

Effect of alternative carbon-based filler on rubber compounds properties

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Abstract: Instead of conventional carbon black, an alternative carbon filler (ACB) obtained in the pyrolysis process from tire waste was used for rubber compounds. The fillers were characterized using FTIR and TGA methods. The mechanical properties of the obtained rubber compounds and the influence of the fillers used on the vulcanization process, as well as the Payne effect, were examined. The results confirm the possibility of replacing conventional carbon black with an alternative carbon filler in rubber compounds.

Keywords: carbon-based filler, rubber compound, thermal properties, mechanical properties, Payne effect.

Wpływ alternatywnego napełniacza węglowego na właściwości mieszanek gumowych

Streszczenie: Do mieszanek gumowych zamiast konwencjonalnej sadzy zastosowano alternatywny napełniacz węglowy (ACB) otrzymany w procesie pirolizy z odpadów opon. Napełniacze scharakteryzowano za pomocą FTIR i TGA. Zbadano właściwości mechaniczne otrzymanych mieszanek gumowych oraz wpływ stosowanych napełniaczy na przebieg procesu wulkanizacji, a także na efekt Payne'a. Wyniki potwierdzają możliwość zastąpienia konwencjonalnej sadzy alternatywnym napełniaczem węglowym w mieszankach gumowych.

Słowa kluczowe: napełniacz węglowy, mieszanka gumowa, właściwości termiczne, właściwości mechaniczne, efekt Payne'a.

The motor vehicles have become necessities in daily life [1]. A tire is a key component of a vehicle driving system and is the only component that interacts with vehicle and road [2]. In recent years, the demand for tires and the number of end-of-life tires is increasing at the same time [1]. About 1.5 billion tires are produced around the world in a year, which corresponds to an estimated seventeen million tons of used tires [3]. The large amount of waste tires aggravates the two major international problems of energy supply and environmental pollution [3, 4].

Pyrolysis is an effective treatment mechanism for biomass and municipal solid waste, it is particularly suit-

able for waste tires as it can effectively separate primarily carbon black derived carbon (which produces char after pyrolysis) from the rubber and textile (from the reinforcing cord) derived carbon compounds (which produce oil and gas), enabling more effective recovery of both product streams. Tire-derived pyrolysis char contains a large amount of carbon black, a material with a high surface area and surface activity, which can retail for up to \$1000/ton, when destined for use as a recycled filler in new polymers, including tires [5]. Carbon black is a carbon material close to pure form of combustion products derived from hydrocarbon or biomass products. Its aggregate size depends on combustion temperature, combustion time, and material used [6]. However, carbon black is by far the dominant filler in rubber products. The global production of carbon black is fifteen million metric tons per year, and 93% of this material goes into rubber applications, where automobile tires take 73% and non-tire rubber products 20%, with the remaining 7% used in paints, coatings, inks, and plastics compounding [7]. Carbon black is the most widely used reinforcing filler in

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rubber industry [8]. In the rubber industry, carbon black plays a significant role as a reinforcing filler, but it is manufactured from non-renewable petrochemical materials. From the perspective of sustainable and green chemistry, the customary status of carbon black has been severely challenged and many efforts have been devoted to the exploration of novel alternatives with low environmental pollution and high performance for rubber reinforcement [9]. Several authors have recently analyzed information regarding the characteristics of recovered carbon black [1, 10]. The latest studies show the application of recovered carbon black in the rubber [1].

Without fillers, cross-linked rubbers are weak materials. Reinforcing filler particles enhance the tensile strength, tear resistance, and abrasion resistance of rubber to varying degrees based on the particle size and shape features and nature of elastomer-filler interactions, while also increasing static and dynamic stiffness characteristics. All these mechanical property improvements can be considered as reinforcement. The most prevalent filler type in demanding rubber applications such as automobile tires and mechanical rubber is carbon black, which has many grades encompassing an enormous range of particle sizes (surface areas) [7]. Carbon black-filled rubber is one of the most important elastomers and can broaden the application of natural rubber by incorporating carbon black filler. After the incorporation of carbon black filler, the mechanical properties of rubber-like materials will be enhanced, but the dynamic non-linear viscoelasticity will be affected, such as the Payne effect [11].

