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FEATURES OF ELASTOMERIC COMPOSITIONS IN THE PRESENCE OF REGENERATED SUNFLOWER OIL PRODUCTION WASTE AS FILLERS

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Abstract

The paper investigates the peculiarities of the properties of elastomeric compositions based on butadiene-α-methylstyrene rubber of the Buna KER 1723 brand in the presence of regenerated sunflower oil production waste in comparison with the equal weight content (20.0 phr) of known mineral fillers. It has been 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 cross-links 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 (diatomite P), regenerated by two-stage washing with dichloroethane and petroleum ether, is superior to a product with 57 wt. % organic component (diatomite D), as well as chalk, but inferior to kaolin. Diatomite P can be recommended for use in industrial rubber formulations.

Keywords: elastomeric composition; filler; bioingredient; sunflower oil production waste; sunflower oil purification adsorbent; environmentally friendly rubber.

ОСОБЛИВОСТІ ЕЛАСТОМЕРНИХ КОМПОЗИЦІЙ ЗА НАЯВНОСТІ РЕГЕНЕРОВАНИХ ВІДХОДІВ ВИРОБНИЦТВА СОНЯШНИКОВОЇ ОЛІЇ ЯК НАПОВНЮВАЧІВ

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Анотація

У роботі досліджено особливості властивостей еластомерних композицій на основі бутадієн-α-метилстирольного каучуку марки Buna KER 1723 за наявності регенерованих відходів виробництва соняшникової олії в порівнянні з рівномасовим вмістом (20.0 мас.ч.) відомих мінеральних наповнювачів. Показано, що гідрофобізовані досліджувані продукти суттєво впливають на технологічність гумових сумішей, перебіг сірчаної вулканізації за різних температур, рівень реокінетичних характеристик і щільність поперечних зшивок еластомерних композицій. Встановлено, що за коефіцієнтом посилення гум регенерований двохстадійним відмиванням дихлоретаном і петролейним етером відпрацьований адсорбент (діатоміт П) з 37 мас.% органічної складової переважає продукт з 57 мас.% органічної складової (діатоміт Д), а також крейду, але поступається каоліну. Діатоміт П може бути рекомендований до застосування в рецептурах гум промислового типу.

Ключові слова: еластомерна композиція; наповнювач; біоінгредієнт; відходи виробництва соняшникової олії; адсорбент очищення соняшникової олії; екологічно безпечна гума.

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Journal of Chemistry and Technologies, 2025, *33*(2), 427-435

Introduction

Current trends in the field of green technologies involve the creation of low-waste or zero-waste production technologies that ensure the integrated use of natural raw materials and the reuse of products [1–3]. The market requires waste utilization and the creation of new materials based on waste. This not only expands the range of products, but also frees up territories from dumps and sludge pits for agricultural use, and improves the overall environmental situation in the country [4–6].

The production of vegetable oil is a powerful sector of Ukraine's agro-industrial complex, with sunflower oil remaining the main type. To purify and bleach sunflower oil, a filtration system is used, the main element of which is sorption materials of natural origin - diatomite filter powders, bentonite clay, etc. Thousands of tons of filter powder, which is a safety class 4 waste, are used and often taken to landfills, worsening the state of the environment. In addition, when air oxygen is exposed to the developed surface of the used adsorbents and the temperature rises, intensive oxidation and subsequent spontaneous combustion begin, releasing a significant amount of harmful gases and other substances into the atmosphere [8–11]. Taking into account the volume of sunflower oil production in Ukraine, the issue of processing and utilization of waste masses is becoming increasingly relevant.

The main methods of utilization of waste from the oil and fat industry at the stage of sunflower oil vinification are their use in livestock farming, in the production of construction and paint and varnish materials, disposal at solid waste landfills, and thermal neutralization. But given the presence of up to 70 wt. % of organic substances – esters of high molecular weight monobasic fatty acids and high molecular weight monoatomic alcohols, unsaturated and saturated hydrocarbons [8–11], it can be involved in the production cycle as secondary material resources to obtain and replace scarce and expensive chemical additives of petrochemical origin, mineral fillers of polymer composite materials.

