

# THE EFFECT OF ALTERNATIVE FILLERS BASED ON POLYPROPYLENE WASTE FROM FIBERS AND FOILS PRODUCTION ON PROPERTIES OF RUBBER COMPOUND

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**Abstract:** The submitted study represents the research of alternative fillers based on polypropylene (PP) waste from the production of PP fibres and PP foils and its application in the polymeric materials applied in rubber industry. Properties of alternative fillers based on PP fibers and PP powder were studied by spectral and thermal analysis. The study is aimed at the use of polypropylene fibres and polypropylene powder as fillers in rubber blend as a partial replacement of carbon black. The influence of amount and the type of used alternative filler in rubber blend was evaluated. Curing characteristics of prepared tread blends were studied. Physical-mechanical properties before and after accelerated thermal aging in air and dynamical mechanical properties of prepared vulcanizates were determined and studied. The evaluation of prepared tread blends respectively tread vulcanizates results the possibility of partial replacement of common used carbon black N339 by studied PP fibres and PP powder.

**Keywords:** polypropylene, alternative fillers, curing characteristics, physical-mechanical properties, dynamical and mechanical analysis.

## 1 INTRODUCTION

Rubber blends consist of various additives whose representation can be up to 80% of the final blend. The development of industrial polymer production is progressing very fast and it is focused mainly to improve the properties of polymeric materials or the combination of different types of polymers. The combination of polymeric materials and addition of various ingredients into a rubber blend is very important to achieve specific performance [1-5].

Additives, mainly fillers significantly affect the physical and mechanical properties of the final product. Fillers are usually solid substances in form of a powder or short fibers. The improvement of the resulting vulcanizates properties is called stiffening [6-12].

Fillers are added to the rubber blend in order to adjust processing properties of the blend, to decrease elasticity, improve physical and mechanical properties, adhesion or aging resistance. On the other hand, fillers help to decrease the cost of the final product [6, 7, 13, 14]. Stiffening effect is formed due to the formation of the physical and chemical bonds between the filler and rubber, that is why the stiffening degree depends on a particle size, specific surface area or porosity and others [1, 4, 8, 15, 16].

For the development of automotive industry is also highly perspective the use of renewable sources of raw materials and the use of products such as starch, cellulose or lactic acid. The suitable renewable products are microbial polymeric materials e.g. polylactide fibers due to their biodegradation and other materials e.g. polypropylene powder respectively polypropylene fibers or alternative clay minerals and silica [13-18].

A polypropylene chain is formed by the carbon atoms, while every second atom is connected to the functional methyl group. According to the position of the functional methyl group is polypropylene PP divided into three types: isotactic, syndiotactic and atactic. Wide range of applications and unique polypropylene properties such as low density, high chemical resistance or resistance to heat or oil and high crystallinity makes PP suitable for application in rubber industry as the alternative filler [12-21].

This work is aimed at the evaluation of curing properties of prepared tread blends, physico-mechanical properties before and after acceleration thermal aging and dynamic mechanical properties of vulcanizates with a certain content of alternative filler based on polypropylene waste from fibers and foils production, such as polypropylene powder and polypropylene fibers and its impact in automotive industry.

## 2 EXPERIMENTAL

### 2.1 Materials

Various types of fillers were examined, namely polypropylene waste from polypropylene fibers or foils production. Short polypropylene fibers (Figure 1) and polypropylene powder (Figure 2) were used instead of commonly used active filler – carbon black (N339) in rubber industry. Polypropylene fibers were used from isotactic polypropylene from the production of textile fibers. The length of the polypropylene fibers was from 0.1 - 100 mm. Polypropylene powder is a waste from the production of polypropylene foils that have been made from hardened polypropylene. The particle size of the polypropylene powder was from 20 to 200  $\mu\text{m}$ .

Alternative fillers based on polypropylene fibers (abbreviated in this work as PV) or polypropylene powder (abbreviated in this work as PP), partially replaced the filler carbon black N339 (87 phr - *parts per hundreds of rubber*) in the amount of 1 phr (PP-1, PV-1), 5 phr (PP-5, PV-5) or 10 phr (PP-10) in the preparation of rubber compounds intended for treads.

### 2.2 Methods and preparation of rubber composites

Evaluation methods of thermal and spectral properties of alternative polypropylene fillers include differential scanning calorimetry (DSC) and infrared spectroscopy (FTIR).

DSC method was performed on a Perkin Elmer DIAMONT DSC instrument. Heating program was started at an initial temperature of 30°C and increased at a constant rate of 10°C/min to a final temperature of 200°C.

Crystalline content was calculated by formula (1), where  $K_p$  is crystalline content [%],  $\Delta H^+$  is enthalpy of melting,  $\Delta H$  (198.11 J/g) is enthalpy of melting of a completely crystalline polymer.

$$K_p = \frac{\Delta H^+}{\Delta H} * 100 \quad (1)$$

Infrared spectra of studied polypropylene fibers and powder were measured by spectrometer IFS8400S with Fourier transformation by a KBr tablet technique.