The purpose of the presented work is the replacement of the conventional filler with the studied alternative carbon-based filler to reduce the price of the rubber products significantly, which would further promote the recycling of waste tires and will contribute to the sustainability of production. The conventional filler plays significant role as a reinforcing filler, but it is manufactured from non-renewable petrochemical materials. The alternative carbon-based filler is recovered carbon black derived from car tire pyrolysis. The research is focused on study of rheological and mechanical properties as well as reinforcing effect in rubber compound.

EXPERIMENTAL PART

Materials

In this study, the conventional filler-carbon black (N339) was replaced by alternative carbon-based filler

derived from the pyrolysis of car tires in rubber compounds. Herein, Brunauer-Emmett-Teller surface area (BET) of alternative carbon-based filler and N339 was measured using a surface area analyzer. Surface area (BET) of N339 was 89.8 m²/g, alternative carbon-based filler had smaller values of BET (39.2 m²/g).

Using EDX analysis, the fillers element analysis is given in Table 1. Over 97% of overall composition is carbon. The alternative carbon-based filler (ACB) contains smaller amount of carbon in compare with the N339 and larger amount of silicon, zinc, and calcium. These elements are included in white fillers, which is beneficial due to the combination of silica and carbon black to exploit the advantages of both fillers in rubber compounds. Successively, different authors [12–14] demonstrated that the addition of lesser amounts of silica into rubber compounds decreases the filler cluster branching and increases the reinforcement efficiency, while higher silica loadings induce segregation phenomena, causing a deterioration in the filler-rubber interface and consequently deterioration properties of rubber compound. Based on this experimental evidence, tire industry has developed the use of hybrid filler systems, where carbon black is only partially substituted by precipitated silica functionalized with silane coupling agents [14]. These rubber compounds show improved dynamic response at low and high temperatures with respect to those reinforced solely with carbon black, which results in better rolling resistance and enhanced wet grip, while keeping satisfactory abrasion resistance [12].

Preparation of rubber compounds

The three rubber compounds (ACB0, ACB50, ACB100) were prepared for this study. Herein, the comparison of alternative carbon-based filler with conventional filler carbon black specifically carbon black N339 is considered. The alternative carbon-based filler was used as half (ACB50) and total replacement (ACB100) for carbon black N339 (ACB0) in the tread blend. The rubber compounds were prepared by mixing via Brabender EC PLUS (Duisburg, Germany) with 80 cm³ volume of chamber. The mixing process was based on one step within temperature of 80°C at the 50 rpm. The synthetic rubbers were masticated for 3 min before the addition of ZnO and steric acid. Then, the filler was incorporated into the chamber and mixed for 4 min. Mixing process for the preparation of rubber compounds is shown in Figure 1.

Table 1. EDX analysis of alternative carbon-based filler

Sample	Element, %					
	C	Si	Zn	S	Ca	Fe
ACB	93.397	3.142	1.535	1.262	0.320	0.228
CB (N339)	99.134	0.121	0.008	0.647	0.062	0.009

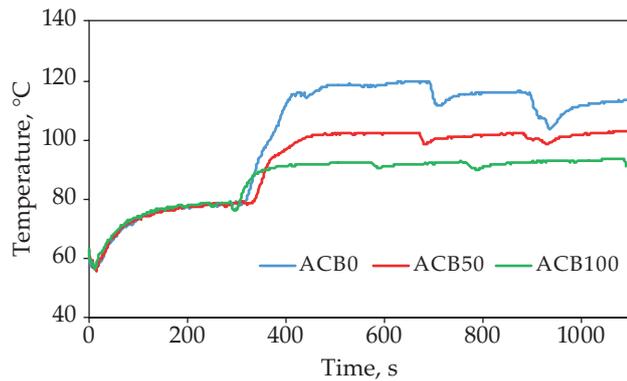


Fig. 1. Temperature during the mixing process for various fillers

After incorporation of filler carbon black (N339) into the chamber the temperature of rubber compound was rising rapidly. In compare with alternative carbon-based filler - the temperature of compound in chamber was rising slowly. Subsequently, activators, antioxidant and anti-ozonant as well as plasticizer – residual aromatic extract (RAE) were added. The curing system was applied as the last one. After mixing process, the tread blends were left to be used for the further measurement. The characterization of the prepared samples is given in the Table 2.

