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SPECIAL SECTION
Machine modelling and simulations

Targeted modification of the composition of polymer systems for industrial applications

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Abstract. The targeted modification of the material composition is a common procedure used to improve the parameters of the final products. This paper deals with the targeted modification of polymer systems composition using two various types of alternative fillers. The first type of alternative filler (SVD) has been obtained from energetics where it arises as a by-product of flue gas desulfurization. The second alternative filler used (KAL) is based on waste from glass production. The elastomeric systems designed for the production of car tires and solid wheels for transport systems were used in the role of modified polymer systems. Alternative fillers (SVD, KAL) have been applied as a substitution of commonly used fillers (carbon black, silica). The filler – elastomeric matrix interaction, rheology, cure characteristics, as well as hardness and rebound resilience of vulcanizates, which are important parameters for their industrial application, have been studied in the new prepared polymeric systems. The main output of the work is a new formulation of an elastomeric system for industrial applications with high rebound resilience and low rolling resistance, which is the subject of the international patent [1]. The modification of composition using raw material substitution can also bring significant environmental and economic effects.

Key words: polymer elastomeric system; industrial application; rheology; cure characteristics; hardness; rebound resilience.

1. Introduction

From the discovery of natural rubber and the process of vulcanization to the present time, rubber technology has undergone intensive development. The current rubber compounds and their vulcanizates are characterized by several exceptional quality parameters that cannot be achieved with other types of materials. Their important properties include, e.g. strength, elongation, elasticity, rebound resilience, or corrosion resistance. An indisputable advantage of the elastomer systems is also the possibility to purposefully change and adjust their properties depending on the desired application. By selecting an appropriate type of rubber, the type and amount of fillers, as well as by setting the necessary conditions of the preparation process, it is possible to obtain a final product with properties suitable for a particular preselected application [2, 3]. The aim is to achieve a suitable combination of the required rheological and physical-mechanical properties of rubber compounds and vulcanizates in order to ensure the correct function and the necessary operating life of a particular product in connection with its application. The current development of polymeric systems is intensively focused on the application of various types of alternative fillers, especially in connection with the greening and economization of the production of various polymeric components [4–6]. The emphasis is placed on the possibility of using wastes from other industries as alternative fillers, in order to obtain an elastomeric material with specific properties. The addition of an alternative filler to the rubber compound results in a completely new type of composite elastomeric material, in which an improvement in several important quality parameters can be observed simultaneously. The resulting products containing alternative fillers show, e.g. a positive increase in the values of rebound resilience and a decrease in the values of rolling resistance, while maintaining the optimal values of hardness [6–8].

The continuous improvement of the final product quality with the simultaneous elimination of waste from the technological process and the reduction of production costs are in the best interests of producers [9]. Such a suitable combination of specific properties is usually difficult to achieve using only conventional types of fillers such as carbon black or silica. A specific combination of quality parameters can be achieved using the new types of alternative additives based on selected kinds of waste from industrial productions. Especially by application procedures of suitable inorganic materials into organic polymers, a significant improvement of physical and mechanical properties of elastomers can be achieved, specifically of hardness, tensile straight, modulus, but also rebound resilience and rolling resistance, which are very important parameters for industrial applications [10]. Composite materials with polymeric matrix prepared this way show generally better properties than pure and homogeneous materials. The positive changes have been observed at low concentrations of the inorganic components in polymers [10, 11].

This work is focused on the development and study of new types of elastomeric systems using alternative fillers based on selected industrial wastes, in order to achieve the required

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properties of the final products for their specific industrial applications. For the targeted modification of polymer systems composition, two kinds of alternative fillers based on different industrial wastes were used. Alternative filler (SVD) arises as a by-product in the flue gas desulphurization process in energetics and its chemical composition is very similar to gypsum composition ($CaSO_4 \cdot 2H_2O$). The other type of alternative additive (KAL) arises in a dust form in the process of weighing and mixing glass raw materials in glass production, which is then captured in a mechanical separator in the form of sludge. The chemical composition of this alternative filler is similar to the chemical composition of kaolin or silica that are commonly used in the industrial production of polymer composites. Alternative fillers SVD and KAL were applied into the polymeric elastomeric systems as a replacement for commonly used fillers – carbon black and silica. The aim of this targeted modification of the composition is the preparation of elastomer mixtures with specific properties for specific industrial applications – the production of solid wheels for transport systems and the production of tires. The main emphasis is, therefore, on improving the values of rebound resilience, rolling resistance, and hardness of resulting vulcanizates [1, 2]. The work also deals with the investigation of the interaction of alternative SVD and KAL fillers with the polymer matrix and their influence on the rheology and cure characteristics of the prepared elastomeric systems.

