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THE EFFECT OF WOOD ASH ON THE MECHANICAL PROPERTIES OF RUBBER COMPOUNDS

VPLIV LESNEGA PEPELA NA MEHANSKE LASTNOSTI GUMIJASTIH ZMESI

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In this study, the effect of adding wood ash (oak tree) on the mechanical properties (tensile, abrasion, tearing, elongation %, etc.) of NR/SBR-type elastomer materials was investigated. Five different compounds were prepared by adding 16 %, 32 %, 48 % and 64 % wood ash to the filler and additive materials in the general composition (G). The mechanical test results and production costs of the new compounds composed were compared between themselves and with the G compound. The fracture surfaces of the compounds as well as the shape and morphological distribution of the fillers in the rubber matrix were examined using a Scanning Electron Microscope (SEM). As a result of the experimental studies, it was found that the new compounds composed had an increased tensile and tearing strength, abrasion amounts, bending capability, unit elongation amounts and densities in comparison to the G compound. A decrease was observed in the hardness values and material costs (production cost) of the compounds.

Keywords: compounds, mechanical properties, wood ash

Avtorji opisujejo raziskavo vpliva dodatka lesnega pepela (hrasta) na mehanske lastnosti (natezne lastnosti, abrazija, trganje, raztezek v %, itd.) elastomerov tipa NR/SBR. Pripravili so pet različnih vrst zmesi z dodatkom (16, 32, 48 in 64) *w*/% lesnega pepela, ki so ga uporabili kot polnilni in dodajni material k osnovni sestavi (G; angl.: General). Rezultate mehanskih preizkusov in stroške izdelave novih zmesi so primerjali med seboj in z zmesjo osnovne sestave G. Prelomne površine zmesi, kakor tudi morfološko porazdelitev polnila v matrici iz gume so opazovali in analizirali s pomočjo vrstičnega elektronskega mikroskopa (SEM). Na osnovi analize eksperimentalnih rezultatov avtorji ugotavljajo, da imajo izdelane nove zmesi povišano natezno in strižno trdnost, večjo odpornost proti abraziji in sposobnost upogibanja ter enovit raztezek v primerjavi z gumo osnovne sestave G. Ugotavljajo tudi, da se je zmanjšala trdota zmesi ter da so se znižali stroški njihove izdelave.

Ključne besede: gumijaste zmesi, mehanske lastnosti, lesni pepel

1 INTRODUCTION

Natural rubber (NR) has superior mechanical properties and excellent physical properties. Styrene Butadiene Rubber (SBR) is a synthetic rubber that resembles natural rubber in terms of its physical properties. When combined with natural rubber, they enhance the physical and chemical properties as well as mechanical properties.

NR and SBR are used in many fields, especially in shoe soles. Blending natural and synthetic rubber is not a new idea. Studies in recent years have shown that binary compounds have come to the forefront in providing the desired results with the compounded rubber using the appropriate material and methodology.[1] Natural and synthetic rubber are not used alone, except in special circumstances. In practice, these rubbers may not meet the expected specifications when used alone. Rubber compounds are frequently used in the rubber industry to obtain compounds with lower costs as well as to improve processability and the desired physical properties.[2] The use of two different rubber materials in the compounds could present features that are not possible with respect to uniform rubber used compounds.[3] In recent years, the increase in the demand to improve environmental awareness and material properties has led to many studies.[4] Factors such as global warming, raw-material problems and environmental pollution have been influential in the use of renewable resources as backfilling materials for investigated and recyclable materials. The addition of particle-reinforced fillers to rubber materials is generally intended to produce the desired commercial compounds.[5] According to literature studies, glass sphere, rice husk, wollastonite, mica powder, gum, carbon black, phosphate, nanoclay, silica, calcite and nano calcium carbonate, etc. were used by adding different fillers to rubber compounds.[6–17] Carbon black is the most common filler material used to impart both plastic properties to compounds and to improve their physico-chemical properties.[18,19] However, due to unstable oil prices, it was recently used both as a filler in mineral-based elastomer materials and as a filler in non-recyclable materials.[20–22]

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Thus, oak wood ash, one of the most important materials among the materials that have not been recycled contains 31.35 % calcium (Ca), 10.25 % potassium (K_2O), 7.57 % magnesium (Mg), 1.21 % sulfur (S) and a small ratio of zinc (Zn). It is also a known fact that these materials are used as fillers and additives in the polymer sector.[23,24] According to a study of Şaban et al. carried out in 2014, they proved that wood ash could be used as an effective filler material in polymer materials.[25]

It should be noted that our natural resources are not infinite, that every source of production is reduced and that our natural resources are completely lost if they are carelessly used. The aim of this study is to show that wood ash in the form of garbage that leads to environmental pollution is a recyclable material that goes to waste and to shed light on similar studies.

