

EFFECT OF MICA POWDER-FILLED STYRENE-BUTADIENE RUBBER COMPOUNDS ON CROSSLINK DENSITY AND MECHANICAL PROPERTIES

by

Saban BULBUL^{a*} and Mehmet Emin ERGUN^b

^a Faculty of Engineer Seydisehir Ahmet Cengiz, Necmettin Erbakan University, Konya, Turkey

^b Akseki Vocational High School, Alanya Alaaddin Keykubat University, Antalya, Turkey

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In this study, four different compounds were produced by adding different proportions of mica powder (0-5-10-20%) to styrene-butadiene rubber. After vulcanization, the crosslink density, hardness, density, tensile strength, elongation percentage, and tearing strength of the compounds were measured. In addition, the effects of crosslink density on mechanical properties were discussed. The physio-mechanical properties of the new compounds produced were compared both among themselves and with the properties of the rubber being referenced. It was observed that increasing the crosslink density of mica powder provided an advantage in terms of hardness, tensile strength, tearing strength, and percentage elongation properties. To explain the changes in mechanical properties, the tensile fracture surfaces were determined by SEM and energy dispersive spectroscopy. In the light of the obtained results, it was determined that the use of carbon black could be reduced by using mica powder in the rubber industry.

Key words: compound, rubber, mechanical properties, crosslink, mica powder

Introduction

Rubber is a crucial and versatile polymer material, which is considerably used in shoe outsole, pharmaceutical, health industries, aerospace, military, and automotive [1]. One of the many rubber types is also the styrene-butadiene rubber (SBR) which is synthesized from 1,3-butadiene and styrene monomers. The SBR has attracted a lot of attention in recent years because of its resistance to cracking, aging, weathering, and wear. It is expected that the global SBR capacity will reach 8 million metric tons by 2022 and the compound annual growth rate will be between 2.8% and 3% [2]. Despite rising demand for SBR-based compounds, SBR has a number of shortcomings in terms of thermal stability and mechanical properties that require improvement [3]. The addition of particular filler materials into the rubber matrix to enhance the mechanical properties of rubber compounds is commonly known. Fillers are used both to reduce the cost of production and to strengthen the rubber [4]. A range of fillers, such as petroleum-based carbon black and silica, has been utilized to enhance the mechanical, thermal, and electromagnetic characteristics of rubber. However, prices of carbon black are dependent on petrol prices and also it causes substantial environmental harm [5]. On the other hand, the use of silica brings higher costs and it leads to restrictions in some formulations [6].

Recent studies have focused on alternative fillers such as rice husk, wood ash, regenerated (recycled) rubber [7], wollastonite and mica [8], activated calcium silicate [1]. Mica has

* Corresponding author, e-mail: sabanbulbul42@hotmail.com

been being applied as a filler in the plastics and rubber industry since it has some advantages such as low cost, availability, good heat, and chemical and electrical resistances [9]. On the surfaces of micas, there is a silica group (SiO_4) which dehydrates with water to produce a saline group ($-\text{SiOH}$). It is acidic and reacts with amines and alcohol to form a carbocyclic acidic group. This reaction has a direct impact on the vulcanization systems of rubber product properties because the viscosity of the filler decreases and its wettability increases, which also causes the filler particles to diffuse through the elastomer chains [10, 11].

Crosslink density is one of the most important properties in the characterization of rubber compounds. Crosslinked rubber is made up of monosulfide, disulfide, and polysulfide linkages that are formed by sulfur-based chemical reactions between the rubber's main chains [12]. The density of these linkages directly impacts the chemical, physical and mechanical properties [13]. Boopasiri *et al.* [14] investigated the reinforcing effect of pyrolyzed waste coffee ground in SBR. As the addition of the filler (pyrolyzed waste coffee ground) increased, hardness and tensile strength increased due to the increase in crosslink density but elongation % values decreased.

In some other studies, scholars determined that as the crosslink density increased with the addition of filler, the tensile strength, tear strength, and hardness values increased between 58% and 500%, 38% and 360, and 4.47% and 22%, respectively [15-19]. In addition, it was stated that excessive crosslink formation reduced the elongation % due to the more brittle structure of the compounds [20].

