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Araştırma / Research

SYNTHESIS AND CHARACTERIZATION OF ETHYLENE PROPYLENE DIENE MONOMER (EPDM) RUBBER MIXTURE

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ABSTRACT

Rubbers have a wide range of usage in polymeric materials since materials with a wide variety of mechanical properties can be obtained by varying the type or addition rates of the materials contained in the rubber in addition to the important properties such as high elasticity, good abrasion resistance, and low deformation. High-performance properties of the material are provided by cross-linking (vulcanization). Raw rubbers do not contain cross-links. In this study, it was aimed to examine the changes that may occur in the mechanical properties of the newly prepared EPDM rubber by changing the composition of the available Ethylene Propylene Diene Monomer (EPDM) rubber. For this purpose, a new rubber mixture, of which composition had been previously known and which was developed in this study, was prepared in two different Banbury mixers (tangential and intermeshing types), and both samples were subjected to mechanical and rheological analyses. The most important criteria in mechanical properties are tensile strength and elongation at break. As these properties increase, the final product becomes more durable. When tensile strength and elongation at break of the EPDM rubber prepared according to the new formulation were compared with the available EPDM rubber, a significant increase was observed in these two values in the EPDM rubber sample prepared with the new formula. In conclusion, it was shown with the characterization analyses that the newly developed modified EPDM rubber can be used in the rubber industry.

Keywords: Polymer, Banbury, Rubber, Ethylene Propylene Diene Monomer (EPDM) Rubber.

ETİLEN PROPİLEN DİEN MONOMER (EPDM) KAUÇUK KARIŞIMININ SENTEZİ VE KARAKTERİZASYONU

ÖZ

Polimerik malzemeler içerisinde geniş bir kullanım alanı olan kauçuklar; yüksek elastikiyet, iyi aşınma dayanımı, düşük deformasyon gibi önemli özelliklerinin yanı sıra, içerisine katılan malzemelerin türü veya katkı oranları değiştirilerek çok çeşitli mekanik özelliklerde malzemeler elde edilebildiğinden, oldukça geniş bir kullanım alanına sahiptirler. Malzemeye yüksek performans özellikleri çapraz bağlanma (vulkanizasyon) ile sağlanır. Ham kauçuklar (çiğ) çapraz bağları içermez. Bu çalışma kapsamında, mevcut Etilen Propilen Dien Monomer (EPDM) kauçuğunun kompozisyonu değiştirilerek yeni hazırlanan EPDM kauçuğunun mekanik özelliklerinde meydana gelebilecek değişimlerin incelenmesi amaçlanmıştır. Bu amaçla iki farklı banbury rotorunda (teğetsel ve iç içe geçmeli tip) kompozisyonu daha önceden bilinen ve bu çalışmada geliştirilen yeni bir kauçuk karışımı hazırlanmış ve her iki örneğe de mekanik ve reolojik analizler uygulanmıştır. Mekanik özellikler içerisinde en önemli kriter kopma gerilmesi ve kopma uzamasıdır. Bu özellikler ne kadar yüksek olursa nihai ürün o kadar dayanıklıdır. Mevcut EPDM kauçuğu ile, yeni formülasyona uygun hazırlanan EPDM kauçuğunun kopma gerilmesi ve kopma uzamaları kıyaslandığında yeni formülle hazırlanan örnekte bu iki değerde önemli bir artış gözlenmiştir. Sonuç olarak yeni geliştirilen modifiye EPDM kauçuğunun, kauçuk sektöründe kullanılabileceği gösterilmiş ve karakterizasyon analizleri ile ortaya konulmuştur.

Anahtar Kelimeler: Polimer, Banbury, Kauçuk, Etilen Propilen Dien Monomer (EPDM) Kauçuk.