2 EXPERIMENTAL PART

2.1 Materials

NR RRS3 natural rubber and SBR 1502 styrene-butadiene rubber were obtained from Defne Kauçuk Import and Export Co. Ltd., Turkey. Silica (SiO_2), Kalsit ($CaCO_3$) and wood ash were purchased from Petkim Inc., Miner Madencilik Co. Ltd. and Kafkas Etliekmek Co. Ltd. Turkey, respectively. The other rubber compounding ingredients used in the study were obtained from Defne Kauçuk Import and Export Co. Ltd., Turkey. Basic technical properties of the fillers are presented in **Table 1.**

Table 1: General technical properties for the rubbers and fillers used in the study.

Materials	Commercial Names	Density (g/cm^3)	Particle Size (μm)
Natural Rubber	RSS 3	0.93	–
Styrene Butadien Rubber	SBR 1502	0.94	–
Silica (SiO_2)	Egesil BS 20 A	2	20
Kalsit ($CaCO_3$)	Mikronize Kalsit 0-40	2.6	25–40
Wood Ash	–	2.3	20 (in diameter)

2.2 Methods

2.2.1 Rubber compounding

The NR-SBR rubbers and fillers were compunded in a laboratory-type banbury (Farrell brand) at 80 °C and 60 min^{-1} for 10 min. After blending, the blend was left to rest for 24 h. The dough was then blended in a two-roll open mixer at 80 °C and at a speed of 40 min^{-1} for 5 min, followed by the addition of additives such as softeners, activators, vulcanization accelerators and sulfur. This stirring was continued for 3 min. Compound formulations are given in **Table 2.**

Table 2: Composition of rubber compounds.

Compounds	Sample Codes				
	G	WA1	WA2	WA3	WA4
	phr				
Natural Rubber (NR-RSS3)	50	50	50	50	50
Styrene Butadiene Rubber (SBR 1502)	50	50	50	50	50
Silica	33.4	33.4	33.4	33.4	33.4
Calcite	26.6	26.6	26.6	26.6	26.6
Wood Ash	-	16	32	48	64
Zinc Oxide	3	3	3	3	3
Sulfur	1.5	1.5	1.5	1.5	1.5
Stearic Acid	1.5	1.5	1.5	1.5	1.5
PEG 4000[a]	2	2	2	2	2
DM[b]	1	1	1	1	1
CZ[c]	1	1	1	1	1
DPG[d]	1	1	1	1	1

[a]Polyethylene glycol, [b]Dibenzothiazole disulphide, [c]N-Cyclohexyl-2-benzothiazole sulphonamide, [d]Diphenyl guanidine

2.2.2 Vulcanization of rubber compounds

The rubber compounds were cut into small pieces and placed into a compressible mold, specially designed in accordance with test standards, and vulcanized in a Hidrosan brand hot press, under a pressure of 160 MPa at 160 °C. The vulcanization process continued for 5 min, which is a pre-determined optimum time for all compounds. All these compounds used in the experimental studies were prepared under the same conditions.

2.2.3 Physico-mechanical characterization

All tests were performed after the compounds were cut into the standard dimensions according to the related test standards and were kept in an environment having 50 % relative humidity at 25±2 °C for 24 h. The tensile test was performed (by using a Tinius H25KS tensile device) with 10 mm/s according to ISO 37. During the tensile test, the tensile strengths and elongation at the breaking values of the samples were measured. Tear tests were made according to ISO 34-1 by using the same equipment as in the tensile testing. The hardness measurement of the vulcanizates was conducted by using a Shore A durometer (Commerciale, AFFRI 3001 model) according to ISO 868. The densities of the compounds were measured in line with Archimedes' principle (ISO 2781). An abrasion test (with a Pegasil, CTC model device) was applied in line with the ISO 4649 standard. In the abrasion tests, the standard test sample placed on the drum completed 84 rotations in a 40-meter shifting distance under 10 N perpendicular forces. A dimension stability test was performed in accordance with the EN 12770 standard norm. Additionally, a stretching (fatigue) test was applied to the compounds in line with the Ross-Flex method (with a SATRA, STM 141/F model device) and the ISO 5423 standard.