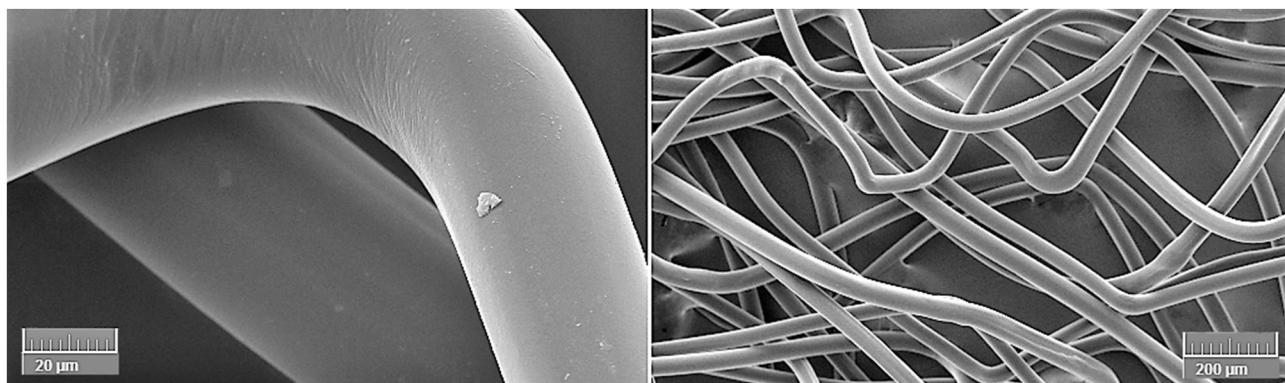


Figure 1 SEM images of short polypropylene fibers

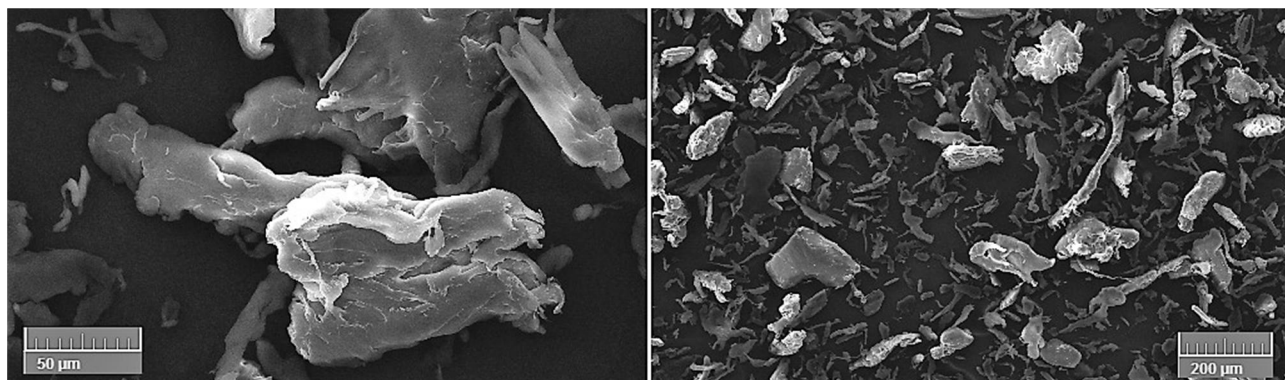


Figure 2 SEM images of polypropylene powder

The subjects PP fibers and PP powder were analyzed by a thermosemission scanning electron microscope in the secondary electron mode. Scanning electron microscopy (SEM) images of the PP fibers (Figure 1) and PP powder (Figure 2) were taken using the Vega 3 Tescan scanning electron microscope with EDX analyser x-act; Oxford Instruments. Image magnification was in the range of 100 to 1 000 x. HV (accelerating voltage): 0.20 - 30 kV, beam intensity level: 1 - 20, absorbed current: 1 pA - 35 nA. The fibers were ultrasonically cleaned, fixed on a conductive substrate and plated with Au-Pd alloy to achieve the optimal signal from the analyzed areas. This type of analysis allows the visualization of the micromorphology of the surface structures of the fibers for their evaluation. The fibers were observed in a macro view and also in a more detailed view of their surface micromorphology.

Synthetic rubber - SBR, activator of curing process - ZnO, stearic acid, accelerator of curing process based on N-cyclohexyl-2-benzothiazolesulfenamide, curing agent - sulfur, plasticizer - aromatic oils, relevant antioxidants and fillers (commercially used carbon black filler N339 and fillers based on polypropylene PP - as waste from the production of PP fibers and foils were used to prepare rubber compound.