Table 2. Composition of rubber compound for different fillers

Sample	Rubber 100 wt%	CB-N339 wt%	ACB wt%
ACB0	SBR 1723	87	0
ACB50	SBR 1500	43.5	43.5
ACB100		0	87

Methods

Thermogravimetric analysis

The thermal degradation behavior of fillers (ACB, CB-N339) was studied. Thermal analysis of the fillers was carried out in the temperature range from 30°C to 800°C with a heating rate of 10°C/min in N₂ atmosphere by using TGA/DSC 2 (Metler Toledo Greifensee, Switzerland).

FTIR analysis

FTIR spectra of fillers (ACB, CB-N339) were gathered on a FTIR spectrometer with FT-Raman module Thermo Scientific Nicolet iS50 (Waltham, MA, USA) within a wavenumber range from 4000 to 500 cm⁻¹.

Table 3. Thermal degradation of fillers

Filler	The first step		The second step	
	Temperature range, °C	Δm , %	Temperature range, °C	Δm , %
CB (N339)	40–220	1.56	220–800	4.58
ACB	40–220	4.61	220–800	11.42

Cure characteristics

The cure characteristics of rubber compounds were determined via production rubber process analyzer (PRPA) oscillation rheometer (PRPA 2000, Alfa Technologies, Akron, OH, USA) at the constant temperature of 160°C, with oscillating arch of 0.5°, frequency of 1.67 Hz. Cure characteristics in terms of minimum torque (M_L), maximum torque (M_H), Scorch time (t_{s2}) and cure time (t_{c90}) was calculated as the time of 90% cure. CRI was calculated as $[100/(t_{c90} - t_{s2})]$.

Payne effect

Using PRPA, it was also possible to study the elastic modulus (G') in cured and uncured sample in the strain range between 0.28% and 100% and frequency of 1 Hz. Payne effect of the uncured rubber was measured at a temperature of 70°C. Payne effect of the cured sample was cured at a temperature of 170°C for 10 minutes and then this sample was measured at a temperature of 70°C too. In the introduced study the Payne Effect was calculated by difference between the elastic modulus at 0.28% and the 100% strain ($\Delta G' = G'_{0.28\%} - G'_{100.02\%}$) for cured and uncured samples.

Mechanical properties

Prepared vulcanizate test samples in shape of the double-sided blades were mechanically cut out from the vulcanized plate. Using Shimadzu Autograph AG-X plus (Kyoto, Japan) machine with a head speed of 500 mm/min, the mechanical properties were measured. The tensile strength and elongation were obtained from the average of five loaded blades. The hardness was measured by Shore A hardness tester.

RESULTS AND DISCUSSION

Thermogravimetric analysis

TGA results, see Table 3, indicated changes in the thermal degradation of alternative carbon-based filler, as seen in Figure 1 in compare with carbon black (N339), as seen in Figure 2. The TGA curves reveal the thermal degradation of fillers in two phases: 40–220°C – a mass loss is primarily due to evaporation of volatile substance and water. The alternative carbon-based filler indicates higher loss of weight in compare with carbon black (N339). Natthawat