2.2.4 Scanning electron microscopy (SEM) analysis

SEM images (with a Zeiss brand, Ultra/Plus model) of the samples' fracture surfaces after the tensile test were observed. The fracture surfaces of the samples were coated with 5-nm thick 100 % gold in order to prevent electrostatic loading and increase the conductivity.[17,25,26] A Quorum, Q 150R ES model device was used for coating. During the examination of the microstructure the preferred working voltage of the microscope was 20 kV. *The energy-dispersive x-ray spectroscopy* (EDS) analysis method was used to distinguish the components in the sample.

3 RESULTS AND DISCUSSION

In this study, wood-ash-added NR/SBR rubber compounds were examined in terms of their physical, mechanical and morphological properties.

3.1 Physical and mechanical properties

The chemical and physical properties of rubber compounds are directly affected by factors such as chemical composition, particle sizes, physical properties, production processes, ambient temperatures and the fillers used in the rubber compounds. However, the rubber filler ratios of the compounds are also one of the most important factors affecting the rubber compounds.

3.1.1 Dimension stability and hardness

The wood ash added to the G compound firstly decreased the hardness of the compound and then the hardness of the compounds increased with an increasing ratio of wood ash in the compounds (**Figure 1**). The hardness of the G compound was measured as 65 Shore A. This value is the same as the hardness value of the WA4 compound added with 64 % wood ash. The hardness of the WA1 compound added with 16 % wood ash is 60 Shore A. When compared to the G compound; WA1, WA2 and WA3 compounds, except the WA4 compound, were found to have a decreased hardness. First of all, the decrease of the hardness of the rubber compounds and an increase of the hardness again with the increasing fill amount result from the rubber filler ratio. It was suggested by Savran that that the rubber fill ratio should be maximum 100 %.[27,28] In this study, except for the WA1 compound, there is no other compound providing this condition. Therefore, the effect on the hardness of wood ash in rubber compounds is not exactly reflected (**Figure 1**).

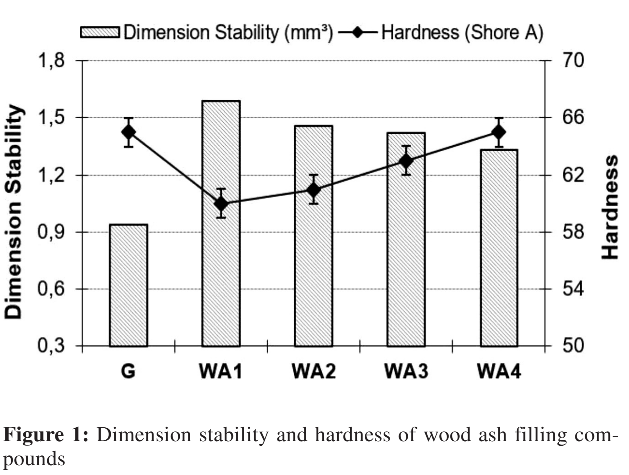

Figure 1: Dimension stability and hardness of wood ash filling compounds

The change in dimension of the G compound against the temperature is 0.94 mm^3. The maximum dimension change in the new compounds composed was found in the 16 % wood ash added WA1 compound (1.59 mm^3). It was seen that as the wood ash ratio in the compounds increased, the dimensional changes decreased. The best results in the new compounds were obtained in the WA4 compound. Compared with the G compound, the dimension change of the WA4 compound was increased by 29.8 %. In the searchable literature review, no data were found about a comparison with regard to the dimensional stability of elastomer materials. Therefore, in the comparison process of this study, the change in dimension stability of rubber compounds in plants that are manufacturing polymer shoe soles was taken as a measure. According to these evaluations, it is indicated that compounds with 70 Shore A and a higher hardness should have a dimension change of 1 %, and those with a hardness lower than 70 Shore A should have a maximum dimension change of 3 %. When looking at **Figure 1**, it can be seen that the hardness of the composed compounds is softer than 70 Shore A and at the same time the test results obtained meet this standard.