The rubber compounds were prepared in kneading machine Plastograf BRABENDER at a temperature of 120°C in stage I and at 100°C in stage II, at a speed of 50 rpm. After the first and second stage, the prepared rubber compounds were homogenized on laboratory twin-cylinder. 6 tread rubber compounds with different amount of filler were prepared. Curing characteristics of prepared rubber blends were measured on the instrument RHEOMETER 100 MONSANTO at the temperature of 150°C for 60 min. Basic parameters such as minimum and maximum torque, the onset of curing process  $t_s$ , optimal curing time  $t_{c(90)}$  and optimal curing rate coefficient  $R_v$  (2) which characterizes the induction period length of the curing curve [ $\text{min}^{-1}$ ] were measured by rheometer instrument.

$$R_v = \frac{100}{t_{c(90)} - t_s} \quad (2)$$

Physical-mechanical properties of prepared vulcanizates were measured on the ZWICK blasting device at the laboratory temperature of 25°C.

Tensile strength was determined by formula (3) where  $F_m$  is max. recorded strength [N],  $W$  is width of the narrowed part of the cutting knife [mm] and  $t$  - thickness of working part [mm].

$$TS = \frac{F_m}{W.t} \quad (3)$$

Elongation at break ( $E_b$ ) was determined by formula (4), where  $L_b$  - length of the working part at break [mm],  $L_0$  - initial length of the working part [mm].

$$E_b = \frac{100.(L_b - L_0)}{L_0} \quad (4)$$

Hardness of resulting vulcanizates was determined by tester according to IRHD. Change of the physical-mechanical properties given by accelerated thermal aging in air can be determined by general formula (5), where  $A_0$  is a physical property before accelerated thermal aging in air and  $A_1$  is a physical property after accelerated thermal aging in air.

$$S = \frac{A_1 - A_0}{A_0} * 100 \quad (5)$$

Dynamical-mechanical properties of selected vulcanizates were measured on DMA Q800, TA instruments device at temperature range from -70°C to 40°C, at frequency 1 Hz with a heating and cooling rate of 3°C/min<sup>-1</sup>, an amplitude of 20 μm.

### 3 RESULTS AND DISCUSSION

#### 3.1 Study of spectral and thermal properties of prepared alternative fillers

Infrared spectroscopy was used to study spectral properties of PP fibers and PP particles (powder form). Significant absorption peaks corresponding to the valence vibration of methyl group -CH<sub>3</sub> were observed in the area ~2960 - 2723 cm<sup>-1</sup>. Characteristic absorption peaks of PP were observed at wavenumber ~1458 cm<sup>-1</sup>, corresponding to the asymmetric deformation vibration of -CH<sub>3</sub> group and peak at wavenumber ~1377 cm<sup>-1</sup> which corresponds to deformation vibration of -CH<sub>2</sub> [22]. Absorption peaks at wavenumber ~997 - 974 cm<sup>-1</sup> correspond to valence vibration of -CH<sub>3</sub> group.

Thermal properties are described in Table 1, where  $T_m$  is sample melting point,  $\Delta H$  is enthalpy of sample melting point,  $K_p$  is crystalline content [%]. The results show that the melting temperatures  $T_m$  of the studied samples of materials are comparable. The highest values of the enthalpy of melting  $\Delta H$  are shown by polypropylene fibers. Crystalline content  $K_p$  results of PV and PP powder are comparable.

**Table 1** Thermal properties of polypropylene fibers and polypropylene powder

Sample	$T_m$ [°C]	$\Delta H$ [J/g]	$K_p$ [%]
Polypropylene fibers	169.7	71.22	35.95
Polypropylene powder	169.8	69.15	34.90

#### 3.2 Study of vulcanization characteristics of tread blends

Table 2 shows the results of determination of curing characteristics of selected parameters (minimum  $M_L$  and maximum  $M_H$  torque, curing rate coefficient  $R_v$ , safety processing  $t_s$  and optimal  $t_{c(90)}$  curing time) of tread blends, namely standard ST, blend samples containing 1 phr of PP powder as a filler (PP-1), samples PP-5 containing 5 phr of PP powder, blend samples PP-10 containing 10 phr of PP powder and

samples of blends containing 1 phr of PP fibers (PV-1) and samples PV-5 containing 5 phr of PP fibers. Vulcanization characteristics results are shown in Figures 3-6.

The resulting values of minimal torque  $M_L$  (Figure 3) of analysed samples PV-1, PV-5 and PP-10 are comparable to minimal torque  $M_L$  of standard sample ST. Except for the sample PP-5 with PP powder, where value of minimal torque was lower than minimal torque  $M_L$  of standard sample.

In the case of the PP-1 sample containing 1 phr of filler, based on polypropylene powder, an increase in the minimum torque  $M_L$  is visible compared to the standard ST, which indicates a higher stiffness and viscosity of the sample with polypropylene powder. Maximum torque  $M_H$  of all tread blends is comparable to maximum torque  $M_H$  of standard sample ST. The highest maximum

torque was obtained in the sample of blend PV-1, based on the PP fibres, compared to standard ST sample.