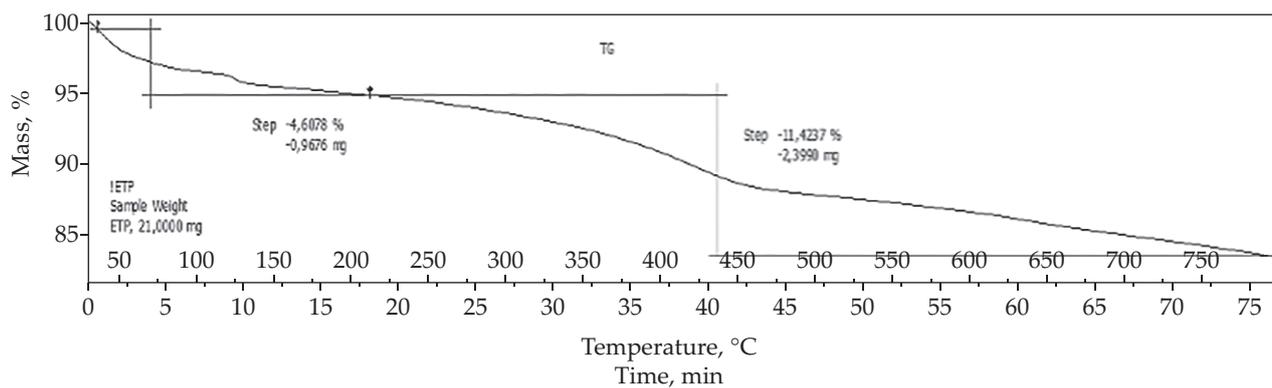


Fig. 2. TGA curve of alternative carbon-based filler (ACB)

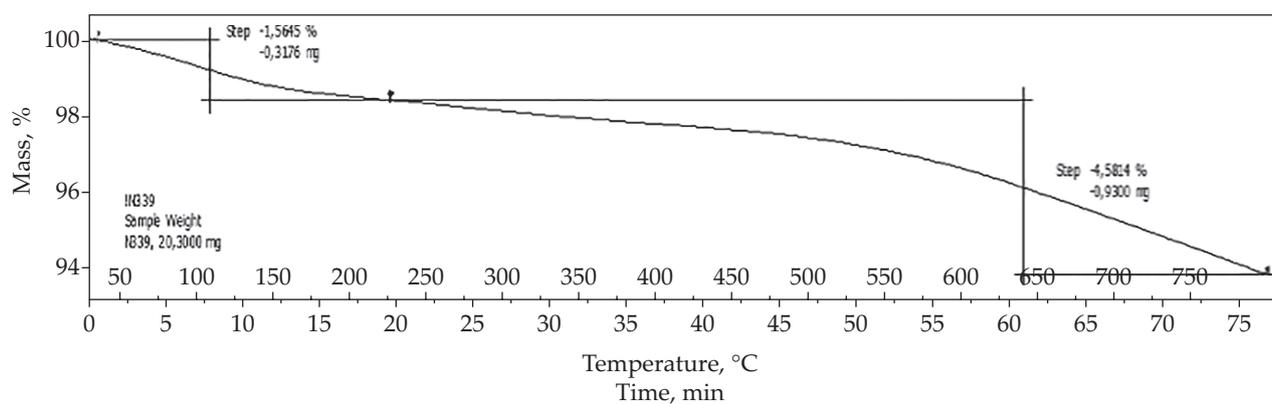


Fig. 3. TGA curve of carbon black N339 (CB)

