR 60 CV brand and polybutadiene rubber of BR 44 brand. A sulfur vulcanizing group was used in rubber mixture formulations.

The base rubber compounds were prepared in a PC-250-40 rubber mixer with a mixing chamber volume of 250 L under standard conditions. The additives under study were introduced into the base rubber compound on rollers under identical mixing conditions.

The vulcanization of test samples was carried out under optimal conditions on hydraulic presses at a temperature of 150^{0}C.

Research methods

The properties of elastomeric compositions were evaluated using instrumental methods in accordance with current standards and known techniques [11]. The rheological and vulcanization properties of elastomeric compositions were studied according to international standards DIN 53 529, ASTM D 6204, ASTM D 6601, ISO 6502-2, ASTM D 2084 using a shear vibro-rheometer from Monsanto and RheoCheck MD. Based on the rheometry data, vulcanization parameters were calculated, in particular: rate, conditional rate constant, and temperature coefficient according to the conditional rate constant. To evaluate the reinforcing effect of the studied fillers, the reinforcement coefficient (I^3) was determined [12]:

$$I_3 = \frac{f_{300} \cdot f_p \cdot B}{f_{300}^0 \cdot f_p^0 \cdot B^0},$$

where f_{300} and f^0_{300} are the conditional stress at 300% elongation of rubber with and without the studied additive, respectively (MPa); f_p and f^0_p are the conditional tensile strength of rubber with and without the studied additive, respectively (MPa); B and B^0 are the tear resistance of rubber with and without the studied additive, respectively (kN·m).

Table 1

Physico-chemical characteristics of studied additives

Parameter	Value	
	P-2 kaolin	AS-SOpur$_2$ product
Residue on a 014 sieve, %	3.9	4.7
Bulk density, g/cm^3	0.322	0.234
Absorption index DBF, ml/100 g (by the D 2414 method)	121.0	176.0
Moisture content (T=105^{0}C; 2 hours), wt.%	0.64	2.11
Mineral content, wt.%	89.6	63.0

Results and discussion

Analysis of the technological characteristics of rubber compounds showed that, unlike kaolin, which reduces the plasticity of rubber compounds when added, the AS-SOpur$_2$ product increases this indicator and increases thermoplasticity at a test temperature of 150^{0}C by 29%, and at 160^{0}C by 14% (Fig. 1).

This trend in changing characteristics, especially the increase in thermoplasticity, probably indicates the presence of organic residues in the form of a thin film on the surface of the filter powder, which makes the surface of the mineral less polar and more compatible with the rubber matrix. This, in turn, reduces internal friction in the rubber compound matrix and promotes the mobility of polymer chains when heated.

The increase in thermoplasticity when AS-SOpur$_2$ is added reduces energy consumption during the processing of rubber compounds and could potentially become critically important for possible subsequent technological operations.

Analysis of vulcanization kinetic curves at temperatures of 150^{0}C and 160^{0}C (Fig. 2) showed that when AS-SOpur$_2$ is introduced, and kaolin is replaced in equal amounts, the induction period is shortened, the vulcanization rate in the main period increases, and the highest values of the maximum torque are achieved. At the same time, rubbers with AS-SOpur$_2$ are characterized by a wider vulcanization plateau and a lower reaction rate of destruction.

The vulcanization characteristics calculated from the kinetic curves (Table 2) indicate that the results of the technological properties study correlate with the minimum torque and, accordingly, with the viscosity of rubber compounds. While at a temperature of 150^{0}C, the thermoplasticity and the minimum torque moments of rubber compounds with kaolin and AS-SOpur$_2$ are almost identical, then with the temperature increase to 160^{0}C, the thermoplasticity of the compound with kaolin is higher, and the viscosity and minimum torque moment are lower. The relative degree of rubber cross-linking when adding AS-SOpur$_2$ increases to 13%, and the process is also significantly intensified, namely, the time to reach the optimum vulcanization is reduced to 16%, and the vulcanization rate increases to 32%.

Changing the vulcanization time parameters affects the conditional rate constant, which increases by 1.6 times at a temperature of 150^{0}C when using the AS-SOpur$_2$ product. The temperature coefficient (K_{10}), which characterizes the change in the process rate with the temperature increase by 10^{0}C, is minimal in rubber with AS-SOpur$_2$. That is, when using the studied additive, the cross-linking process is less dependent on the temperature, and the product will vulcanize more evenly.