According to the results of experimental studies, we have established the possibility of obtaining regenerated inorganic and organic products from sunflower oil production wastes that can exhibit a semi-amplifying effect as fillers (inorganic component) and act as technological additives (organic component) of elastomeric compositions based on diene rubbers [12–14]. The regeneration of hydrophobized mineral adsorbent by annealing at a temperature of 800 °C is an extremely energy-consuming method. It is likely to be accompanied by the destruction of its crystal structure [8], which leads to the production of rubbers with unsatisfactory dynamic characteristics [12]. Therefore, the purpose of this work was to study further the possibility of using a more appropriate method of deep regeneration of the hydrophobized adsorbent using organic solvents and to study the properties of elastomeric compositions in the presence of a renewed mineral component as a filler.

Materials and methods of the study

The subject under study was mineral products from the spent adsorbent of the vinification stage of refined sunflower oil, which were purified using a two-stage technology with organic solvents. At the first stage, a sunflower oil-based oil and fat fraction and hydrophobized filter powder were obtained from the spent filter powder by dichloroethane washing. At the second stage, the hydrophobized filter powder was used to produce a filter powder and a wax-containing product by washing with petroleum ether.

The products obtained as a result of washing with organic solvents were highly dispersed powders of light beige color. According to the X-ray fluorescence analysis, it was a mineral based on the elements of silicon (~70 wt.%), potassium (~15 wt.%), calcium (more than 5 wt.%), which should probably be attributed to diatomites of the kieselguhr type, which are widely used for filtering vegetable oils, fruit and vegetable juices, and in rubber production are used as non-toxic fillers or technological additives [15; 16].

In further studies of regeneration products from the waste of the sunflower oil vinification stage as potential fillers of elastomeric compositions, the following names were used

- product after washing with dichloroethane - “diatomite D”;
- product after washing with dichloroethane and petroleum ether - “diatomite P”.

The experimental products diatomite D and diatomite P were evaluated as fillers in the compositions of elastomeric compositions based on non-stereo-regular butadiene-α-methylstyrene rubber of low-temperature polymerization of Buna KER 1723 brand and compared with the control composition without filler and compositions with known mineral fillers kaolin and chalk at an equal weight content of 20.0 wt.% per 100.0 wt.% of the rubber base

Journal of Chemistry and Technologies, 2025, *33*(2), 427-435

(designated as phr), in the presence of a sulfur vulcanizing system. The rubber compounds were manufactured according to known methods on laboratory rollers per 300 g of rubber. Compared to the known mineral fillers, the combination of hydrophobized products under study with the rubber matrix was faster and better. Vulcanization of samples of elastomeric compositions for physical and mechanical tests was carried out in a hydraulic press at the optimum of vulcanization at a temperature of 155 °C.

The determination of the technological characteristics of rubber compounds and rubber properties was carried out following current standards and relevant methods [17–18]. In particular, according to the international standards DIN 53 529, the vulcanization properties and the reaction-kinetic evaluation of vulcanization isotherms were determined using a Monsanto 100 rheometer. The analytical support of the rheometer allowed us to obtain the following characteristics: ML – minimum torsional moment; MH - maximum torsional moment; TS1 – time of vulcanization start; TS90 – optimal vulcanization time (time of reaching 90 % crosslinking degree). Subsequently, we also calculated the time to achieve 25 %, 50 %, and 75 % crosslinking and the relative degree of crosslinking (ΔM = MH – ML), which were used to determine the conditional vulcanization rate constant (k_2) at temperatures of 155 °C and 165 °C, the effective activation energy of the vulcanization process (E), and other parameters [19].

The study of the elastic-strength characteristics of rubbers (stress-elongation curves) was carried out on the MultiTest-1 dV tensile and compression test bench of Mecmesin UK.