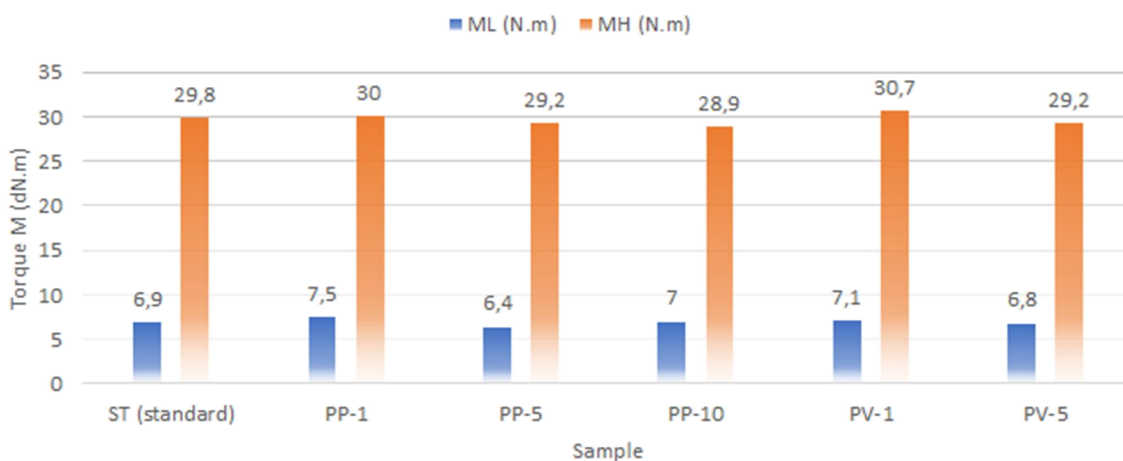
Almost all tread compounds obtained lower safety processing values  $t_s$  (Figure 4), compared to the standard's safety processing value. The increase of  $t_s$  is visible at the sample PV-5, which positively affects processing and physical-mechanical properties of given vulcanizate.

Almost all tread blend samples obtained optimal curing time values  $t_{c(90)}$  (Figure 5) smaller than standard ST sample. For the sample PV-5, a slight increase in  $t_{c(90)}$  is observed over the standard ST.

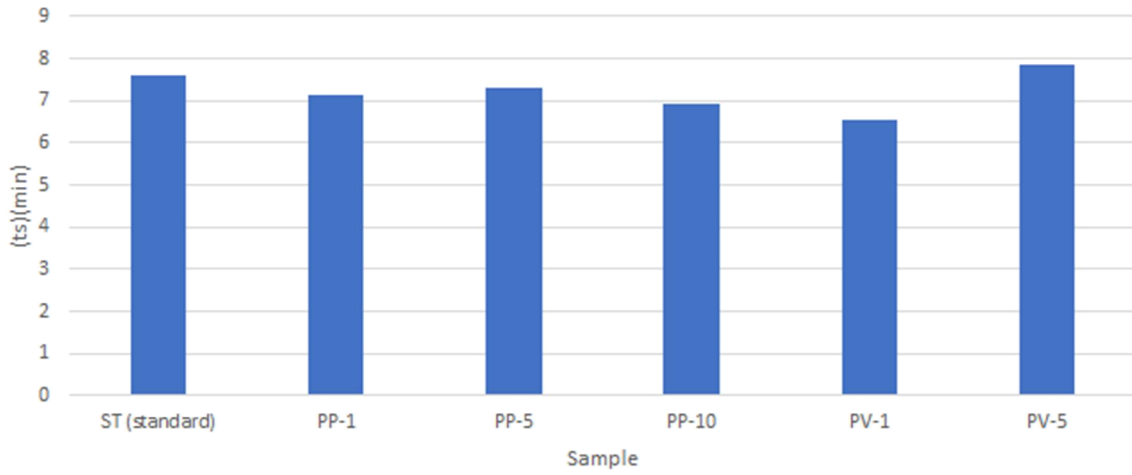
The resulting values of curing rate coefficient  $R_v$  (Figure 6) are comparable for all tread compounds with the standard's curing rate coefficient  $R_v$ , which indicates an efficient and intense mutual interactions of the used curing system components.