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1. INTRODUCTION

Elasticity is a measure of the tendency of a material to return to its former shape after a force is applied to it. Perfectly elastic materials return to their former shape and size when the applied force is removed, and the flexibility of such materials is 100%. Some polymers may exhibit high elastic behaviors due to their structural properties. Natural rubber is one of the first polymers that showed high elasticity [1,2]. In the past years, different synthetic rubbers have been developed with numerous studies carried out in the field of polymers. Rubbers are vulcanizable polymers which are non-cross-linked but are cross-linkable. After vulcanization, rubbers show good mechanical strength due to their cross-linking. Ethylene propylene diene monomer (EPDM) rubber is a saturated polymer produced by the copolymerization of propylene and unsaturated diene with high resistance to ozone and high temperature compared to general purpose rubbers [3-6]. Due to its high performance, EPDM rubber is often used in the automotive industry, construction and building materials, cables and wires, as well as in coating and insulation [7].

In the rubber industry, mixing is the most critical step in the process. The purpose of mixing is to produce a product having a sufficiently dispersed composition to be easily processed in the next operation, to be cured effectively, and to provide the required properties for the final application. In this study, a Banbury mixer with tangential and intermeshing rotors was used. The Banbury mixer is a tangential or intermeshing internal mixing machine in which the double helical rotors rotate towards each other in a closed chamber shaped as two short cylinders adjacent to each other. The rotors serve as the heart of the mixers. The rotor design has a significant effect on mixer performance [8]. Mixing requires the formulation of terms such as time, speed, pressure, temperature, equipment to be used. Behera et al. successfully prepared Poly (butylene succinate-co-lactate) (PBSL) and hydroxyapatite (HAp) and poly (lactic acid) (PLA) composites using a conventional melt-mixing process with a Banbury machine [9]. Mohan et al. prepared different concentrations of nanoclay-rubber composites using a Banbury machine and showed that further studies on the abrasion and barrier properties of composites should be continued [2]. Rubber materials are defined as a mixture of some chemicals and raw rubber. Chemical components may be fillers, activators, antioxidants, accelerators, and vulcanizing agents. The specified materials affect not only the final product properties but also the behavior of the components. Many researchers attempted to develop alternative methods to improve the formulation of the EPDM dough mixture and improve the properties of the final product [10-13]. In this study, different compositions of EPDM rubber were prepared using carbon black, precipitated silica, silane, antiozonants, homogenizer, and peroxide cross-linking agent, and mixing was carried out in two different Banbury rotors (tangential and intermeshing types). After mixing, mechanical and rheological analyses were applied to both rubber samples.

2. MATERIAL AND METHODS

All materials and devices used in this study were provided from Rekor Kauçuk company. EPDM dough was prepared with a composition of 100% EPDM rubber, 25% carbon black, 10% precipitated silica, 4% binder, 5% antiozonant, 3% homogenizer, 3% peroxide cross-linking agent. During the preparation of the dough, all additives were carefully weighed and well mixed. Rubber dough was made in Banbury and laboratory press devices used in the production department. The recipe designed for the preparation of the EPDM rubber dough to be used as the base was processed in a Banbury machine shown in Figure 1, and after the dough (raw) was prepared, the hydraulic press device shown in Figure 2 was used for the vulcanization process.

The rheometer used in the study measures the vulcanization properties of the mixture and records the vulcanization curve. The rheometer precisely and quickly identifies the curing and processing properties of vulcanizable rubber compounds. It applies the oscillation voltage to the mixture under high temperature and pressure and shows the increase in torque as a result of the increase in cross-linking density (the force acting at a certain distance from the center of rotation, moment force) as a function of time. The Moving Die Rheometer (MDR) was used during the studies. Approximately 2 g of the non-vulcanized (raw) dough to be tested in the device was placed on a double conical disc under continuous high temperature and pressure. Discs close and start to oscillate (1.7 ± 0.1 Hz). It requires a force to oscillate depending on the rigidity of the rubber. This force (torque) is recorded as a function of time. When the recorded torque reaches a balance or maximum value, a completed curve is observed. The time required for the vulcanization curve is a function of the temperature and rubber properties. The explanations of the symbols indicated on the curve in Figure 3 are given below.