Differential thermal studies of mineral-containing products were performed on the Q-1500D derivatograph of the F system. Paulik, J. Paulik, L. Erdey, IOM, under conditions of uniform heating to a temperature of 1000 °C with a temperature rise rate (air) of 10 °C/min. The X-ray fluorescence spectroscopy of the studied products was carried out on the Expert 3L device.

Results and discussion

According to the data of differential thermal analysis (Fig. 1), the solvent-washed products were hydrophobized diatomite with an organic component content of 57 wt.% after the first stage of regeneration with dichloroethane, after the second stage of regeneration with petroleum ether – 37 wt.%.

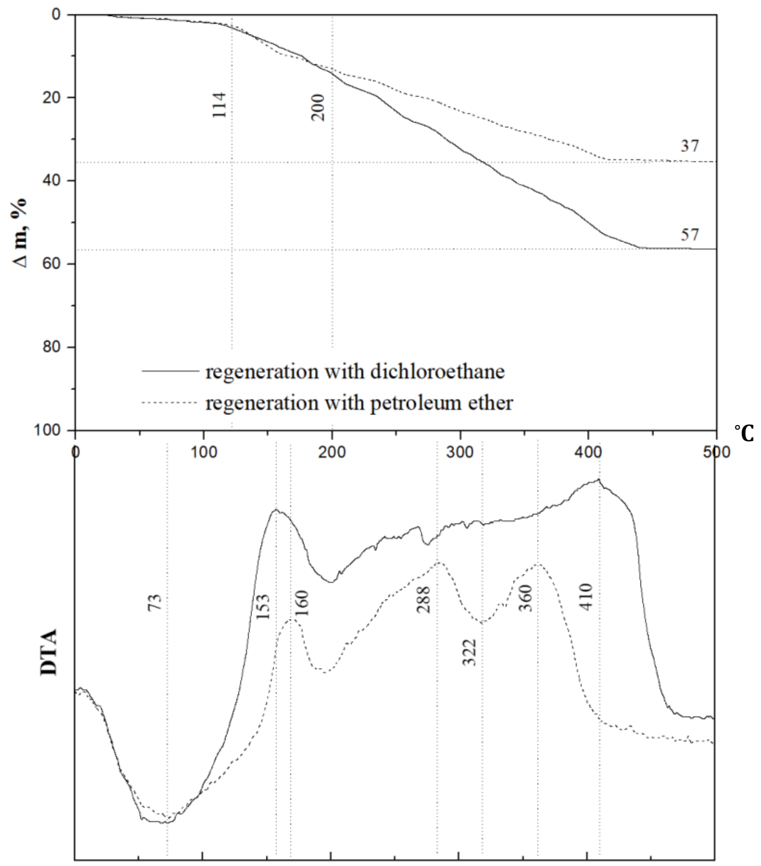

Fig. 1. Curves of differential thermal analysis of spent diatomite, sequentially regenerated with different solvents (medium - air, heating rate 10⁰C/min)