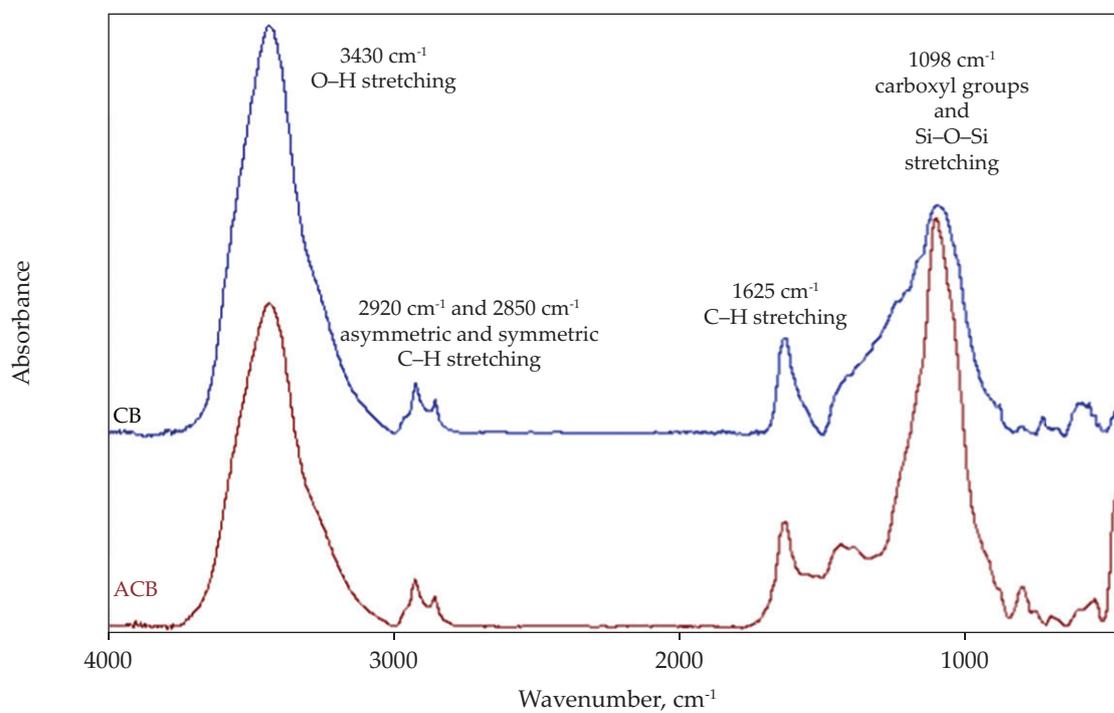


Fig. 4. FTIR spectra of CB-N339 and ACB fillers

Table 4. Cure characteristics of ACB0, ACB50 and ACB100 samples

Samples	M_L , Nm	M_H , Nm	ΔM , Nm	$t_{c2'}$, min	$t_{c90'}$, min	CRI, min ⁻¹
ACB0	0.382	2.241	1.859	1.90	5.49	27.86
ACB50	0.304	1.771	1.467	1.99	6.60	21.69
ACB100	0.212	1.557	1.345	1.93	4.80	34.84

[15] described in his study of recovered carbon black, that in the initial stage up to 100°C, less than 5% moisture removal was observed. The thermal decomposition of fillers started at 220–800°C. A bigger variation in the weight loss was observed for alternative carbon-based filler, indicating of removing the inorganic residues too. Authors [15] described in article that when the temperature is in the range of 620°C to around 800°C, a significant loss of weight becomes apparent because of the removal of the carbon black additive from recovered carbon black.

Fourier transform infrared analysis

Figure 4 illustrates the FTIR spectra of both fillers (ACB and CB-N339). The spectra of alternative carbon-based filler (ACB) showed one significant difference in the intensity of peak and broad of a band. As in Urrego-Yepes [16] the band around 1100 cm⁻¹ is much broader in CB-N339 than in ACB, which suggests a higher concentration of carboxyl groups in the former. In our case, the broad of band around 1098 cm⁻¹ suggests a higher concentration of carboxyl groups in CB-N339 and intensity of peak relates to Si–O–Si stretching due to the presence of Si element in the EDX analysis of alternative carbon-based filler. Alomairy [17] in article describes that the band at 1095–1048 cm⁻¹ is related to Si–O–Si asymmetric stretching. Another author [18] describes the absorption bands at 920 and 1098 cm⁻¹ are related to the Si–O–H bending and Si–O–Si stretching vibrations, respectively. Mohammadi [19] studied the spectra of pure SiO₂ sample which showed two main bands at 800 cm⁻¹ and 1097 cm⁻¹ correspond to the Si–O–Si bending vibrations and Si–O–Si asymmetric stretching, respectively. The FTIR analysis indicated a higher presence of carboxyl functional groups in CB-N339 and ACB spectra, furthermore, indicated Si group in ACB spectrum.