Figure 1. Banbury mixer

Figure 2. Laboratory hydraulic press

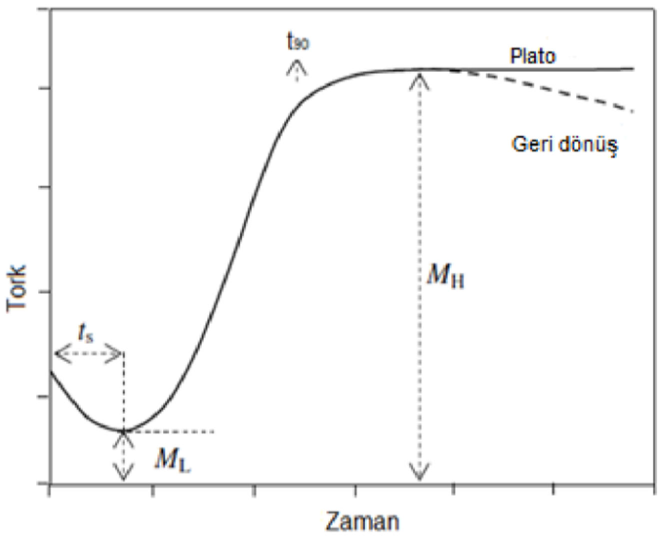

Figure 3. Vulcanization Curve [14]

t_s: Pre-vulcanization (scorch) time (min)

M_L: Minimum torque (dN.m)

M_H: Maximum torque (dN.m)

t_{90}: Time when 90% of the maximum torque is reached (min)

In this study, hardness measurements were made according to the DIN 53505 standard. Usually, two types of hardness measurement devices are used: IRHD (International Rubber Hardness Degree) and Shore type A hardness tester. A shore type A hardness meter was used in this study. At least 4 samples 6 mm in thickness from each rubber in different compositions were tested, and the mean value was taken. The tensile strength and mechanical strength tests of the samples taken from the test plates of 2 mm thick EPDM rubber prepared within the scope of this study were carried out according to the ISO 37 standard by using the Zwick Line Z2.5 testing machine. 5 samples were taken from both rubber types, and the mean values of the results were taken.

The tear strength test was performed in accordance with the ISO 34-1 standard. Angle type specimens from 2 mm thick samples taken from the EPDM rubber were processed in a Zwick Line Z2.5 testing machine. 5

samples were obtained from both rubber types, and the averages of the results were taken. The density was calculated using the density scale. First, the weight of the rubber sample in the air was measured and recorded. Then, the sample was taken into a bag of pure water, and its weight in water was measured and recorded. The density was recorded after the calculation.

3. RESULTS AND DISCUSSION

In this study, mechanical and rheological analyses of EPDM rubber mixtures were performed and compared for different types. The available EPDM dough was mixed with the tangential rotor, and the modified EPDM dough was mixed with the intermeshing rotor.
To determine the rheological properties of the EPDM dough, approximately 2 g of the test sample was tested at 190 ºC for 10 min using the rheometer, and the torque change over time was given in Figures 4 and 5.

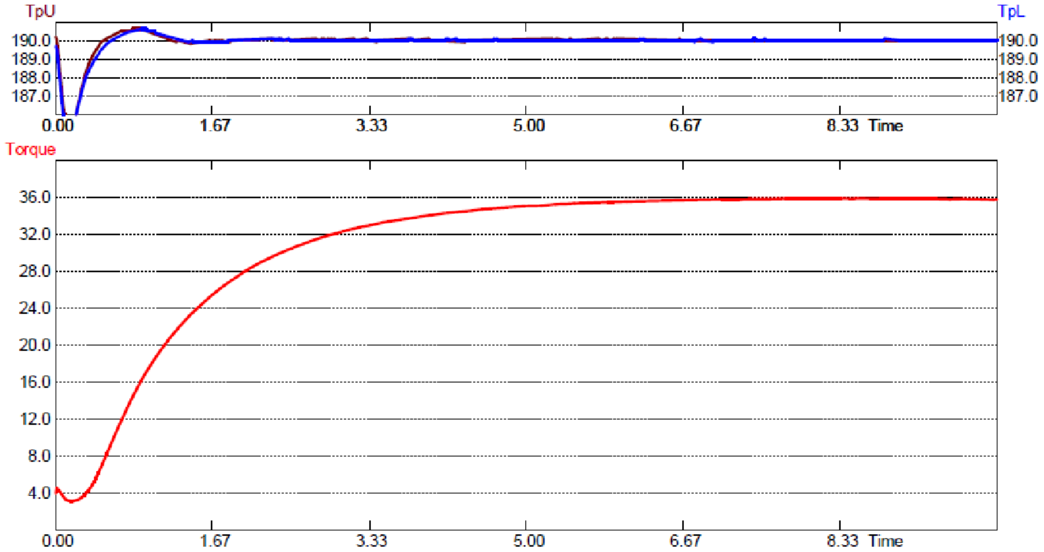

Figure 4. Test result showing the rheological properties of the available EPDM dough