Journal of Chemistry and Technologies, 2025, *33*(2), 427-435

That is, at the first stage of waste regeneration, different fractions of hydrocarbons were washed. After the second stage of washing, diatomite, judging by the nature of the TG and DTA curves (Fig. 1), also remained hydrophobized by organic compounds, but more firmly held by the crystal matrix of the mineral. It should be noted that the initial temperature of mass loss (up to 1 wt.%) is 60 °C for both samples. In the temperature range of rubber compound processing, diatomite washed with dichloroethane loses more mass (1.7 wt.%) than diatomite with additional washing with petroleum ether (1.5 wt.%). Starting from 114 °C, a sharp mass loss occurs for both diatomite samples. The diatomite washed with dichloroethane alone loses weight faster. In the temperature range of vulcanization (max 200 °C), an organic fraction separately isolated from the studied products can enter the elastomeric composition, namely from diatomite washed with dichloroethane – 14.2 wt.% and 13 wt.% from diatomite washed with petroleum ether. The DTA curves of both samples have an identical pattern of changes in the exo- and endopikes, slightly shifted relative to each other, which indicates the influence of the organic component in the studied products on the course of reactions under the influence of temperature. The demonstrated effects on the DTA curves are more often related to changes in the composition of diatomite [9]. The first effects in the interval up to 200 °C (peaks of 153 °C and 160 °C) relate to the decomposition of opal minerals and the loss of molecular (physically bound) water by the product. The temperatures of the second effect (250–370 °C) are characterized by the loss of constitutional water. The endothermic effect at a temperature of 322 °C is characteristic only of diatomite washed with petroleum ether, which indicates chemical changes in the composition of the organic fraction of the sample under study. Further effects at higher temperatures are likely due to changes and destruction of the mineral's crystal lattice. That is, in the temperature ranges of processing and vulcanization of elastomeric compositions from regenerated diatomite samples, a certain amount of organic component can be desorbed and have an impact on the formation of the overall set of properties of elastomeric compositions.

Figure 2 shows the rheometric curves of sulfur vulcanization of elastomeric compositions based on butadiene-α-methylstyrene rubber of the Buna KER 1723 brand at temperatures of 155 °C and 165 °C with an equal content (20.0 phr) of known mineral fillers and sunflower oil production wastes regenerated in one stage with dichloroethane (diatomite D) and in two stages with dichloroethane and petroleum ether (diatomite P).

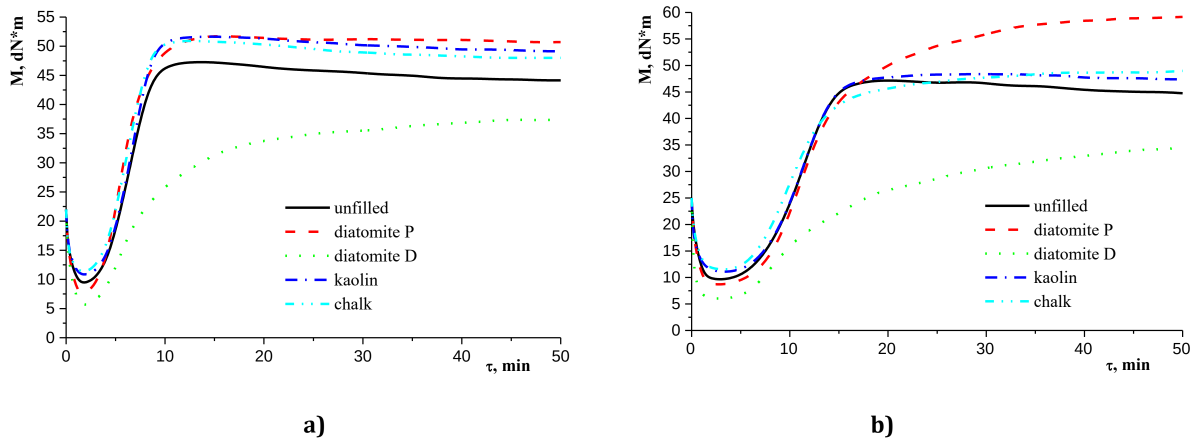

Fig. 2. Kinetic curves of sulfur vulcanization at 155^{0}C (a) and 165^{0}C (b) of elastomeric compositions in the presence of different type of mineral products

It is shown that, unlike kaolin and chalk, in the presence of which the vulcanization process follows close S-shaped kinetic curves (similar to the control rubber compound without filler), diatomite D and diatomite P studied in this work significantly affect the course of sulfur vulcanization at the stage of the induction period, the stage of active formation of vulcanization bonds, the plateau of vulcanization within a given temperature interval and the degree of formation of vulcanization cross-links. In particular, in the presence of diatomite D, the vulcanization process occurs with a constant increase in torque, which is significantly inferior to the torque of the control unfilled elastomeric composition and compositions with known mineral fillers. More positively, in relation to compositions with known fillers, the vulcanization process should be