**Table 2** Vulcanization characteristics of tread compounds ST, PP-1, PP-5, PP-10, PV-1 and PV-5

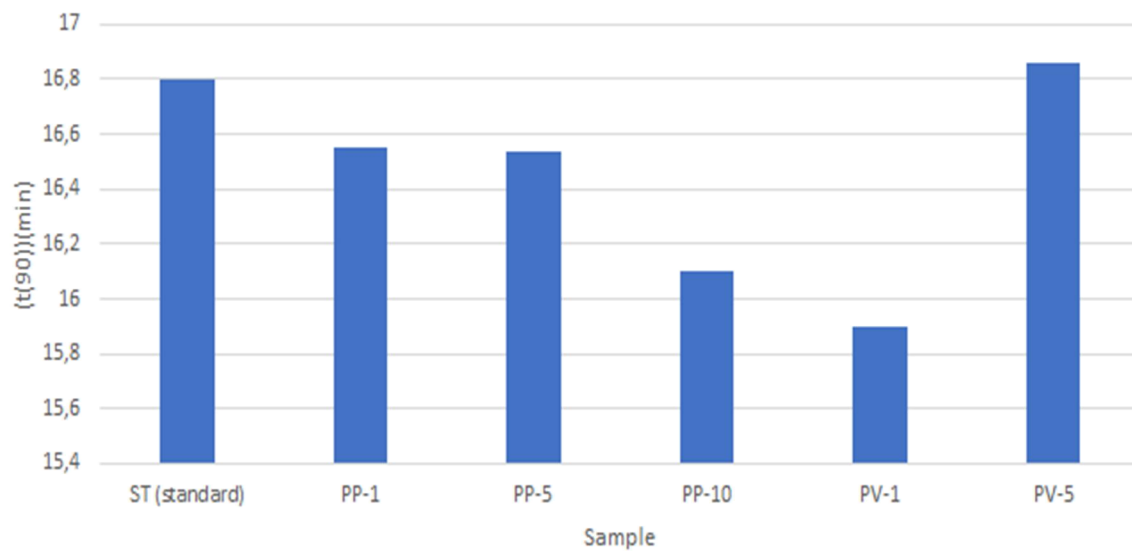
Samples	$M_L$ [N.m]	$M_H$ [N.m]	$t_s$ [min]	$t_{c(90)}$ [min]	$R_v$ [min <sup>-1</sup> ]
ST	6.9	29.8	7.60	16.80	10.87
(PP-1)	7.5	30.0	7.15	16.55	10.64
(PP-5)	6.4	29.2	7.31	16.54	10.83
(PP-10)	7.0	28.9	6.94	16.10	10.92
(PV-1)	7.1	30.7	6.53	15.90	10.67
(PV-5)	6.8	29.2	7.84	16.86	11.09



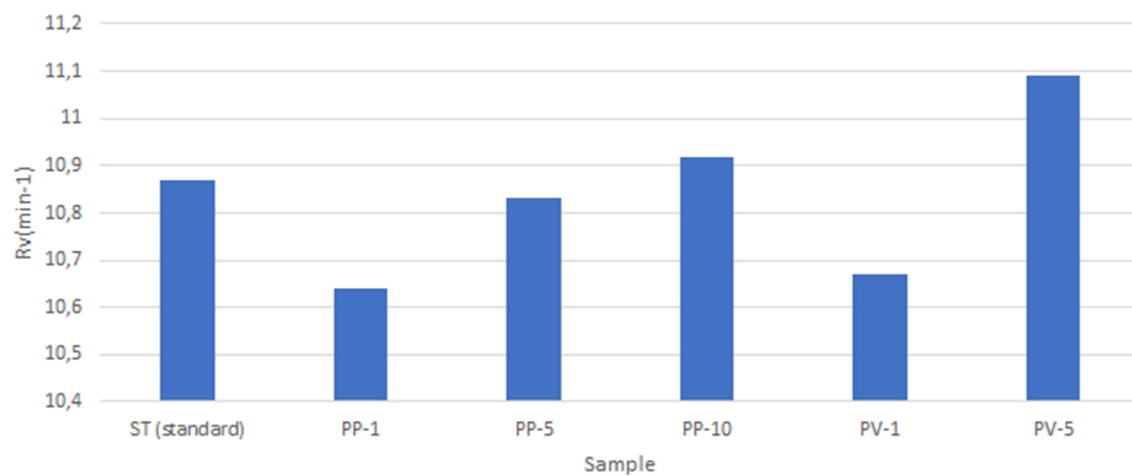
**Figure 3** Minimum and maximum torque of tread blends ST, PP-1, PP-5, PP-10, PV-1 and PV-5



**Figure 4** Safety processing of tread blends ST, PP-1, PP-5, PP-10, PV-1 and PV-5



**Figure 5** Optimal curing time of tread blends ST, PP-1, PP-5, PP-10, PV-1 and PV-5



**Figure 6** Curing rate coefficient of tread blends ST, PP-1, PP-5, PP-10, PV-1 and PV-5

### 3.3 Study of the physical-mechanical properties before and after accelerated thermal aging

Table 3 represents the results of the physical-mechanical properties (hardness, elongation at break and tensile strength) of studied vulcanizates (PP-1, PP-5, PP-10, PV-1 and PV-5) and standard ST before accelerated thermal aging. The results of physical-mechanical properties of studied vulcanizates and standard sample after accelerated thermal aging are given in Table 4. The results of selected physical-mechanical properties before and after accelerated thermal aging are visible in Figures 7-9.

Measured hardness values (Figure 7) were slightly higher for all tread vulcanizates before accelerated thermal aging compared to the hardness values of standard ST sample.