Cure characteristics

Table 4 summarizes the cure characteristics, including M_L , M_H , $\Delta M = (M_H - M_L)$, $t_{c2'}$, $t_{c90'}$, CRI. The results revealed that the presence of alternative carbon-based filler ACB obtained lower M_L in compare with sample filled with carbon black (N339). M_L refers to the viscosity (stiffness) of the rubber compounds [20, 21]. During vulcanization period, the decrease in torque was caused by the decrease in the cross-linking of the rubber matrix. ACB100 sample showed low M_H and ΔM however the difference is not so significant for the total replacement. The ΔM implied the cross-link density of the rubber compounds. ACB0

sample showed the highest cross-link density. The total replacement of alternative carbon-based filler for carbon black N 339 in the tread blend (ACB100 sample) shows similar crosslink density as ACB50 sample with half replacement of carbon black.

The t_{s2} parameter was higher for compound with alternative carbon-based filler. The t_{s2} parameter is the time taken for the M_L to increase by two units and it indicates how fast the material starts to be cured [20]. The presence of alternative carbon-based filler provided low CRI for ACB50 sample and higher CRI for ACB100 sample in compare with ACB0 sample. The $t_{c90'}$ had opposite character. The ACB50 sample showed longer cure time and the ACB100 sample showed shorter cure time of the rubber compound in compare with ACB sample filled of carbon black (N339). From economic point of view, a shorter cure time is more beneficial.

We can state, that applying of alternative carbon-based filler is possible for replacement of carbon black N339 in the tread blend. Yet, for better result of torques, the modification of alternative carbon-based filler is necessary.

Payne effect of rubber compounds

The storage modulus (G') decreased with the increase in strain due to the breakdown of the filler network within the rubber matrix. The storage modulus in the strain range of uncured and cured samples can be seen in Figure 5a and Figure 5b, respectively. As shown in Figure 5, the storage modulus of ACB0 sample decreased rapidly in compare with ACB50 and ACB100 samples. We can suppose the quick breakdown of the filler network within the rubber matrix. The storage modulus for uncured and cured ACB0 sample is rapidly higher at low strain and low at high strain. This increase is caused by the better formation of filler-filler interactions (higher reinforcement) which are formed in matrix. The reinforcement is decreasing with higher amount of alternative carbon-based filler in rubber compound.

An indication of the degree of filler–filler interactions within the rubber matrix can be estimated by the difference in the storage modulus at 0.28% ($G'_{0.28}$) and 100% (G'_{100}) strains, *i.e.*, $\Delta G' = G'_{0.28} - G'_{100.02\%}$. The Payne effect will be higher, the greater is the value of $\Delta G'$, meaning a higher amount of filler-filler interaction [22]. On the other side, the smaller the filler-filler interaction, the better the filler particles are dispersed [23–26]. The results Figure 6 confirmed, the filler–filler interactions are aggravated with increasing amount of alternative