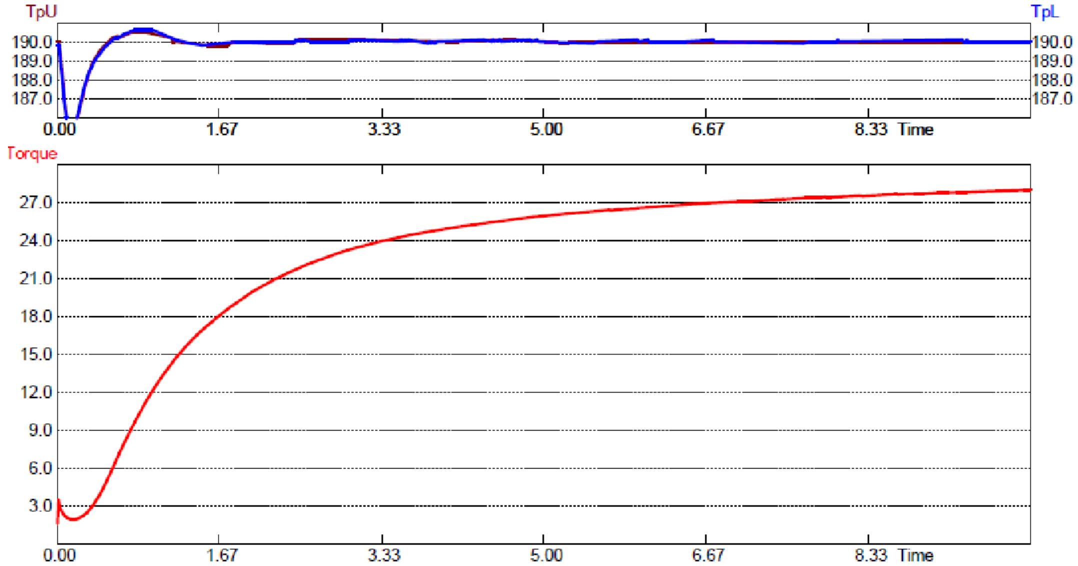

Figure 5. Test result showing the rheological properties of the modified EPDM dough

The lowest rheometer torque, M_L, the highest rheometer torque, M_H, pre-vulcanization time, t_S and curing time, t_{90} values from the test results obtained after the rheological analysis are presented in Table 1 for both samples.

Table 1. Rheological properties of the available and modified EPDM dough

Sample Name	M_L (dN.m)	M_H (dN.m)	ts (min)	t_{90} (min)	Viscosity (Pa.s)
Available EPDM	3.1	35.85	00:24	03:10	94.2
Modified EPDM	1.34	28	00:26	04:25	88.7

When the viscosity of the available EPDM and the modified EPDM is compared, the viscosity decreases when the content changes. In rheometer readings, the M_L values of the samples are directly related to the viscosity of the compound, and this value decreases similarly to viscosity in the modified EPDM. Considering the M_H value in order to interpret the vulcanization force, it is observed that this value decreases in the modified EPDM.

The mechanical properties of the EPDM dough, of which recipe was improved, were investigated. The additives and proportions of the modified EPDM dough were changed and mixed in a Banbury machine with the intermeshing rotor. The test results of the modified EPDM dough with the available EPDM dough are presented in Figures 6 and 7, and the comparison is given in Table 2. The test samples were vulcanized at 190 °C for 10 minutes in a laboratory press and results were obtained.