In case of the PV-5 sample with 5 phr PP fibres are hardness results the same before and after accelerated thermal aging.

Results from the elongation at break measurements of tread vulcanizates (Figure 8) show that the elongation at break of all studied samples is lower compared to the standard ST, which indicates a lower elasticity of prepared vulcanizates.

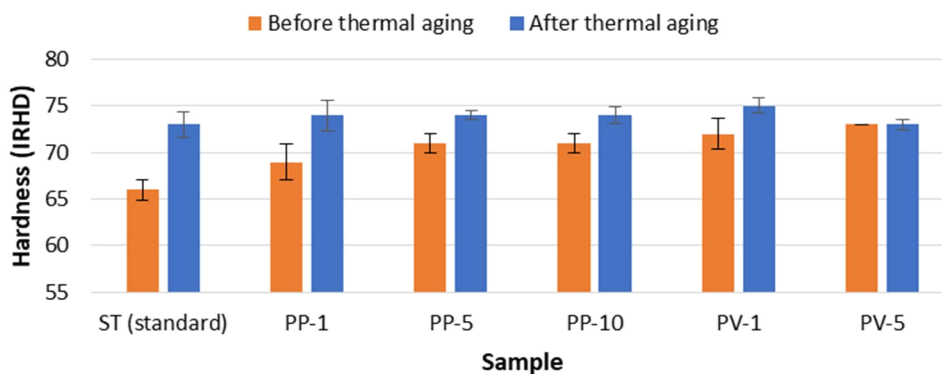
The smallest elongation at break is achieved by the PP-10 sample with filler content 10 phr based on PP powder.

**Table 3** Physical-mechanical properties of tread vulcanizates before accelerated thermal aging in air

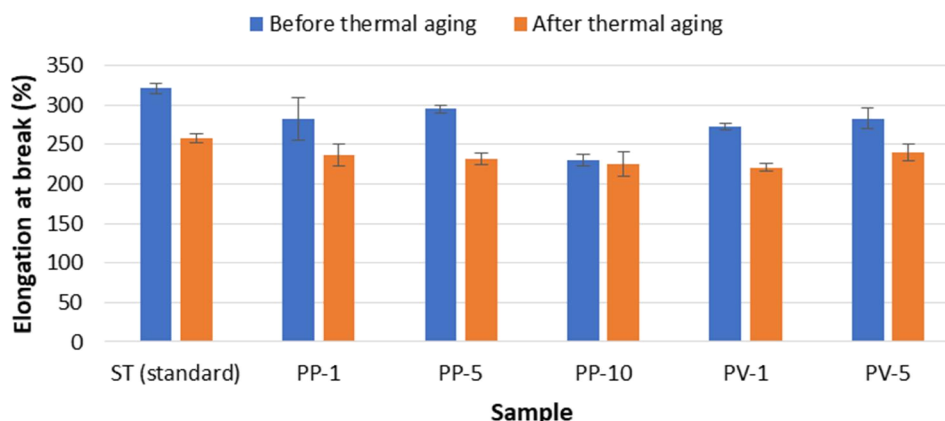
Sample	Hardness [IRHD]	Elongation at break [%]	Tensile strength [MPa]
ST	66±1.09	321±6.88	17.71±0.70
PP-1	69±1.94	282±26.80	16.06±1.40
PP-5	71±1.09	295±4.81	15.84±0.14
PP-10	71±1.09	230±7.59	14.06±0.26
PV-1	72±1.64	272±3.91	16.87±0.57
PV-5	73±0.00	283±12.69	14.63±0.52

**Table 4** Physical-mechanical properties of tread vulcanizates after accelerated thermal aging in air

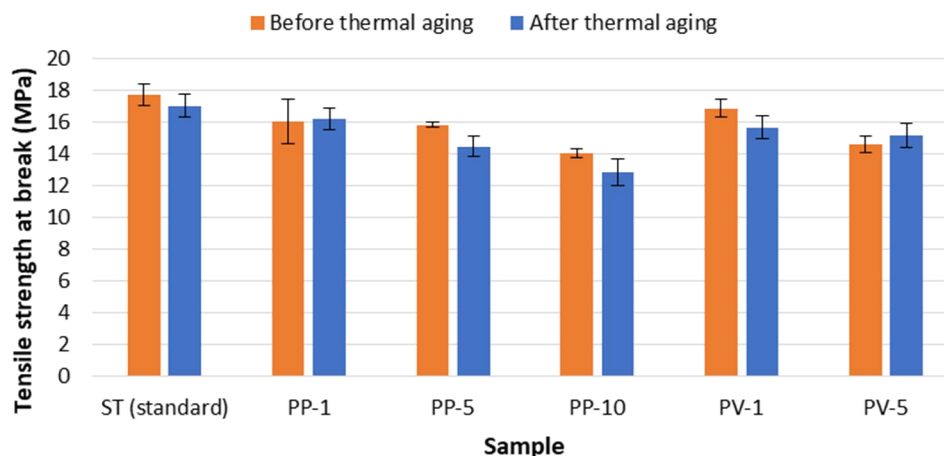
Sample	Hardness [IRHD]	Elongation at break [%]	Tensile strength [MPa]
ST	73±1.41	258±5.83	17.03±0.70
PP-1	74±1.64	237±13.68	16.20±0.65
PP-5	74±0.44	232±7.22	14.47±0.66
PP-10	74±0.89	225±15.13	12.85±0.81
PV-1	75±0.83	221±5.26	15.68±0.71
PV-5	73±0.54	240±10.65	15.13±0.76