3.1.2 Density and material cost analysis

The desired maximum density value of soles, according to the ISO 4649 standard, is 1.5 g/cm^3. **Figure 2** shows that the samples' densities are below the upper limit value specified in the standard. As the supplement ratio of wood ash filler increased, the densities of composites expectedly increased as well. The density of the compound G is 1.2 g/cm^3. For the new compounds, the highest density belongs to the WA4 compound. This result is 15.83 % higher than the density of the G compound and 7.91 % lower than the ISO 2781 standard. The densities of the WA1, WA2 and WA3 compounds

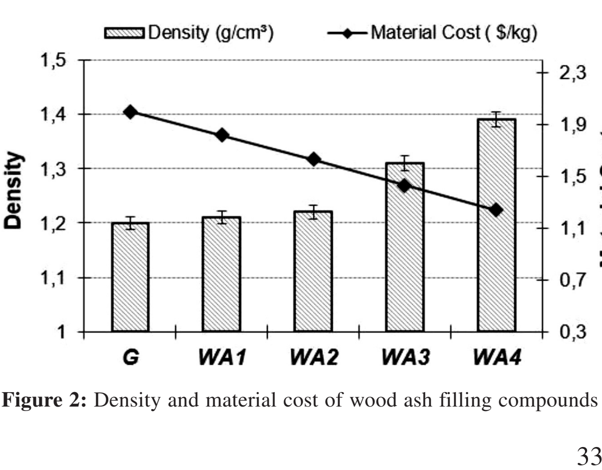

Figure 2: Density and material cost of wood ash filling compounds

are below the value specified in the ISO 2781 standard. The density of wood ash (2.3 g/cm^3) used in the new compounds is higher than silica, SBR and NR. In the new compounds, the ratio of rubber in the compounds and silica reduced as the wood ash ratio increased.

It was observed that the cost of the compounds decreased as the ratio of wood ash added to compound G increased (**Figure 2**). The WA4 compound with 64 % wood ash reduced the production cost by about 40 % compared to the G compound. The WA1 compound reduced the production costs by 10 %. WA2 and WA3 decreased by 20 % and 30 %, respectively. It has been determined that the absence of any commercial value of the wood ash will have the effect of reducing the production of such compounds and the resulting costs of production.

3.1.3 Abrasion and elongation

The abrasion resistance depends on factors such as the surface roughness, size of the material, temperature, humidity, atmosphere and pressure.[29] These factors must be taken into account when preparing the compounds. When the relative volume losses given in **Figure 3** are examined, it is seen that the calculated relative volume losses increase with an increase of the wood ash added to the compounds. According to the ISO 4649 standard, the maximum volume loss of rubber shoe soles should be 250 mm^3. The least relative volume loss in newly composed compounds was seen in WA1. When the rubber amount was compared with the filler amounts of the new compounds used in the experimental studies, it was found to be more than 100 %, except for WA1. It was found that the total amount of filler in the WA3 compound was 111.3 %, compared to the used rubber, and 128.4 % in the WA4 compound, thereby increasing the relative volume loss. Micro roughening occurs on the surfaces of the samples when prepared by adding 48 %, 64 % of the filler (due to the decrease of the rubber content in the composition). As a result, the relative volume losses of the test specimens increased. In a study of Tangudom et al., it is emphasized that the decrease of the rubber ratios in the compound increases the relative volume loss.[30] The results obtained after the abrasion test show similarities with those obtained by Tangudom.