The purpose of this study was to investigate the effect of crosslink density on some physical, mechanical, chemical, and morphological properties of the mica powder added SBR compounds.

Material and method

Materials

The SBR 1502 was bought from LBS Composition and Laboratory Technologies LLC (Turkey). The styrene content was 23.5 % and the Mooney viscosity was 50 (ML1+4, 100 °C). The N-cyclohexyl-2-benzothiazole sulfonamide (CZ) (melting point 98 °C, molecular weight. 264.40 g/mol) was obtained from Bayer Chemical Medical Science Trade and Industry LLC, Turkey. For vulcanization, active zinc and sulfur were supplied from Defne Kaucuk Company. Mica powder, carbon black and calcite were purchased from Kaltun Madencilik Co. Ltd., Pektim Inc., and Mikrokalkalsit Co. Ltd. Turkey, respectively. The other materials (*i.e.*, plasticizer, homogenizer, process-facilitating chemicals, and crosslink makers) used in this study were supplied from Bayer Chemical Medical Science Trade and Industry LLC, Turkey. In addition, the solvents (acetone and chloroform) used in the swelling test were supplied from Bereket Chemical Co. Ltd., Turkey. When the chemical analysis results of mica powder are examined, it is seen that there is a high content of SiO_2 and Al_2O_3 in it. Besides, the existence of metal oxides such as Fe_2O_3 and MgO also attracts attention. The use of metal oxides in compounds with rubber plays an active role in the transport of accelerator radicals to the main chain. Although the main activator for exhibiting this activity is ZnO , all metal oxides perform tasks similar to ZnO . The chemical analysis of the mica powder used in the study is shown in tab. 1.

Table 1. Chemical analysis of mica powder used in the study

Content	SiO_2	Al_2O_3	Fe_2O_3	MgO	TiO_2	K_2O	Other
wt. %	49.97	27.30	5.53	1.43	0.79	9.85	5.13

Preparation of the SBR composites

The SBR rubber matrix (MP0, MP5, MP10, and MP20) filling were prepared by the mixture procedure according to our previous study [8]. Composition and filler density of compounds were given in tab. 2.

Table 2. Composition of rubber compounds

Components	Mix (phr)				Density values [gcm ⁻³]
	G	MP5	MP10	MP20	
SBR-1502	100	100	100	100	0.94
ZnO	3	3	3	3	–
Stearic acid	2	2	2	2	–
MBT	5.5	5.5	5.5	5.5	–
CZ	1	1	1	1	–
DPG	1	1	1	1	–
Process oil	10	10	10	10	–
Sulphur	2.5	2.5	2.5	2.5	–
Carbon black	30	30	30	30	1.8
Calcite	20	20	20	20	2.71
Mica powder	00	5	10	20	2.9

Characterization

By cutting in accordance with the standards of tests to be applied to compounds, samples were obtained. The samples were conditioned for 24 hours in an environment with 50% relative humidity at a temperature of ± 25 °C before testing, and then they were subjected to testing. The tensile test applied to the samples was performed on an AGS-X Floor model Shimadzu brand device at a speed of 10 mm/s based on the ISO 37 standard [21]. The tearing test was also performed in accordance with ISO 34 [22] norm on the same device. The density measurements of the samples were performed using helium, He, gas in the AccuPyc II 1340 (micromeritics, norcross, and GA) gas pycnometer. Hardness measurements of the samples were also carried out based on the ISO 868 [23] standard by a durometer (AFFRI 3001, AFFRI, Wood Dale, IL) performing measurements on the Shore A-type. The crosslink densities of the samples were calculated with the Flory-Rehner equilibrium volume swelling equations [24].

In the study, in order to see the distribution of mica powder used as an additional filler in the matrix material and to understand its behavior in this matrix material, the tensile fracture surfaces of the samples after tensile testing were examined with Zeiss ultra/plus SEM. The tensile fracture surfaces of the samples were coated with pure gold with a thickness of 5 nm [25]. The experimental samples were analyzed by the energy distribution spectroscopy (EDS) analysis method.