**Figure 7** Hardness of tread vulcanizates before and after thermal aging



**Figure 8** Elongation at break of tread vulcanizates before and after thermal aging



**Figure 9** Tensile strength of tread vulcanizates before and after thermal aging

Comparing all results, it is clear that the elongation at break after accelerated thermal aging is lower than before running aging experiment.

Resulting values of tensile strength before accelerated thermal aging shows that all of the tread vulcanizates with PP obtains lower values than standard vulcanizate ST, except for the PV-1 sample which is comparable to standard. Lower tensile strength may relate to weaker interaction between filler particles and rubber matrix. Tensile strength decreases with increasing PP powder or fibres. The lowest tensile strength is achieved by the PP-10 vulcanizate with 10 phr PP filler.

Tensile strength changes results before and after accelerated thermal aging in air of tread vulcanizates (Figure 9) samples PV-1, PP-5 and PP-10 achieved larger tensile strength decrease than standard ST. Although the tensile strength values after aging of tread vulcanizates with alternative fillers are lower compared to the ST standard, they all meet the limit values set for treads.

The physico-mechanical properties of all vulcanizates before and after accelerated thermal aging were compared and the observed increase or decrease in the values of the studied parameters was expressed as a percentage (Table 5).

The obtained hardness results of tread vulcanizates with alternative PP fillers and the standard ST before and after accelerated thermal aging show that the all samples (PP-1, PP-5, PP-10, PV-1, PV-5) achieve a lower percentage increase in hardness, compared to the standard ST. It is a positive trend. Evaluation of the elongation at break results of the tread vulcanizates before and after accelerated thermal aging shows lower percentage changes in elongation at break compared to the ST standard, except for PP-5 vulcanizate. In the case of the PP-5 sample, the largest percentage change is visible compared to the standard ST. The PP-10 sample with 10 phr PP filler replacement achieved

the smallest percentage change in elongation at break. An increase in tensile strength after thermal aging (expressed as a percentage) is observed for the PP-1 sample containing PP powder (1 phr) and the PV-5 sample with PP fibers (5 phr) compared to the ST standard, which is a positive finding.

**Table 5** Changes in physical-mechanical properties of tread vulcanizates before and after accelerated thermal aging expressed in %

Sample	Hardness increment ↑ [%]	Elongation at break decline ↓ [%]	Tensile strength decline ↓ /increment ↑ [%]
ST	↑ 10.6	↓ 19.62	↓ 3.83
PP-1	↑ 7.2	↓ 15.95	↑ 0.87
PP-5	↑ 4.2	↓ 21.35	↓ 8.64
PP-10	↑ 4.2	↓ 2.17	↓ 8.60
PV-1	↑ 4.2	↓ 18.75	↓ 7.05
PV-5	=	↓ 15.19	↑ 3.41

### 3.4 Study of selected properties of vulcanizates from DMA analysis

Viscoelastic behavior of prepared PV-1 vulcanizates containing filler based on PP fibers and vulcanizates PP-1, based on PP in powder form, was evaluated on the basis of obtained results by dynamic-mechanical analysis in the temperature range from -70°C to 40°C.

The studied values of  $T_g$ ,  $tg \delta_{max}$ ,  $tg \delta$  (at -25°C) and  $tg \delta$  (at 0°C) for selected samples PP-1 and PV-1 compared to the standard ST are given in Table 6. Loss angles  $tg \delta$  maximum peaks of study vulcanizates ST, PP-1 and PV-1 specify the glass transition temperatures. The values of the glass transition temperatures  $T_g$  are comparable for the vulcanizates PV-1 and PP-1 with alternative fillers with the standard vulcanizate ST. From the values of the maximum peaks  $tg \delta$  (Table 6), it is possible to predict the stiffening effect of the chosen

fillers based on PP powder or PP fibers in vulcanizates PP-1 and PV-1 in comparison with standard vulcanizate ST. The stiffening effect of the filler is related to the restriction of the mobility of the segments of the rubber molecules. The lower the value of  $tg \delta_{max}$ , the higher the stiffening effect of the filler. In this case, the interaction of the filler with the rubber matrix increases [7, 23]. The peaks  $tg \delta_{max}$  for vulcanizates samples PP-1 and PV-1 are lower compared to standard ST. The vulcanizate sample PV-1 with filler content based on PP fibers obtains lowest  $tg \delta_{max}$  which indicates a better stiffening effect of resulting vulcanizates. The results show that the obtained  $tg$  values of the studied vulcanizates are influenced by the chemical composition and particle shape of the alternative filler.