Unlike kaolin, which intensifies the reversion process in rubber (Table 2), the AS-SOpur$_2$ product reduces the relative degree of change in torque at the reversion stage (ΔS_r) by an average of 2.2 times compared to rubber without fillers and by an average of 3.6 times compared to kaolin at different vulcanization temperatures. The introduction of AS-SOpur$_2$ is accompanied by a decrease in the reversal rate (V_r) by an average of 2.4 times, and compared to kaolin, by an average of 3.9 times.

Thus, the AS-SOpur$_2$ product not only accelerates and deepens the cross-linking process of the studied elastomeric compositions, but also provides a wide vulcanization plateau. That is, having an

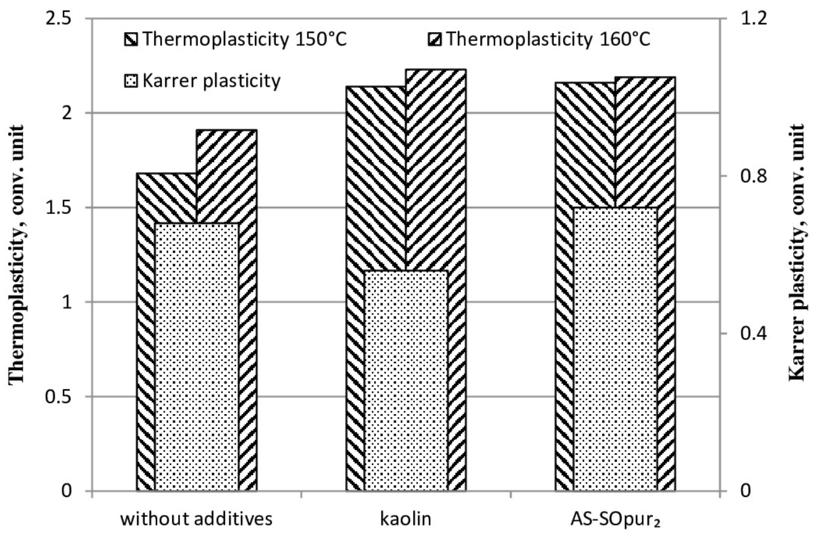

Fig. 1. Technological properties of rubber compounds

initiating action, AS-SOpur$_2$ allows the creation of activated sulfur vulcanization systems with increased resistance to reversion.

Analysis of the physical and mechanical characteristics showed that the trend in the change in the values of the conditional stress of rubber at both 100% and 300% elongation correlates with the change in the relative degree of cross-linking: the maximum value is characteristic of rubber containing the AS-SOpur$_2$ product, and the minimum value is characteristic of rubber without the studied additives (Table 3). This effect may be associated with the formation of physicochemical «rubber-filler» bonds, which act as additional mesh nodes. A direct consequence of this effect is an increase in Shore hardness under various test conditions. The rebound elasticity of rubber containing AS-SOpur$_2$ has the lowest value.

The main advantages of using AS-SOpur$_2$ in rubber compounds are noted in terms of conditional tensile strength, relative elongation at break, and tear resistance (Fig. 3).

When filling the elastomeric composition with 17.0 phr of the AS-SOpur$_2$ product, the conditional tensile strength increases (Fig. 3a) by 18% after rubber aging (at 100^{0}C for 72 hours). Under the same conditions, the relative elongation at break increases by 13% (Fig. 3b). Unlike kaolin, which reduces the tear resistance index (Fig. 3c), the introduction of AS-SOpur$_2$ allows increasing this indicator under normal test conditions by 1.2 times compared to rubber without additives, and by 1.5 times compared to kaolin. After aging, the tear resistance increases by 1.4 times and 1.7 times, respectively.