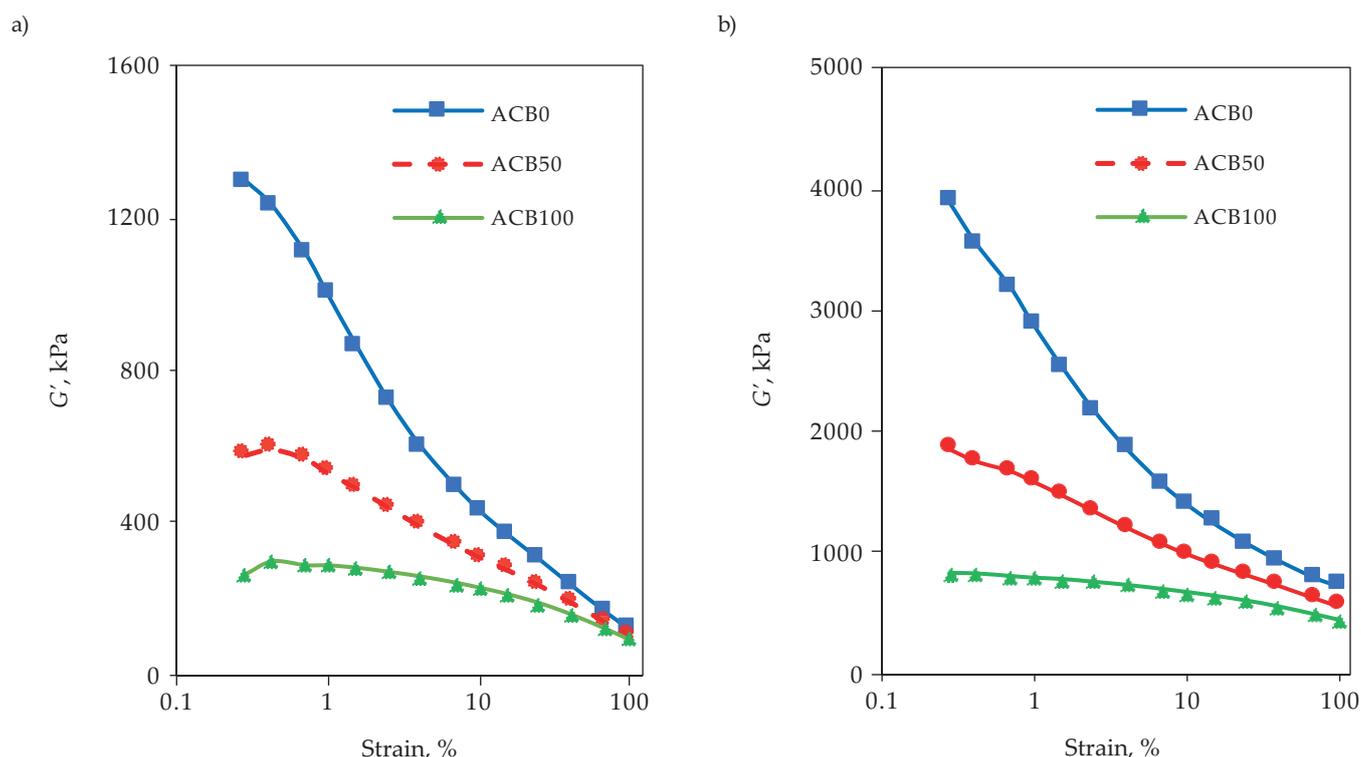


Fig. 5. The storage modulus (G') in the strain range of ACB0, ACB50 and ACB100 samples: a) uncured, b) cured

carbon-based filler. Based on the aggravated interactions of filler-filler and filler-rubber, it is possible to attribute the less reinforcing of ACB50 and ACB100 due to the decreased G' .

Mechanical properties

The mechanical properties as tensile strength, elongation and hardness are shown in Figures 7–9. The results revealed that the incorporation of alternative carbon-based filler in the rubber compounds worsened the tensile properties. But ACB50 sample with half replacement of carbon black has slight decrease of tensile strength. Here is the possibility of modification of filler which would improve the reinforcement of alternative carbon-based filler. Zhang [1] in article wrote that using the coupling agent is a simple and effective approach to the high-valued application of recovered carbon black

in natural rubber. A novel coupling agent of sodium (Z)-4-((4-aminophenyl)amino)-4-oxobut-2-enoate was added natural rubber during mixing process, and significantly improved the reinforcement properties of recovered carbon black for natural rubber.

As can be seen in Figure 8 the incorporation of alternative carbon-based filler increased the elongation at break. This finding was attributed to the low cross-link density of the alternative carbon-based filler added into the rubber compound, which displayed the influence on the tensile properties. The higher reinforcement restricts the mobility of polymer segments, which finally result in a reduction of elongation (ACB0 sample) and corresponds with result of G' and the tensile properties.

As seen in Figure 9 hardness has decreased with higher amount of the alternative carbon-based filler in comparison with ACB0. These results may be explained in terms of lower crosslinking density.