2. Experiments

All experiments were performed in specialized laboratories (CEDITEK) at the Faculty of Industrial Technologies in Púchov.

2.1. Study of the composition of alternative fillers. Energy-dispersive X-ray spectroscopy – EDX analysis for determination of the chemical composition of alternative fillers SVD and KAL was used, which is important for the description of processes in the incorporation of alternative fillers into elastomer mixture and the interaction between the elastomeric matrix and the filler. Analyses were performed using the EDP-7000 fluorescence spectrometer. Results of EDX analysis of alternative fillers SVD and KAL are given in Table 2.

2.2. Preparation of elastomer systems with alternative fillers. Alternative fillers studied (SVD, KAL) were applied into polymer systems in combination with commonly used fillers – carbon black (CB) and silica. Eight elastomeric systems with the addition of alternative fillers have been prepared. As an elastomeric matrix, the real rubber compound for the tyre production based on natural rubber grade SMR 10 has been used. Eight elastomeric systems with a total filler content of 77 phr were prepared using two steps mixing at the temperature of 90°C using the Plastograph Bradender laboratory mixer. The fillers SVD and KAL/silica were added to the polymer matrix in combination with carbon black, as the partial replacement of this common filler. 180 seconds were used for mixing the fillers to ensure the sufficient incorporation of the fillers in the polymer matrix [12]. Additional homogenization was carried out by the laboratory twin-roll machine (Calander) – LaboWalz W 80 T (Manufacturer: VOGT) for each blend to increase the homogeneity of the blends after each mixing step. After the second step of mixing, the blends were left at a laboratory temperature of 25 ± 2°C for 24 hours [13]. The composition and labeling of the prepared elastomeric systems are given in Table 1.

Table 1
Content of fillers and labeling of prepared elastomeric systems

System →	**K1**	**K2**	**K3**	**K4**	**S1**	**S2**	**S3**	**S4**
Filler ↓	Content (phr)							
CB	30.0	30.0	30.0	30.0	30.0	30.0	30.0	30.0
SVD	45.0	43.0	41.0	39.0	45.0	43.0	41.0	39.0
KAL	2.0	4.0	6.0	8.0	–	–	–	–
Silica	–	–	–	–	2.0	4.0	6.0	8.0

2.3. Study of rheology and cure characteristics. Rheological properties and cure characteristics of elastomeric systems prepared with the content of alternative fillers SVD and KAL have been determined using the Rubber Process Analyzer RPA 2000. The samples were prepared by cutting to prepare the required shape of samples from the obtained rubber sheets using an instrument Cutter 2000 (Alpha Technologies). For the test, the (vulcanization) temperature of 160°C with a frequency of 1.66 Hz at 60 minutes and a 5% oscillation angle was used. The temperature of 160°C was used in connection with the industrial application of the prepared polymeric systems with SVD and KAL fillers, with an emphasis on economic and time aspects of the curing process. Rheology involves the study of the deformation and mass flow [14]. The aim is to establish the relationships between the stress and deformation of the materials, where neither Newton's Law nor Hooke's Law is enough to explain their mechanical behaviour [15]. The investigated characteristics include minimum and maximum torque (M_L, M_H), scorch time (t_{S02}), and optimal time of cure (t_{C90}). The obtained results are given in Table 3 and graphically evaluated in Figs. 1 and 2.

2.4. The determination of hardness. The hardness of elastomeric vulcanizates containing the alternative fillers was determined using a Shore A hardness tester. The samples were measured according to ASTM [16]. The measured values of the hardness of the vulcanizates studied are summarized in Table 4 and graphically processed in Fig. 3.

2.5. Determination of rebound resilience. The measurement of the rebound resilience of vulcanizates is in most cases performed by the impact of a pendulum with a spherical projection on the surface of the elastomeric vulcanizate sample and by calculating the relative kinetic energy (R) which was needed to reach the height designated as h_2, while the initial or original height was designated as h_1 (1) [17]:

$$R = \frac{h_2}{h_1}. \tag{1}$$

The energy supplied to the impactor in the form of mechanical vibration caused by the impact should not be attributed to the energy losses in the sample. The determination of rebound resilience of the prepared vulcanizates was performed by the Polymertest apparatus with a digital indicator of rebound resilience (%). The obtained results are given in Table 5 and graphically evaluated in Fig. 4.