Increased tear resistance and growth of conditional tensile strength may be a consequence of

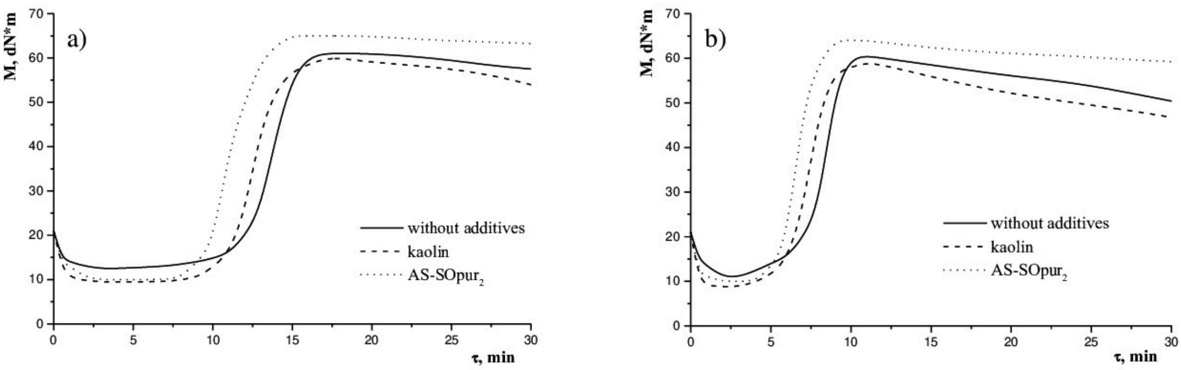

Fig. 2. Kinetic curves of sulfur vulcanization of elastomeric compositions at 150^{0}C (a) and 160^{0}C (b)

Table 2

Vulcanization properties of elastomeric compositions (test conditions: T=150^{0}C/160^{0}C*; τ=30 min)

Property	Value		
	without additives	kaolin	AS-SOpur$_2$
Minimum torque (S_{Min}), dN·m	12.5/11.0*	9.8/8.8*	10.0/10.0*
Maximum torque (S_{Max}), dN·m	61.0/60.0*	60.0/59.0*	65.0/64.0*
Relative degree of crosslinking ($\Delta S=(S_{Max}-S_{Min})$), dN·m	48.5/49.0*	50.2/50.2*	55.0/54.0*
Scorching resistance (Scorch Time (TS1)), min	10.10/2.75*	9.40/2.80*	8.70/2.90*
Time to reach 50% vulcanization (TC50), min	12.6/5.5*	12.4/5.2*	11.1/4.3*
Time to reach 90% vulcanization (TC90), min	15.3/9.2*	14.2/8.8*	12.8/7.8*
Vulcanization rate (R_V=100/TC90–TS1), min^{-1}	19.23/15.50*	20.83/16.67*	24.39/20.41*
Conditional constant of vulcanization rate (k_2=1.608/TC90–TC50), min^{-1}	0.596/0.435*	0.893/0.447*	0.946/0.459*
Temperature coefficient for indicator k_2, ($K_{10}=k_{2\,160}/k_{2\,150}$)	0.73	0.50	0.49
Relative degree of change in torque at the reversion stage ($\Delta S_r=(S_{Max}-S_{30})/S_{Max}\cdot 100$), %	6.56/18.33*	13.33/22.88*	3.08/7.81*
Reversal rate ($V_r=(S_{Max}-S_{30})/(t_{30}-t_{Max})$), dN·m/min	0.31/0.57*	0.64/0.71*	0.13/0.24*

the high structurization of the AS-SOpur$_2$ product, which provides effective mechanical fixation and partial immobilization of rubber macromolecules in the surface layer of filler particles [13,14], which determines the increased resistance of rubber to tensile and tear loads [15].

The reinforcing effect of the studied fillers was evaluated using the calculated reinforcement coefficient (I^3), which is 1.5 times higher in rubber containing the AS-SOpur$_2$ product than in rubber containing kaolin (Table 3), which indicates a more effective participation of the AS-SOpur$_2$ filler in the formation of a spatial network in the elastomeric matrix, presumably due to more uniform dispersion of particles and a reduction in their agglomeration.

Thus, the use of the AS-SOpur$_2$ product allows forming the increased strength and wearing resistance, high tear resistance, controlled elasticity, and stable properties of the rubber under conditions of thermal and mechanical influence of external factors. This balance of properties is optimal for the working parts (beating fingers) of feather removal devices, where the rubber must work softly and not break down under mechanical stress and increased temperatures.

Thus, experimental studies have confirmed the feasibility of using a two-stage purification product for used sunflower oil winterization adsorbent (AS-SOpur$_2$) in an elastomeric composition based on a combination of SVR 60 CV natural rubber and polybutadiene rubber BR 44 for the production of beating fingers for feather removal devices from poultry carcasses. As an ingredient, the AS-SOpur$_2$ product combines the functions of a semi-active filler, a sulfur vulcanization process modifier, and an anti-reversion additive, providing a balanced set of technological and physico-mechanical properties of rubber compounds and rubbers.