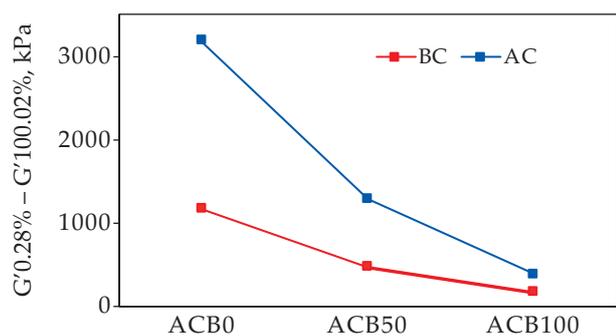


Fig. 6. Payne effect evaluation of ACB0, ACB50 and ACB100 samples (BC – before cure, AC – after cure)

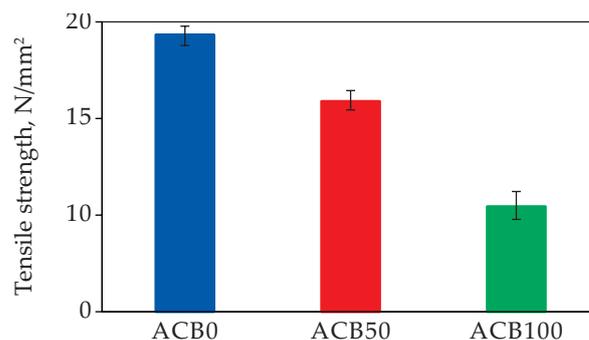


Fig. 7. Tensile strength of ACB0, ACB50 and ACB100 samples

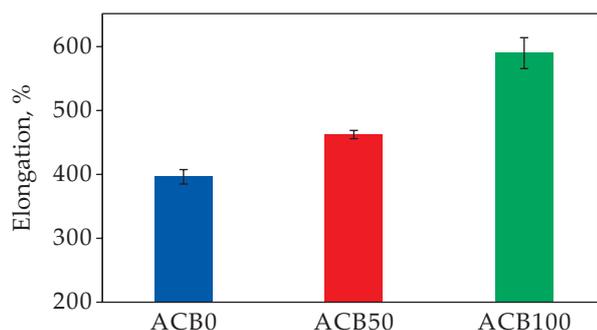


Fig. 8. Elongation of ACB0, ACB50 and ACB100 samples

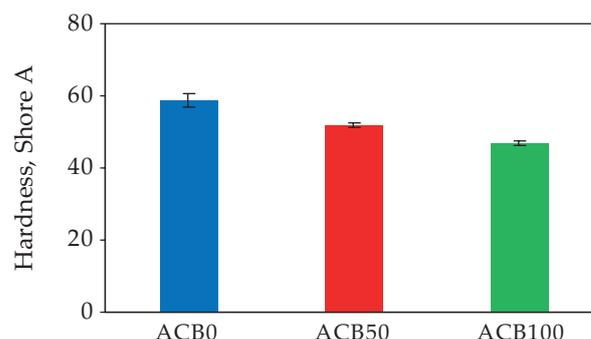


Fig. 9. Hardness of ACB0, ACB50 ACB100 samples

CONCLUSION

Motor vehicles have become a necessity in everyday life, and number of end-of-life tires has increased substantially. The purpose of the submitted work was to characterize the alternative filler based on carbon and its application in rubber compounds. Subsequently, the rubber compounds properties were evaluated. From the performed measurements can be seen, that the alternative carbon-based filler from waste tire pyrolysis, in 100 phr loading improved optimum cure time to 4.80 min, comparing with the CB-N339 loading in ACB0, hence the use of alternative carbon-based filler is beneficial on behalf of economic sphere. In addition, the full loading of 100 phr in ACB100 has proven higher efficiency of curing, thus it still maintains beneficial from the economic point of view. However, in this work was used only the rubber matrix based on SBR 1723 and SBR 1500, then the use of natural rubber and a modification of alternative carbon-based filler could allow the improvement of rubber compound reinforcement, thus the improvement of mechanical properties.

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