**Table 6** The values  $T_g$ ,  $tg \delta_{max}$ ,  $tg \delta$  (at  $-25^\circ\text{C}$ ) and  $tg \delta$  (at  $0^\circ\text{C}$ ) of selected samples of vulcanizates PP-1 and PV-1 compared to standard ST

Vulcanizates	$T_g$ [ $^\circ\text{C}$ ]	$tg \delta_{max}$	$tg \delta$ (at $-25^\circ\text{C}$ )	$tg \delta$ (at $0^\circ\text{C}$ )
ST	-28.0	0.81	0.76	0.36
PP-1	-27.0	0.79	0.77	0.38
PV-1	-28.0	0.73	0.68	0.34

Properties such as traction of the tire tread on snow and ice ( $tg \delta$  at  $-25^\circ\text{C}$ ) and wet traction ( $tg \delta$  at  $0^\circ\text{C}$ ) can be observed by the dependence of the loss angle on temperature for resulting vulcanizates.

Traction of the tire tread on snow and ice is characterized by  $tg \delta$  at  $-25^\circ\text{C}$ . The results of the measurements show that the sample of vulcanizate PP-1, has a slightly higher value of  $tg \delta$  (at  $-25^\circ\text{C}$ ) compared to the ST standard, which is required for the evaluation of traction on snow and ice. The values of  $tg \delta$  ( $-25^\circ\text{C}$ ) increase in order:  $v$  (PV-1) <  $v$  (ST) <  $v$  (PP-1). Wet traction of the tire tread is characterized by  $tg \delta$  at  $0^\circ\text{C}$ . The results of the measurements show that the prepared sample of vulcanizate PP-1 in the amount of PP powder 1 phr, shows a higher value of  $tg \delta$  (at  $0^\circ\text{C}$ ) compared to the ST standard, which is positive in the evaluation of wet traction. The values of  $tg \delta$  ( $0^\circ\text{C}$ ) increase in order:  $v$  (PV-1) <  $v$  (ST) <  $v$  (PP-1).

#### 4 CONCLUSION

Five tread blends with certain amount of alternative fillers were prepared and study, namely three tread blends PP-1, PP-5, PP-10 with amount of 1 phr, 5 phr and 10 phr fillers based on polypropylene powder and 2 blends PV-1, PV-5 with amount of 1 phr and 5 phr of filler based on polypropylene fibers and one standard ST tread blends containing filler - carbon black (N339). Evaluation of optimal curing time results in the partial decrease of optimal curing time for PP-1 PP-5 PP-10 and PV-1 tread blends comparing with optimal curing time

of standard ST. The vulcanization process was faster at described vulcanizates. Vulcanizates with a content of PP filler obtained lower values of tensile strength comparing to standard ST during the evaluation of vulcanizates physical mechanical properties before and after accelerated thermal aging in air at temperature of  $100^\circ\text{C}$ . Smaller values of tensile strength may be connected to a weaker interaction of filler particles with rubber matrix. Tensile strength values were decreasing gradually with an increasing content of alternative filler replacement. All of the prepared vulcanizates achieved the required values of tensile strength 12-18 MPa, determined for tread. Values of vulcanizates elongation at break with a content of alternative filler were lower comparing to standard ST before and after thermal aging. The trend for hardness values was opposite. Hardness values of vulcanizates with a content of PP fillers were higher before and after accelerated thermal aging in air comparing to standard.

The evaluation of the DMA analysis of the PP-1 and PV-1 tread vulcanizates showed that the glass transition temperature  $T_g$  of the PP-1 and PV-1 vulcanizates did not change significantly compared to the standard ST. Size values of max. peak  $tg \delta_{max}$  were lower for vulcanizate samples comparing to the standard ST. The lowest values of  $tg \delta_{max}$  were obtained from vulcanizate PV-1 with a content of 1 phr filler based on PP fibers, which indicates the best solidification of the resulting vulcanizates. Selected properties of tread vulcanizates, traction on snow and ice ( $-25^\circ\text{C}$ ) and wet traction ( $0^\circ\text{C}$ ) were evaluated by the dependence of the loss angle  $tg \delta$  on the temperature. The results show that the prepared vulcanizate PP-1 had higher values of  $tg \delta$  ( $-25^\circ\text{C}$ ) and  $tg \delta$  ( $0^\circ\text{C}$ ) compared to the standard ST, which is a positive finding when evaluating traction on snow and ice and wet.

The evaluation results the possibility of the prepared tread blends to use selected studied materials in the function of fillers, as a partial replacement of standardly used fillers - carbon black N339. The results of the study provide new knowledge about the application of polypropylene waste from the production of PP fibers and PP foils in the function of fillers in rubber blends.

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