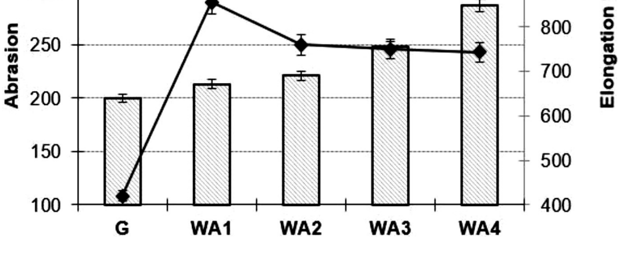

Figure 3: Abrasion and % elongation of wood-ash filling compounds

The percent elongation of the compounds after the tensile test exhibited the behavior shown in **Figure 3**. According to the ISO 5676 standard, the minimum elongation percent for shoe soles should be 200 %. When the test results of the percentage unit elongation values of compound G and wood-ash supplemented compounds were reviewed, it was seen that while the breaking elongation of compound G was 420 %, that of wood ash supplemented compounds varied between 856 % and 743 %. Among the new compounds added with wood ash, the highest unit elongation value was obtained for the WA1 compound. This increase is associated with the 1 % sulfur in the wood ash. In the study carried out by Manshaie and Sengloyluan, it was also emphasized that the amount of sulphuric cross-links was increased, thereby increasing the amount of unit elongation.[31,32] The unit-elongation values obtained from the tensile tests are supported by these previous studies. It is known that when the filler materials added to the compound are decreasing, the interaction with the rubber (intercalation), the tensile strength and the unit-elongation value will decrease.[22,33] When the new compounds were compared among themselves, it was seen that the amount of filler increased, as the rubber filler interaction decreased. For this reason, it is considered that the decrease in the amount of elongation of the new compounds is due to the rubber fill interaction.

3.1.4 Tensile and tearing strength

Factors affecting the tensile strength are the ratio of filler materials in the compound, particle size, interaction of filler materials with the compound, cross-links and production techniques.[9,27,28] According to ISO 37 standard, a tensile strength of min. 5.88 MPa is required in rubber-based compounds. The cross-link formation in the NR/SBR matrix materials is seen in the form of C-C, C-S and S-S. The tensile strength and chain lengths of these three crosslinks are different from each other. The cross-link with the shortest chain length (0.154 nm) is the C-C bond. The energy used to break the C-C bond is 85 kcal/mol, and the energy required to break the other bonds is much larger. The C-S chain length is 0.181 nm. The minimum energy required to break this bond is 64 kcal/mol. S-S crosslinks are bonds with the longest chain length. The bond size is 0.188 nm, and the energy required to break them is 57 kcal/mol.[34] The lengths of the bonds composed in the compounds directly affect the tensile strength. The tensile strength of the compounds after the tensile test showed a behavior as seen in the **Figure 4.** It was seen that the tensile strenghts of the compounds used as wood ash filling material containing Zn and S in their chemical composition increased to 16 % and 32 %, when added to the G compound, was increased, compared to G compound. It decreased when to the G compound was added 48 % and 64 % wood ash.

It is thought that the C-C bonds in the NR/SBR configuration create C-S bonds with longer chain lengths when reacting with sulfur. In a similar study about the

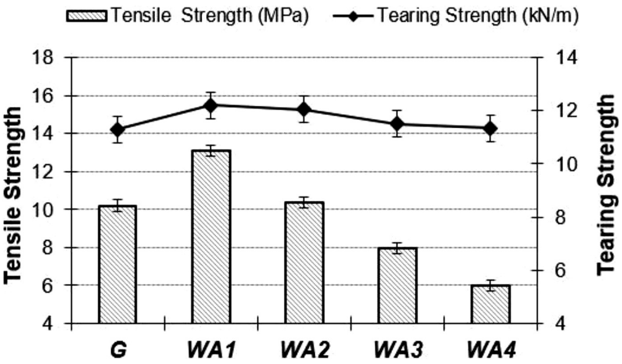

Figure 4: Tensile and tearing strength of wood ash filling compounds

NR/SBR compound, it was seen that the tensile test results were similar to the tensile strengths obtained from the C-S cross-links as a result of the reaction from C-C bonds with sulfur. This situation is similar to a study conducted by Shen.[35] Studies on SBR and NR type elastomer materials indicate that with the increase of the SiO_2 amount in the compounds, the cross-link density also increases.[30,32] The ratio of SiO_2 in the compounds decreases with the increase of the wood ash in the compounds (amount in total mass). For this reason, the tensile strength of the compounds decreases as the ratio of the wood ash used increases.

The results of the tearing strength test in this study show that the decrease in the tearing strength in 48 % and 64 % wood ash entrained compounds is due to the increase of the S content in the fillers used and the decrease of the SiO_2 filling material. It was seen that the tearing strength of the compounds in all combinations of wood ash added to the test results were higher than the tearing strength of the G compound and that with an increase of the filler ratios, the tearing strength was reduced (**Figure 4**).