2.6. Microstructural SEM analysis. The microstructure, morphology, and the interaction of the fillers with the elastomeric matrix were observed on the fracture surfaces of the samples of elastomeric vulcanizates after the tensile tests. A TESCAN VEGA 3 electron microscope was used for the microstructural analysis. All samples of elastomeric vulcanizates were plated using the SC7620 Mini Spuer Coater to observe the morphology and to ensure the sample conductivity. The microstructure of the fracture surfaces of the elastomeric systems vulcanizates is shown in Figs. 5 and 6.

3. Results and discussion

This part summarizes the results from EDX analysis of the used alternative fillers (SVD, KAL), rheological properties, and cure characteristics, rebound resilience, and hardness of prepared elastomeric systems with the addition of alternative fillers SVD and KAL.

3.1. Results of EDX analysis – composition of alternative fillers. The chemical compositions determined using the EDX analysis of alternative fillers SVD and KAL used for targeted modification of polymeric systems are given in Table 2.

Table 2
Composition of alternative fillers SVD and KAL

SVD		KAL	
Element	Weight %	Element	Weight %
Ca	68.64	Si	54.93
S	29.55	Ca	18.22
Si	1.16	Sb	8.72
P	0.26	Ba	8.55
Fe	0.21	Zn	3.11
Sr	0.05	Al	2.96
Cu	0.04	K	1.49
K	0.09	S	0.73

The results show that the chemical composition of the alternative filler SVD is very similar to gypsum composition ($CaSO_4 \cdot 2H_2O$) and the chemical composition of the alternative filler KAL is similar to the chemical composition of a mineral filler kaolin which is used in the industrial production of polymer composites. Also, silicon, commonly used in the production of car tires, is the main element contained in the KAL filler, which is also the majority element of the active light silica filler. The obtained results presuppose the successful application of the studied waste products as alternative fillers in polymer systems.

3.2. Rheological properties and curing characteristics of modified elastomeric systems. The measured values of rheological properties and curing characteristics for eight modified polymeric systems prepared with the addition of the alternative fillers SVD and KAL as a partial substitution of common fillers (carbon black, silica are summarized in Table 3, and they are graphically processed in Figs. 1 and 2. The studied rheological properties of polymer elastomeric systems were Minimum (M_L) and Maximum torque (M_H) and curing characteristics studied were the Scorch time (t_{s02}) and Optimal cure time (t_{c90}).

Table 3
Rheological properties and curing characteristics of modified polymer systems

System	M_L (dNm)	M_H (dNm)	t_{s02} (min)	t_{c90} (min)
K1	3.01	32.04	1.47	3.16
K2	3.00	31.90	1.48	3.13
K3	2.98	30.31	1.51	3.10
K4	3.04	30.57	1.46	2.99
S1	3.08	30.85	1.55	3.13
S2	3.33	30.39	1.63	3.17
S3	3.53	30.36	1.69	3.26
S4	3.89	30.72	1.76	3.34