To determine the changes in the chemical structure of the compounds with rubber, a Fourier transform infrared spectrophotometer (FTIR) (Thermo Fisher Scientific brand Nicolet™ iS10 model) was used, and the measurements were made in the range of 400-4000 cm⁻¹ spectrum.

Results and discussions

Crosslink density and FTIR spectra of compounds

The crosslink densities of the compounds are given in fig. 1(a). depending on the amount of mica powder added. It was observed that as the amount of mica powder added to the SBR-matrix rubber compounds increased, the crosslink density also systematically increased. Accordingly, the crosslink density calculated after the swelling test in the MP0 compound was $41.89 \cdot 10^6 \text{ mol/cm}^3$. When fig. 1(a). was examined, it was seen that the highest crosslinking density ($51.92 \cdot 10^6 \text{ mol/cm}^3$) was in the MP20 compound, which was a compound containing 20% mica powder. Accordingly, it was observed that with the addition of mica powder by 20%, the crosslinking density increased by 23.84% compared to the MP0 compound. The crosslink densities of MP5 and MP10 compounds after the swelling test were calculated as $48.38 \cdot 10^6 \text{ mol/cm}^3$ and $49.71 \cdot 10^6 \text{ mol/cm}^3$, respectively. When crosslinking densities of the mica powder-added compounds are compared among themselves, it can be said that there is no significant crosslink density increase. On the other hand, compared to the MP0 compound, the increase observed in other compounds is due to the chemical content of mica powder. The chemical composition of mica powder includes MgO at a rate of 1.43%, SiO₂ at a rate of 49.97%, and Al₂O₃ at a rate of 27.30%. It is known that as a result of the addition of these additives to rubber compounds, the crosslink density increases [26, 27]. Metal oxides play an active role in the transport of accelerator radicals to the rubber main chain. In addition, metal oxides provide additional post-cure time for compounds while the temperature is decreasing slower after vulcanization. This helps to increase the crosslink density in both conditions. Although ZnO is used to exhibit this task, it is known that all metal oxides perform tasks similar to ZnO [28, 29]. Murniati *et al.* [30] were found that as a result of adding the Si/zeolite/Fe₃O₄ mixture to the NR, the crosslink density and tensile strength increased significantly.

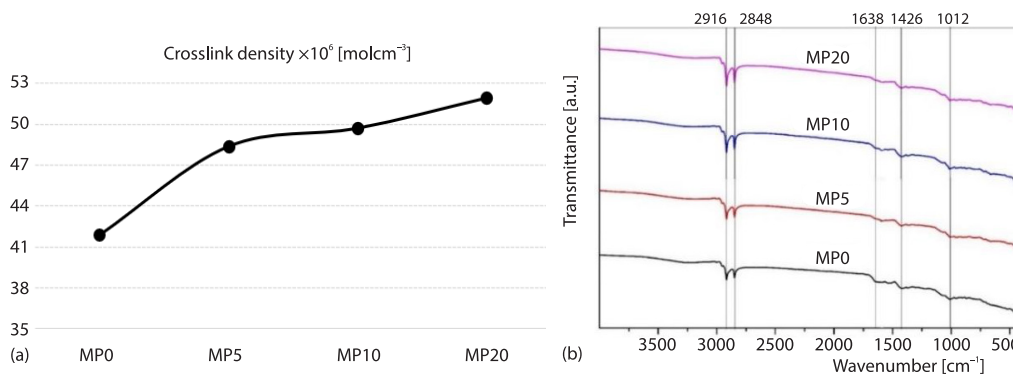


Figure 1. (a) Crosslink densities and (b) FTIR spectra of the MP-filled compounds