Journal of Chemistry and Technologies, 2025, *33*(2), 427-435

characterized in the presence of diatomite P, which was additionally washed with petroleum ether to maximize the removal of the wax-containing organic component. It follows that the change in the course of sulfur vulcanization, the degree of crosslinking of elastomeric compositions based on a diene elastomer in the presence of the hydrophobized diatomites studied in this work depends on the wax-containing fraction of the organic component (unsaturated bonds in their structure), with which the actual vulcanization agents or their reactive components probably interact [20] even before the formation of transverse (vulcanization) cross-links.

The influence of regenerated mineral products on the level of the minimum torque parameters (ML) at the stage of the induction period of the vulcanization process is also significant in relation to the influence of known mineral fillers (Fig. 3).

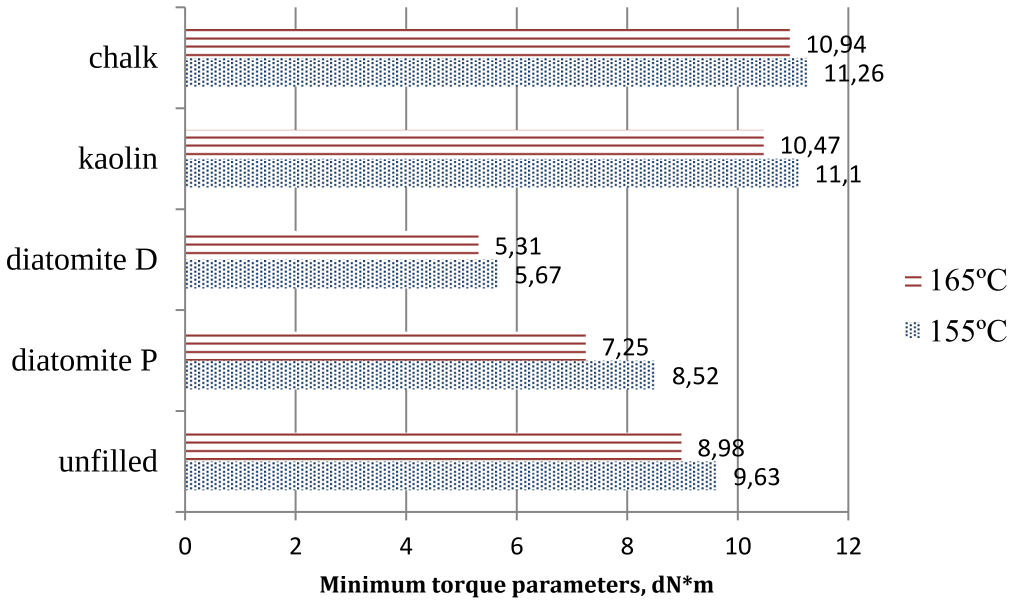

Fig. 3. Influence type of mineral products on the minimum torque parameters at vulcanization temperatures of 155 °C and 165 °C

The rheometry index ML characterizes the technological properties of rubber compounds, and with an increase in its value, the ability of rubber compounds to be processed on technological equipment deteriorates, and the processes become more energy-consuming. The data shown in Fig. 3 quantitatively characterize an increase in the level of the ML index by an average of up to 1.2 times when 20.0 phr of known fillers are introduced into an unfilled elastomeric composition and a decrease in the level of the ML index when an equal amount of diatomite P or diatomite D is introduced by up to 1.2 and 1.7 times, respectively. This fact of the influence of the studied hydrophobized diatomites on the minimum torque of rubber compounds can probably be attributed not only to the peculiarity of the adsorbent as a filler but also to the presence of an organic component in their composition – 37 wt.% in diatomite P, 57 wt.% in diatomite D (Fig. 1). According to their influence on the ML index, the products diatomite D and diatomite P should be considered as potential technological additives for elastomeric compositions based on diene rubbers.