The tearing strength of the WA1 compound is 12.19 kN/m and the tearing strength of the WA4 compound is 11.33 kN/m. It is seen that the tearing strength difference between these two compounds is 0.86 kN/m. The tearing strengths of the wood ash added compounds are very close to each other. According to the ISO 5676 standard, a tearing strength of at least 6 kN/m is required. Accordingly, the tearing strength of the new produced compounds increased by 100 % as to the standard ISO 5676. The WA1 compound increased by 7.87 % with respect to the tearing strength of the G compound. The tearing strength compared to the G compound increased 6.63 % in the WA2 compound, 1.76 % in the WA3 compound and 0.2 % in the WA4 compound. It is known that the tearing strength of the compounds will vary according to the filler amount and the filler type used.[1] It was seen that the wood ash added to the compound has the property of increasing the tearing strength.

3.1.5 Morphological properties

SEM images of the rupture surfaces of the *G*, WA1, WA2, WA3 *and* WA4 compounds obtained by adding 16 %, 32 %, 48 % and 64 % wood ash to the compound G after tensile tests, are shown in **Figure 5**.

When SEM images of the fracture surfaces of the compounds added with wood ash are examined, it was seen that as the wood ash fill ratio in the compounds in-

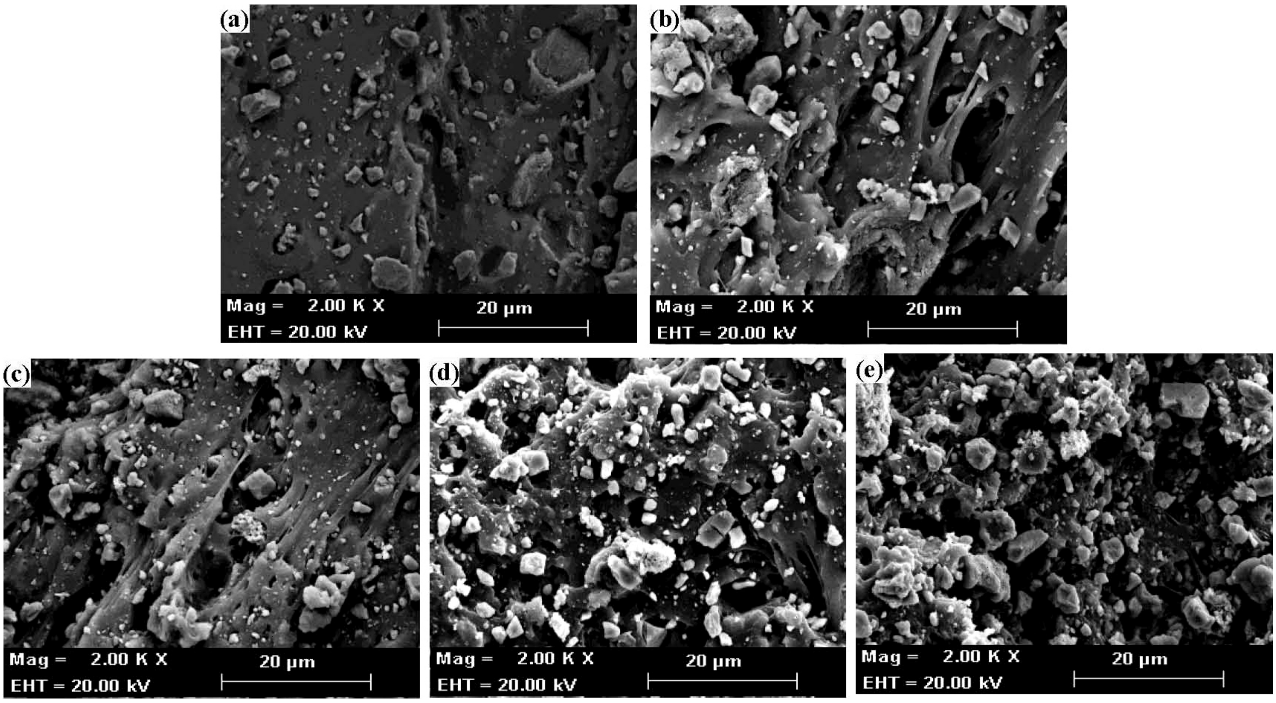

Figure 5: SEM images of tensile fracture surface of a) G, b) WA1, c) WA2, d) WA3 and e) WA4

creased, the distance between the fillers decreased. On the other hand, it was seen that the filler materials come together (aggregate) to form interfaces. These interfaces appear to be regions where the tearing of the wood-ash-added compounds begins. The formation of interfaces increases the plastic deformation. It is also confirmed by the tensile test results that the interaction between the added fillers and the rubber decreases as the fillers used in the rubber matrix form interfaces. Ismail et al., stated that, in the case of the formation of interfaces between the matrix material and the filler material, the tensile and tearing strengths will decrease.[4]