The FTIR spectra of the MP-filled compounds were given in fig. 1(b). The peak at 3023 cm^{-1} observed in the FTIR spectrum of vulcanized SBR is thought to be due to the aromatic C-H stretch. It has been stated that the bands at 2916 and 2846 cm^{-1} may be caused by aliphatic C-H stretching occurred due to the presence of the vinyl group in the SBR chain [31]. In addition, the stretching vibration of the C = C bond at 1638 cm^{-1} is observed. It is thought that the bands at 1426 cm^{-1} may occur due to asymmetric and symmetrical bending vibrations of the C-H bond, and the band at 1012 cm^{-1} may occur due to the stretching vibration of the C-O bond. The results obtained by Gunasekaran *et al.* [32] and Acar *et al.* [33] are in parallel with the results of this study. On the other hand, it is observed that the peak intensity of the

C = C bond observed at 1638 cm^{-1} decreases with an increase in the ratio of added mica powder in the compounds. In a study, it was found that pure mica powder had a C-H bond at around 2900 cm^{-1} [34]. Therefore, it is believed that the presence of the C-H bond in the mica powder may be the reason for the increase in the peak intensity of the C-H bonds at 2916 cm^{-1} and 2846 cm^{-1} .

Physical-mechanical properties

The type, ratio, crosslink degrees, crosslink density, forms of filler materials, and their distribution in compounds play an active role in determining the mechanical properties of compounds with rubber. Therefore, there may be many reasons that affect the mechanical properties. In particular, the effect of crosslink density on mechanical properties was evaluated in this study. The tensile-strength diagram of SBR matrix compounds with and without MP addition is given in fig. 2. Four different compounds were produced in such a way that the filler ratios were 0-5-10-20%. Three samples were tested for each percentage of filler, and the arithmetic mean value was taken as the peak value of the tensile strength.

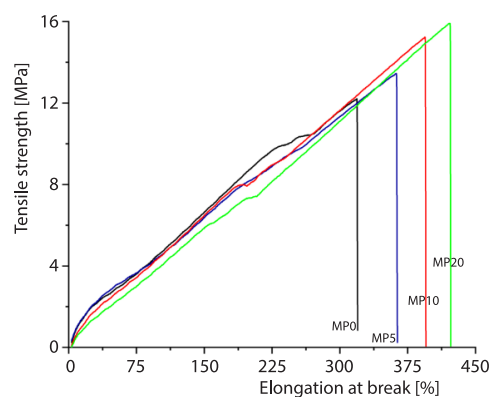


Figure 2. Tensile-Strength diagram of the compounds

Figure 2 shows that as the proportion of mica powder added to the compounds increases, the tensile strength and the percentage unit elongation are systematically increased. The compound with the highest tensile strength is the MP20 compound (15.90 MPa), to which mica powder is added at a rate of 20%. The tensile strength of the compound (MP0), which does not contain mica powder, is 12.17 MPa. When the tensile strengths of the compounds were compared, it was observed that the tensile strength of the MP20 compound increased by 30.65% compared to MP0. The tensile strengths of MP5 and MP10 were 13.41 MPa and 15.2 MPa, respectively. The percentage unit elongation of the compounds also increased with the increase of MP filler in the compounds. While the percentage unit elongation of the MP0 compound was 319.71%, it was observed that the percentage unit elongation of the 20% mica powder-added compound (MP20) increased to 422.85%. The percentage unit elongations of the MP5 and MP10 compounds were 363.56% and 395.28%, respectively. The mechanism of stress transfer by filler particles dispersed in the rubber matrix is one of the main factors determining the mechanical properties of the rubber compound. Therefore, the type and amount of filler added and the ability of a filler to form its own web in the rubber matrix are important [35]. Thus, the increase in the crosslink density also increased the tensile strengths of the compounds and their percentage unit elongation. In some studies conducted by scholars, it has been noted that the increase in the crosslink density will lead to improvement in the tensile strength of the compounds and their percentage unit elongation [36, 37]. In addition, the other reason for the increase in the amounts of tensile strength and percentage unit elongation of compounds is related to the chemical structure of mica powder. When the chemical composition of MP filler is examined, it is seen that there are SiO_2 (45.70%) and Al_2O_3 (33.10%) in it. Some other studies related to silica and alumina matrix added to rubber compounds have reported that these additives increase filler-rubber interaction and filler-filler interaction, and therefore, there will be

better tensile strength and percentage unit elongation [38, 39]. The results obtained in this study and the results revealed in the literature show similarities.