The reaction and kinetic characteristics of the vulcanization isotherms of elastomeric compositions in the presence of 20.0 phr of mineral ingredients (Table 1), in general, quantitatively confirm the previously made conclusion about the significant effect of the studied regenerated products on slowing down the sulfur vulcanization process relative to the control composition without filler and compositions with known mineral fillers. The level of the values of the indicators of the time to reach the optimum of vulcanization (TS90), the vulcanization rate taking into account the increase in the torque in the main period (Vc), the conditional constant of the vulcanization rate (k'_2 and k''_2), calculated by two methods, of elastomeric compositions with diatomite P and diatomite D (especially at a temperature of 155 °C) is almost twice lower compared to the control composition without filler.

Table 1

Reaction and kinetic characteristics [21; 22] of vulcanization isotherms at temperatures of 155⁰C (numerator) and 165⁰C (denominator) of elastomeric compositions with different type of mineral products

Index	Type of mineral products				
	unfilled	diatomite P	diatomite D	kaolin	chalk
TS1, min	$\frac{6.0}{3.7}$	$\frac{6.3}{3.3}$	$\frac{6.4}{4.0}$	$\frac{6.7}{4.0}$	$\frac{6.0}{3.9}$
TC90, min	$\frac{13.4}{7.9}$	$\frac{23.1}{8.9}$	$\frac{26.8}{10.3}$	$\frac{13.8}{8.0}$	$\frac{12.6}{7.7}$
$Vc = \frac{MTC90 - MTS1}{TC90 - TS1}$, $\mathrm{dN * m/min}$	$\frac{4.01}{7.83}$	$\frac{2.59}{6.82}$	$\frac{1.19}{4.42}$	$\frac{4.50}{8.98}$	$\frac{4.58}{9.12}$
$k_2' = \frac{1{,}608}{T90 - T50}$, $\mathrm{min^{-1}}$	$\frac{0.67}{1.14}$	$\frac{0.15}{0.54}$	$\frac{0.11}{0.65}$	$\frac{0.48}{1.23}$	$\frac{0.70}{1.33}$
$k_2'' = \frac{1{,}1}{T75 - T25}$, $\mathrm{min^{-1}}$	$\frac{0.31}{0.56}$	$\frac{0.17}{0.51}$	$\frac{0.08}{0.13}$	$\frac{0.26}{0.61}$	$\frac{0.33}{0.64}$
$E_1 = \frac{4{,}576T1T2}{T2 - T1} \log\frac{k_2''}{k_2'}$, $\frac{\mathrm{kJ}}{\mathrm{mole}}$	91.73	175.29	73.02	131.05	102.06
$E_2 = 19{,}1\,\frac{\log k_2'' - \log k_2'}{\frac{1}{T_1} - \frac{1}{T_2}}$, $\frac{\mathrm{kJ}}{\mathrm{mole}}$	91.51	174.87	72.85	130.73	101.82

This trend is not characteristic of the parameter of the duration of the induction period of vulcanization TS1 – in the presence of the studied mineral regenerated products, the level of TS1 remains close to the control composition (Table 1).

Since the activation energy (E_1, E_2) is a parameter characteristic of chemical reactions, which determines the energy consumption for activating the reaction, and thus the dependence of the rate parameter on temperature, it is quite logical to have a twice higher level of effective activation energy for compositions with diatomite P compared to control compositions in the absence of filler and 1.3–1.7 times higher compared to known mineral fillers (Table 1). In contrast to diatomite P, the introduction of diatomite D into the elastomeric composition is accompanied by a decrease in the level of the effective activation energy by an average of 2.4 times and is less than that of compositions with kaolin or chalk. Diatomite P contains 37 wt.% organic component, while diatomite D contains 57 wt.%. The organic component in diatomite P is more strongly bound to the crystal matrix of the mineral after two-stage regeneration (dichloroethane and petroleum ether). This complicates its desorption during vulcanization, which requires more energy to activate the process.