Conclusions

The overall set of experimental studies demonstrates the effect of the diatomaceous AS-SOpur$_2$ product in elastomeric compositions based on carbon-chain diene rubbers as a specific biofiller. The use of AS-SOpur$_2$ improves plasticity and thermoplasticity, resulting in better processability of rubber compounds and a potential reduction in energy consumption during mixing. During the vulcanization process, the product under study exhibits an intensifying and deepening effect in the main period of the cross-linking process and a certain anti-reversion effect with an expansion of the vulcanization plateau and a decrease in the rate of reversal processes. The pronounced effect of the AS-SOpur$_2$ product as a semi-reinforcing biofiller has been revealed, which allows for an increase in the elasticity and strength characteristics of rubber while maintaining sufficient elasticity. The positive effects obtained from the use of the AS-SOpur$_2$ product may be associated with the high structure, porosity, and partial organic modification (hydrophobization) of the surface of the studied product particles, which increases their compatibility with the rubber matrix.

Considering the combination of the obtained effects, AS-SOpur$_2$ can be recommended for further practical implementation as an effective multifunctional biofiller for modern elastomeric compositions for the manufacture of rubber and other products.

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Table 3

Physical and mechanical properties of elastomeric compositions

Property	Value		
	without additives	kaolin	AS-SOpur$_2$
Conditional stress at 100% elongation (f_{100}), MPa under normal conditions	0.6	0.8	1.0
Conditional stress at 300% elongation (f_{300}), MPa under normal conditions	1.3	1.9	2.0
Shore hardness A (H), rel. units under normal conditions	43	45	49
after aging 100^0C^*72 h	48	51	52
Rebound elasticity (S), % under normal conditions	60	60	56
after aging 100^0C^*72 h	63	62	60
Reinforcement coefficient (I^3)	–	1.33	2.03