When the tearing test results applied to compounds were investigated, it was shown that the tearing strength increased as the MP ratio added to the compounds increased, fig. 3(a).

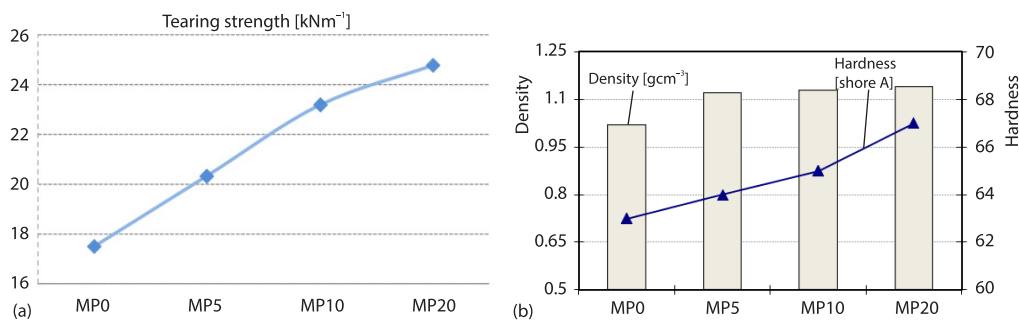


Figure 3. (a) Tearing strengths and (b) density and hardness values of MP-filled compounds

It is a known fact that the tensile strength, elongation percent, and tearing strength properties of compounds will increase with the increase in crosslink density [40, 41]. When the data obtained from the tearing test results of the compounds was analyzed, it was observed that the tearing strength of the compound with the highest crosslink density (MP20) was 24.75 kN/m, while the tearing strength of the MP0 compound with the lowest crosslink density was 17.46 kN/m. The same situation applies to other produced compounds. The tearing strength of the MP5 compound was 20.31 kN/m, and the tearing strength of the MP10 compound was 23.19 kN/m. When these results were referenced, it was seen that there was a directly proportional relationship between the crosslink density and the tearing strength. It is believed that the reason for this increase is the dense SiO₂ substance contained in the mica powder. There are literature studies indicating that silica and its derivatives increase tearing resistance in rubber matrix compounds. In their studies conducted by adding different proportions of silica to the SBR matrix, Zhong *et al.* [42, 43] reported that the tearing strength of compounds increased as the silica content increased. Thus, it can be said that the tearing test results obtained in the previous studies show similarities with the results of this study.

The density and hardness test results performed for determining the physical properties of the mica powder-added compounds are given in fig. 3(b). When the test results were investigated, the hardness and density of the compounds increase depending on the mica powder content. The reason for this increase in density is that the density of mica powder is greater than that of carbon black and the rubber matrix. As the addition rate of mica powder in the compounds increased, the densities of the compounds increased accordingly. But this increase was not so much, there was a difference of 0.12 g/cm³ between the MP20 compound with the highest density (1.14 g/cm³) and the MP0 compound with the lowest density (1.02 g/cm³). In rubber compounds, the target density and hardness values vary by the environment in which the material will be used (*e.g.*, temperature, humidity, sun rays, *etc.*) and the conditions. For this reason, there is no specific standard, but usually, polymer manufacturers do not want the density to be more than 1.2 g/cm³. Therefore, the densities of the compounds produced are lower than the specified value. Furtado *et al.* were reported that mica powder increased the densities of compounds with SBR matrix [44].

The other parameter considered in the evaluation of new compounds produced as a result of the addition of MP is hardness. When the results presented in the hardness graph are

examined, it is seen that as the ratio of mica powder added to the compounds increases, their hardness also increases. The hardness value of the compound without MP addition was 63 Shore A, while the hardness values of the compounds with MP addition were measured as 64 Shore A (MP5), 65 Shore A (MP10), and 66 Shore A (MP20), respectively. It is known that metal oxides and carbide oxides are much harder than polymer materials. An important difference of the MP filler used in the study was the fact that it was composed of SiO_2 , Al_2O_3 , and Fe_2O_3 . Therefore, it is believed that the increase in the hardness value is due to these components. Maciejewska *et al.* [35] were found that the mechanical properties and hardness of compounds would increase as the crosslink density increased. Accordingly, it is thought that another reason for the increase in hardness is the increase in crosslink densities in the compounds.