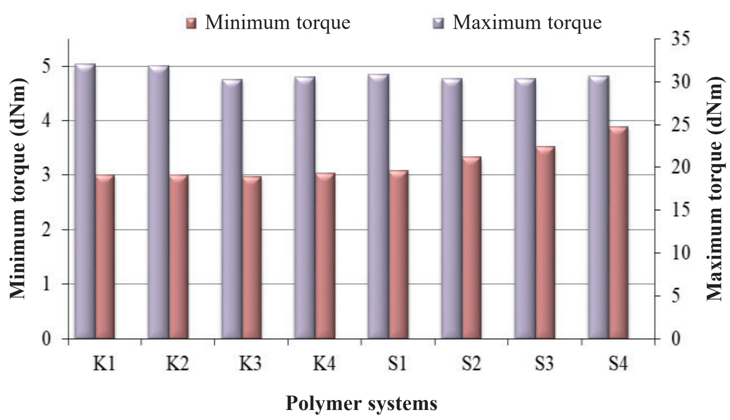

Fig. 1. Values of Minimum (M_L) and Maximum torque (M_H) of polymer systems

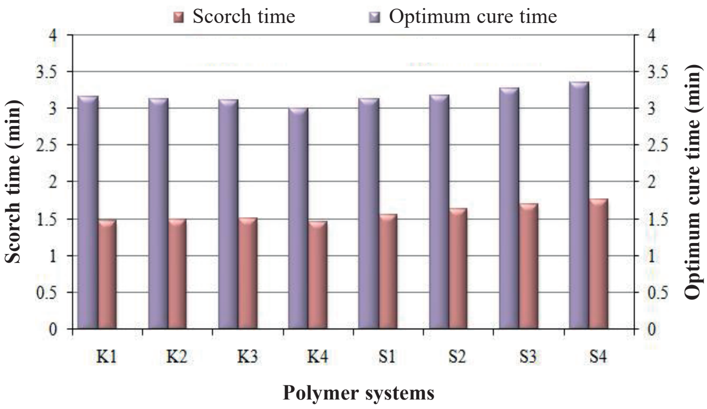

Fig. 2. Values of Scorch time (t_{s02}) and Optimum cure time (t_{c90}) of polymer systems

The value of minimum torque represents the stiffness of the non vulcanized tested sample taken at the lowest point of the vulcanization curve. The maximum torque represents the stiffness or shear modulus of the fully vulcanized tested sample at the vulcanization temperature [18].

From the measured values of the minimum and maximum torque (Fig. 1), it is possible to see the effect of the presence of a combination of alternative fillers SVD and KAL that partially replaced the carbon black filler (systems K1–K4), in comparison with the effect of the used combination of an alternative filler SVD with silica (systems S1–S4). In the case of elastomeric systems containing the combination of alternative fillers SVD + KAL, a decrease in the minimum torque values and increase in the maximum torque values can be observed compared to the parameters of the systems containing the combination of the SVD + silica fillers. An increase in the minimum torque values in the case of the silica-containing systems is evidence of a higher stiffening effect and thus a greater silica activity at the beginning of the vulcanization process. The higher values of the maximum torque in the case of the systems containing the combination of SVD + KAL indicate the activity and reinforcing effect of the alternative SVD filler, for which its active involvement in the vulcanization process has been proven.

The curing characteristics studied include the Scorch time (t_{s02}) and Optimum cure time (t_{c90}) [18]. The Scorch time is the time required at a specified temperature (or heat history) for a rubber blend to form incipient crosslinks. When a scorch point is reached after a blend is exposed to a given heat history from the factory processing, the blend is not able to be processed [19]. The Optimum cure time (t_{90}) is the time required for the torque to reach 90% of the maximum achievable torque and relates to the time which is necessary for the cured rubber to achieve optimal properties [20].

The graphical comparison of the results in Fig. 2 shows that most polymer elastomeric systems K1–K4 containing the combination of alternative fillers SVD + KAL showed lower values of the Scorch time (t_{s02}) and Optimum cure time (t_{c90}) compared to the parameters of systems S1–S4 containing the combination SVD + silica. From the economic point of view, a decrease in the Scorch time and Optimal cure time is a positive phenomenon because the time required for the production of the product decreases which leads to a reduction in the cost of the vulcanization process and thus in the entire production costs.

3.3. The hardness of modified elastomeric systems. The measured values of the hardness of the vulcanizates studied with the addition of alternative fillers SVD and KAL are summarized in Table 4 and graphically represented in Fig. 3. In the case of polymer systems S1-S4 containing a combination of the alternative filler SVD + silica, the higher hardness values are observed in comparison with the systems K1–K4, which contain a combination of alternative fillers SVD + KAL. The increase in hardness values in the case of silica-containing systems is related to the significant stiffening effect of silica as an active stiffening filler commonly used in rubber production.