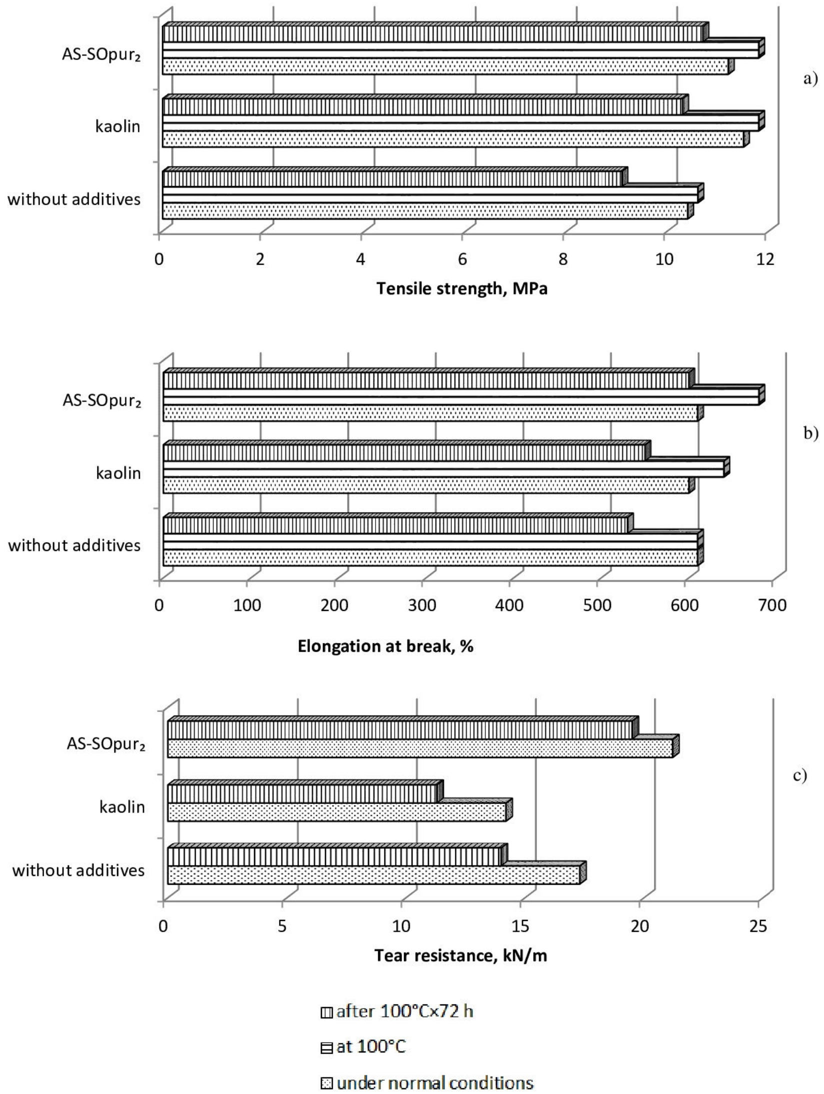

Fig. 3. Stress-elongation properties (a – tensile strength, b – elongation at break, c – tear resistance) of elastomeric compositions (under normal conditions and after aging)

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ЗАСТОСУВАННЯ ОЧИЩЕНОГО АДСОРБЕНТУ ВИРОБНИЦТВА СОНЯШНИКОВОЇ ОЛІЇ ЯК БІОНАПОВНЮВАЧА ЕЛАСТОМЕРНИХ КОМПОЗИЦІЙ ДЛЯ ВИГОТОВЛЕННЯ ГУМОТЕХНІЧНИХ ВИРОБІВ

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У статті наведено результати дослідження застосування очищеного за двостадійною схемою діатомітового адсорбенту виробництва соняшникової олії (АС-СОоч$_2$) як біонаповнювача еластомерних композицій на основі карболанцюгових дієнових каучуків, призначених для виготовлення гумотехнічних виробів. Встановлено, що введення біонаповнювача забезпечує підвищення пластичності гумових сумішей, тобто покращує їх перероблюваність порівняно з композиціями, наповненими каоліном, що сприяє зниженню енерговитрат на стадіях змішування та перероблення. Показано, що АС-СОоч$_2$ проявляє інтенсифікуючий та антиреверсійний вплив на процес сірчаної вулканізації: скорочується час досягнення оптимуму вулканізації, зростає швидкість процесу зшивання та умовна константа швидкості, а також розширюється плато вулканізації. Встановлено, що гуми з АС-СОоч$_2$ характеризуються більш стабільним комплексом фізико-механічних властивостей як за нормальних умов, так і після різних видів старіння. Отримані результати свідчать, що очищений за двостадійною схемою діатомітовий адсорбент може ефективно замінювати каолін як функціональний специфічний біонаповнювач. Застосування АС-СОоч$_2$ є перспективним з огляду на екологічну, ресурсозберігаючу та економічну доцільність використання вторинної сировини у складах еластомерних композицій.

Ключові слова: еластомерна композиція; біонаповнювач; відходи виробництва соняшникової олії; властивості гумових сумішей та гум; поновлювана біосировина; екологічна безпека.

USE OF PURIFIED ADSORBENT DERIVED FROM SUNFLOWER OIL PRODUCTION AS A BIOFILLER FOR ELASTOMERIC COMPOSITIONS FOR THE MANUFACTURE OF RUBBER PRODUCTS

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The article presents the results of a study on the use of a two-stage purified diatomite-based adsorbent derived from sunflower oil production (AS-$SOpur_2$) as a biofiller for elastomeric compositions based on carbon-chain diene rubbers intended for the manufacture of rubber products. It has been established that the introduction of a biofiller increases the plasticity of rubber compounds, i.e., improves their processability compared to compositions filled with kaolin, which contributes to a reduction in energy costs at the mixing and processing stages. It has been shown that AS-$SOpur_2$ has an intensifying and anti-reversion effect on the sulfur vulcanization process: the time to reach the vulcanization optimum is reduced, the cross-linking rate and the apparent rate constant increase, and the vulcanization plateau expands. It has been established that rubbers with AS-$SOpur_2$ are characterized by a more stable complex of physical and mechanical properties both under normal conditions and after various types of aging. The results obtained indicate that the diatomaceous adsorbent purified using a two-stage scheme can effectively replace kaolin as a functional biofiller. The use of AS-$SOpur_2$ is promising in view of the environmental, resource-saving, and economic feasibility of using secondary raw materials in elastomeric compositions.

Keywords: elastomeric composition; biofiller; sunflower oil production waste; properties of rubber compounds and vulcanizates; renewable bio-raw materials; environmental safety.

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