Morphological analysis

In order to find out whether the speculations about the interpretations related to the mechanical test results were valid, SEM images and EDS of the tensile fracture surfaces of the compounds were examined using a x500 magnification rate as shown in fig. 4. When fig. 4(a) was examined, it was observed that the distribution of the rubber filler containing carbon black was excellent, calcite filler, on the other hand, had different sizes, did not distribute homogeneously, and the tensile began from these regions. Thus, the lowest tensile strength was observed in the MP0 compound without the addition of mica powder.

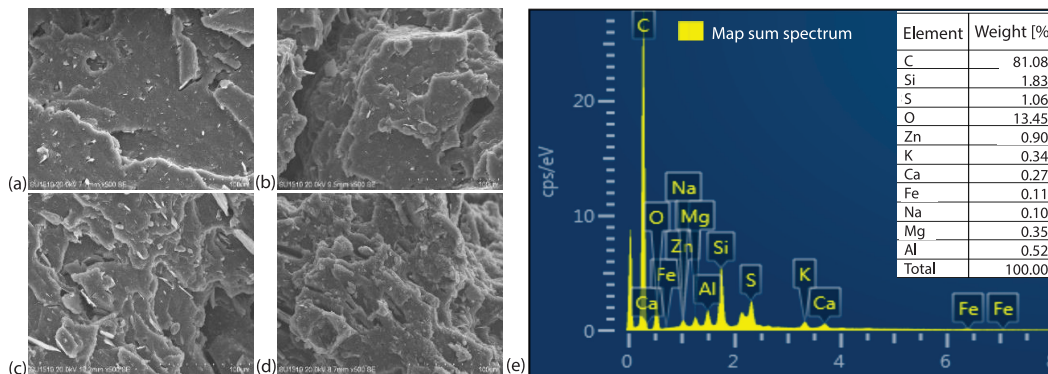


Figure 4. The SEM images of tensile fracture surfaces of the novel and reference compounds; (a) MP 0, (b) MP 5, (c) MP 10, (d) MP 20, and (e) EDS scanning spectral results

It was observed that as the amount of MP added to the compounds increased, the MP filling on the tensile fracture surface became more prominent, the grain sizes changed, and the filler distribution at the interface deteriorated as expected. It is seen that there are spherical micro-pores and agglomerations on the tensile fracture surface of the compound containing mica powder, and they multiply as the ratio of mica powder increases figs. 4(b)-4(d). Also, in some areas of the tensile fracture surfaces of compounds containing mica powder, grooved lines in lesser density are observed. The increase in tensile and tearing strength is largely due to the interaction of the MP filler with rubber and the increase in the crosslink density. It is believed that this situation causes the improvement of the surface area of the MP-added compounds and the increase in the tensile strength. The fact that a more porous internal structure was obtained due to the increase in the amount of filler allowed the bonds to move towards the direction of tensile during the tensile test, and it caused the compounds to become more flexible [45].

Conclusion

In this study, the effect of MP reinforcement on the mechanical properties of SBR-matrix rubber compounds was examined, and the obtained test results were tried to be associated with the crosslink densities of the compounds. After the swelling test, it was determined that the MP filler added to the compounds increased the crosslink density. It was observed that compounds containing metal oxide help the ZnO additive and provide a higher strength increase, a higher crosslink degree and increase the efficiency of the crosslinking system. The change of physical and mechanical properties increased depending on the MP ratio. In this study, it was revealed that mineral-based fillers could be used together with petroleum-derived additives such as carbon black. In addition a porous internal structure, improvement of surface area, and increase in volume, it also improved some physio-mechanical properties such as tensile-tearing strength and high ductility. Finally, it was revealed that mica powder in the specified proportions could be used as a filler in SBR-matrix compounds.

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