3.1.6 Bending capability

One of the properties required for elastomer materials is bending capability. The bending capabilities of elastomeric materials are important in understanding the effect of the possible cracks in the structure on the bending behavior of the material. During the bending test, the notch growth on the samples was calculated by measuring at certain intervals.

According to the ISO 5423 standard, the notch growth at 30,000 steps should be a maximum of 4 mm. The Ross-Flex bending test is normally completed at the finish of 30,000 cycles. However, in this study, the number of cycles was increased first to 100,000, then to 150,000. **Figure 6** shows the Ross-Flex bending test results.

When **Figure 6** is examined, it was seen that the largest growth in 30,000 steps occurred in the WA3 compound (1.4 mm). When the number of steps was increased, the notch growth was most abundant in the WA1 compound (1.7 mm). When the bending test results of the G compound were examined, it was seen that the resistance decreased as the number of steps increased. At 100,000 and 150,000 steps, it was observed that the bending capabilities of the compounds except for the G and WA2 compounds were more stable and there was no growth in the notch length. It can be said that the bending capabilities of the compounds are below the maximum growth rate determined in the ISO 5423 standard. According to these results, it is possible to say that the wood ashes increase the bending capability of rubber materials.

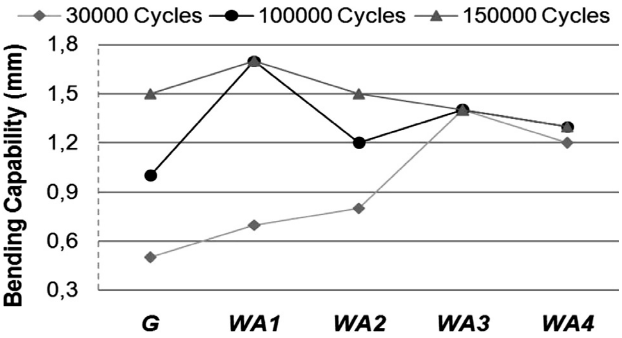

Figure 6: Bending capability of wood ash filling compounds

4 CONCLUSIONS

The wood ash added to the compound increased the density of the compounds. The reason for this is that the used wood ash density is higher than the silica, SBR and NR materials. According to the hardness test results of the G compound, the hardness test results of the new filled compounds (except WA4) were lower. The reason for this reduction is that the hardness of the composition increases as SiO_2 is used as a filling material. Depending on this situation, the SiO_2 ratio (total mass) decreased as the addition ratio of the wood ash filler in the compounds increased. This also reduces the hardness of the new filled compounds. In the WA4 compound, the total amount of filler is more than it should be (more than 100 %), causing the compound to behave in the opposite way. The use of wood ash as a filler material in rubber compounds resulted in a reduction of the abrasion resistance of the compounds. When wood ash was used in 16 % and 32 % ratios in the compounds, it was found to be much better than both in the compound G and other ratios. If it is used more than 32 %, it can be said that the tensile strength is decreased. According to the unit elongation test results of the obtained compounds, they were found to be much higher than the G compound. The tearing strengths of the compounds are also higher in compound G. According to the bending test results; after 150,000 steps, while there was a progress of 1.5 mm after the experiment in the 2 mm opened notch before the start of the experiment in the compound G, this growth was lower in the filling compounds. It was found that the wood-ash filler used increased the bending capability. The dimensional stability test results are below the maximum change of shape determined by the companies that produce shoes. In the SEM images of the tensile surfaces, the grain size was found to be an important factor for the distribution of filler materials added to the NR/SBR matrix. It was found that the chemical composition of the filler and the materials to be added to the compound was an important parameter for their mechanical properties. It was also seen that the amount of filler used in the compounds must be equal to the maximum amount of rubber, otherwise it will adversely affect properties such as abrasion, rupture, tearing, elongation and hardness.

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