Table 4
The hardness of modified polymer systems

System	Hardness (ShA)
K1	61.77 ± 0.34
K2	61.38 ± 0.18
K3	60.67 ± 0.14
K4	61.40 ± 0.28
S1	62.88 ± 0.33
S2	62.78 ± 0.50
S3	63.07 ± 0.38
S4	64.12 ± 0.28

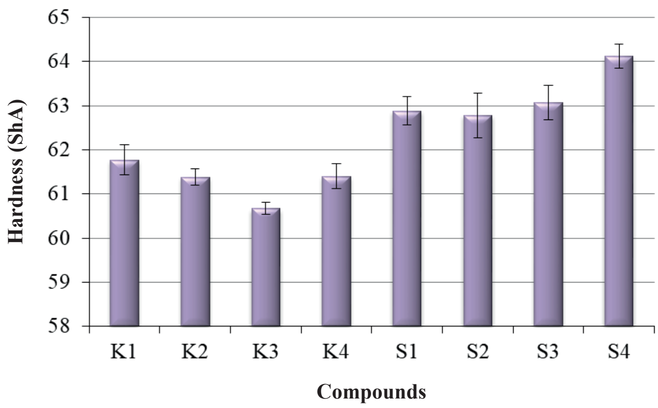

Fig. 3. Values of the hardness of modified polymer systems

In the series of elastomeric systems with silica content, the highest hardness value (64.12 ShA) was displayed by the elastomeric system S4 with the highest silica content, while in the series of elastomeric systems containing the alternative filler KAL, the highest hardness (61.77 ShA) was achieved by system K1 with the lowest addition of KAL filler. The given results confirm that the alternative filler KAL itself does not show a reinforcing effect and can be classified as inactive filler.

3.4. Rebound resilience of modified elastomeric systems. Rebound resilience is the most important parameter in terms of industrial application of the developed new elastomeric systems in the production of solid wheels and car tires. Resilience is a very important parameter in the production of wheels because it directly affects the value of rolling resistance, the reduction of which was demanded by an industrial partner [1, 21]. The measured values of the rebound resilience of the vulcanizates studied with the addition of alternative fillers SVD and KAL are summarized in Table 5 and graphically represented in Fig. 4.

Table 5
Rebound resilience of the modified polymer systems

System	Rebound resilience (%)
K1	58.71 ± 0.05
K2	58.39 ± 0.24
K3	58.14 ± 0.21
K4	58.39 ± 0.39
S1	57.73 ± 0.05
S2	58.00 ± 0.05
S3	56.22 ± 0.32
S4	57.11 ± 0.07

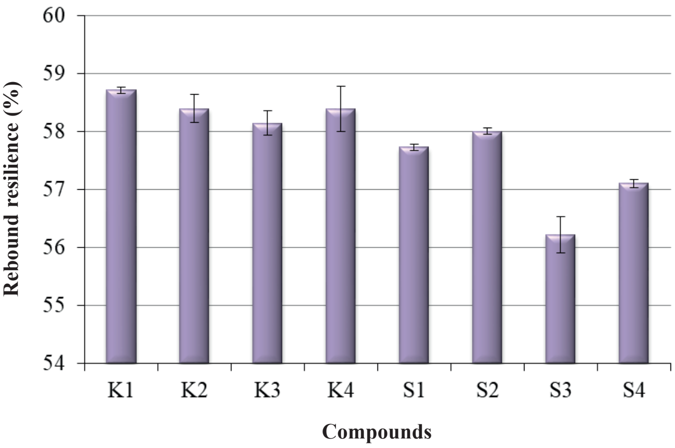

Fig. 4. Values of rebound resilience of the modified polymer systems

The results of the rebound resilience measurement show that elastomeric systems K1–K4 containing a combination of alternative fillers SVD + KAL show a positive increase in the values of rebound resilience in comparison with the parameters of systems containing silica filler. In accordance with a defined lowest hardness value, the highest value of rebound resilience (58.71 %) was achieved by system K1 with the lowest addition of KAL filler and the highest content of SVD filler. Increasing the values of rebound resilience has a direct effect on a significant decrease in the values of rolling resistance, which is a positive result for specific industrial applications of prepared modified polymer systems in the production of solid wheels for transport systems in industry and in the production of car tires. It is true that as the values of rebound resilience increase, the rolling resistance decreases significantly, which results in positive fuel savings during vehicle operation and also a reduction in harmful exhaust gases.

3.5. SEM analysis of fracture surfaces of modified elastomeric systems. The microstructure and morphology of fracture surfaces of samples of vulcanizates of modified elastomeric systems containing alternative fillers SVD and KAL were monitored in order to study the mutual interaction of filler particles with the elastomeric matrix. The results of the SEM microscopic analysis are shown in Figs. 5 and 6.