Rheometric studies can also be used to separate the effect of rubber-filler interaction and the effect of cross-linking [23]. According to the kinetic curves of sulfuric vulcanization of elastomeric compositions (Fig. 2), the influence of the studied mineral products diatomite P and diatomite D on the relative degree of vulcanization (ΔM) of rubbers and the difference in the relative degree of crosslinking of the filled (ΔM_{filled}) – the effect due to crosslinking by the vulcanizing system and the rubber-filler interaction; and unfilled ($\Delta M_{unfilled}$) – the effect of crosslinking due to the vulcanizing system of the elastomeric composition (Table 2). These data indicate a significant positive effect of diatomite P and a negative effect of diatomite D on the indicator of the relative degree of crosslinking of elastomeric compositions in the studied temperature range relative to the indicators of rubbers with kaolin and chalk. As a filler, diatomite P on the vulcanization plateau has the maximum degree of rubber-filler interaction.

Table 2

Influence of the type of mineral products on the relative degree of vulcanization (ΔM=MH – ML) of elastomeric compositions

Type of mineral products	Index			
	ΔM, dN*m	$\Delta M_{filled} - \Delta M_{unfilled}$, dN*m	ΔM, dN*m	$\Delta M_{filled} - \Delta M_{unfilled}$, dN*m
	Vulcanization temperature			
	155 °C		165 °C	
unfilled	37.71		38.49	
diatomite P	50.68	12.97	45.15	6.66
diatomite D	29.33	–8.38	32.89	–5.60
kaolin	37.50	–0.21	41.86	3.37
chalk	35.67	–2.04	40.35	1.86

Journal of Chemistry and Technologies, 2025, *33*(2), 427-435

That is, in terms of their influence on the rheokinetic characteristics of sulfuric vulcanization of elastomeric compositions based on carbocarbon polydiene, the studied diatomite P and diatomite D are quite reactive ingredients and this should be taken into account when using them in industrial elastomeric compositions with regulated parameters.

The obtained curves in the "stress-elongation" coordinates of the vulcanizates (Fig. 4) indicate the influence of the studied mineral products as fillers on the characteristics of rubbers under normal test conditions. In particular, there is a connection between the rheometric parameter relative degree of crosslinking of rubbers at a temperature of 155 °C (Fig. 2, Table 2) and the nature of the stress-elongation curves of rubbers vulcanized at the optimum temperature of 155 °C.

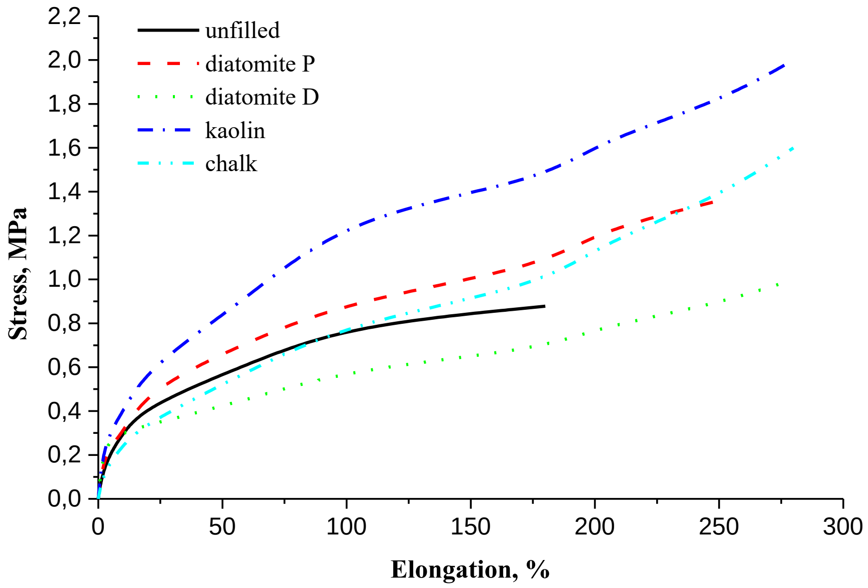

Fig. 4. Stress-elongation relationship of rubbers with different type of mineral products

The strengthening of rubbers in the presence of diatomite P relative to rubbers with diatomite D should probably be considered as a consequence of the formation of a more optimal concentration of vulcanization bonds (provides a better balance between the number and quality of bonds to form a homogeneous structure of the rubber matrix) in the absence of a negative effect of a high degree of hydrophobization (organic component of the waste) of the mineral on the formation of rubber-filler bonds [24; 25].

The results of physical and mechanical tests of rubbers under normal conditions (Table 3) also indicate certain features of the reinforcing effect of regenerated mineral products compared to control rubber without filler and rubbers with an equal mass dosage of kaolin or chalk.

Table 3

Physical and mechanical properties of rubbers with different type of mineral products

Index	Type of mineral products				
	unfilled	diatomite P	diatomite D	kaolin	chalk
Nominal stress at 300 % elongation, f_{300}, MPa	1.3	1.6	1.3	1.9	1.3
Tensile strength, f_p, MPa	1.5	2.0	1.9	2.9	2.0
Elongation at break, ε, %	380	480	550	520	460
Shore A hardness, conv. unit	40	45	43	45	44
Elasticity, %	49	49	46	53	52
Reinforcement coefficient*, $I = \frac{f_{300} \times f_p \times \varepsilon}{f^0_{300} \times f^0_p \times \varepsilon^0}$		2.07	1.83	3.87	1.61

Note: * - for the calculation, the values of the physical and mechanical parameters of rubbers with mineral products were used in the numerator, and without mineral products (rubber without filler) in the denominator [24; 26].

Compared to rubber without filler, the reinforcing effect of mineral products is confirmed by the levels of strength characteristics, tensile elongation, and hardness. The reinforcement effect was evaluated using the reinforcement coefficient, which summarized the effect of mineral products on the elastic strength characteristics of rubbers (Table 3). It was found that the following series can be constructed according to the reinforcement factor: kaolin > diatomite P > diatomite D > chalk. Hence, the mineral regenerated waste of sunflower oil production diatomite P with 37 wt.% organic component as a filler of elastomeric compositions is superior to diatomite D with 57 wt.% organic component and chalk, but inferior to kaolin.

Conclusions

Given the significant potential of Ukrainian sunflower oil production enterprises, the issue of rational processing of multi-tonnage secondary resources is relevant. This will contribute to the creation of competitive domestic products based on waste from the technological processes of oil and fat enterprises, reduce the technogenic load on the environment, and use the resulting materials as ingredients in elastomeric compositions. Therefore, this paper investigates the peculiarities of the properties of elastomeric compositions based on carbocarbon polydiene containing used filter powder regenerated with organic solvents as a filler after the vinification stage of sunflower oil production. The subject of the study was two types of diatomite powders regenerated in stages according to the new method: diatomite D washed in one stage with dichloroethane and diatomite P washed in two stages with dichloroethane and petroleum ether.

The peculiarities of elastomeric compositions based on synthetic non-stereoregular butadiene-α-methylstyrene rubber of the Buna KER 1723 brand in the presence of 20.0 phr of regenerated diatomite powders in comparison with the equal mass content of mineral fillers kaolin and chalk have been determined. It has been shown that the hydrophobized products under study have a positive effect on the processability of rubber compounds and can be recommended as technological additives, as well as have an impact on the course of sulfur vulcanization, the level of rheokinetic characteristics, and the crosslinking density of elastomeric compositions. It was found that diatomite P with 37 wt.% of the organic component is dominated by diatomite D with 57 wt.% organic component and chalk, but inferior to kaolin. Diatomite P can be recommended for use in the formulations of industrial elastomeric compositions.

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