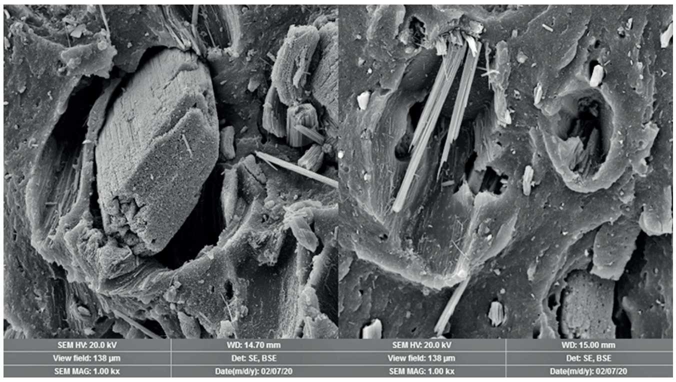

Fig. 5. Detailed images of the fracture surface of the modified polymer system containing the alternative filler SVD at 1000× magnification

On the SEM images, it is possible to observe in detail the holes in the elastomeric matrix which have formed after the SVD alternative filler particle has been torn out. From a closer look, it is possible to see the rest of the SVD alternative filler particle directly in the hole in the matrix. These images are proof of the correctness of the theory of the involvement of the alternative filler SVD in the vulcanization process, and thus confirm the very interaction of the particles of the alternative filler SVD with the matrix of natural rubber.

A closer look at the detailed SEM images in Fig. 6 shows a cavity formed by tearing out a filler particle under the tensile stress of a sample of an elastomer system vulcanizate. It can be

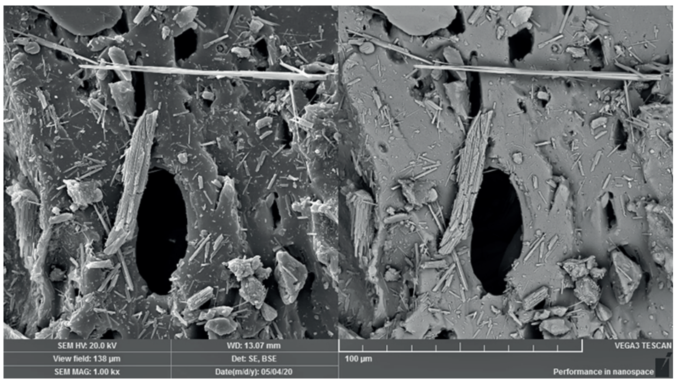

Fig. 6. Detailed images of the fracture surface of modified polymer system containing the alternative filler KAL at 1000× magnification

assumed that the extracted particle belonged to the alternative filler KAL. The alternative filler KAL is only mechanically mixed into the matrix and, unlike the alternative filler SVD, it is not bound to the elastomeric matrix.

4. Conclusions

The use of two kinds of alternative waste-based alternative fillers SVD and KAL coming from the industry for the targeted modification of polymer elastomeric systems has been presented in the paper. The newly developed polymer systems with specific properties are intended for industrial applications in the production of solid wheels for transport systems and in the production of car tires.

The chemical composition of the alternative fillers SVD and KAL has been studied using the EDX analysis which shows that the chemical composition of the alternative filler SVD is very similar to the gypsum composition $(CaSO_4 \cdot 2H_2O)$ and the chemical composition of alternative filler KAL is similar to the chemical composition of the silica filler or mineral filler kaolin, which are commonly used in the industrial production of polymer composites. Eight elastomeric systems with a total filler content of 77 phr were prepared, in which the majority of the common carbon black filler has been replaced by a combination of the alternative fillers SVD + KAL (K1–K4) or SVD + silica (S1–S4). The obtained results of the study of the modified elastomeric systems show the involvement of the alternative filler SVD in the vulcanization process, and thus the very interaction of the particles of the alternative filler SVD with the matrix of natural rubber. Polymer elastomeric systems with the combination of the alternative fillers SVD + KAL showed a positive decrease in the values of the Scorch time (t_{s02}) and Optimum cure time (t_{c90}) and a positive increase in the values of rebound resilience. Increasing the values of rebound resilience has a direct effect on a significant decrease in the values of rolling resistance. This is a positive result in specific industrial applications of the prepared modified polymer systems because an increase in the values of rebound resilience causes a significant reduction in the rolling resistance, which results in positive fuel savings during the vehicle operation.

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