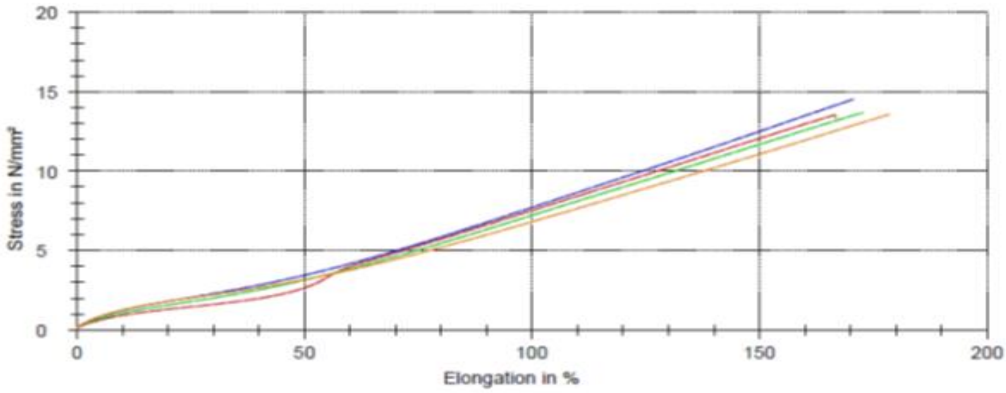

Figure 6. Test results and values of the mechanical properties of the available EPDM dough

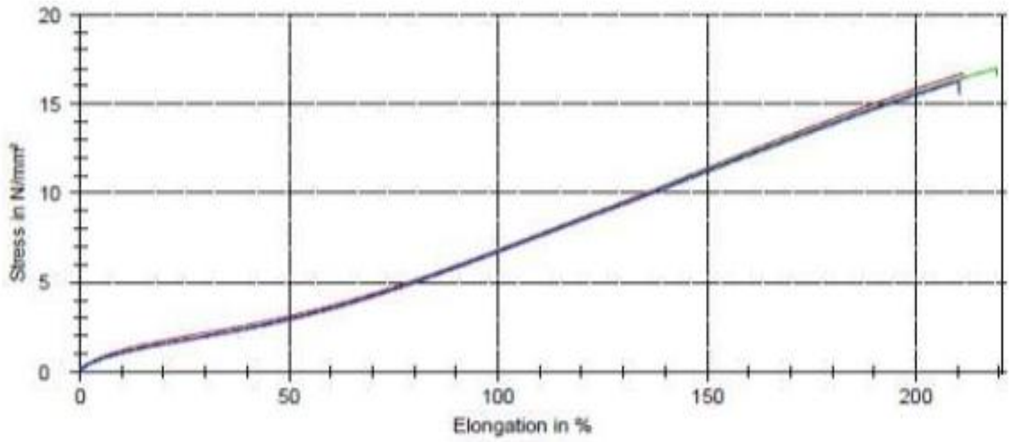

Figure 7. Test results and values of the mechanical properties of the modified EPDM dough

The desired mechanical properties of rubber dough may differ according to the usage area and intended use of rubber. For this purpose, the rheological properties and vulcanization properties of the dough prepared in different formulations were examined and vulcanized using a tangential rotor, and the hardness, elongation at break and tensile strength and tear strength of the dough were analyzed in accordance with the standards of Rekor Kauçuk company, and new dough was prepared within the scope of these standards. These properties were compared with the properties of the currently used EPDM dough. While some rubbers are desired to have high hardness values, some rubbers are desired to have low values. The most important criterion in mechanical properties is the tensile strength, and high tensile strength is desired to be achieved. The higher the tensile strength is, the more durable the final product is. There is a noticeable increase when the values of the currently used EPDM dough are compared with those of the EPDM dough prepared according to the newly developed formulation. As seen in Table 2, the tensile strength in the available EPDM is 13.8 MPa, while this value increases up to 16.6 MPa in the modified EPDM.

Table 2. Mechanical properties of the EPDM dough mixed in different rotor types

Sample Name	Hardness Shore A	Density g/cm^3	Tear kN/m	Tensile Strength σ_R (MPa)	Elongation at Break ϵ_R (%)	σ_{100} (MPa)
Available EPDM	79.8	1.15	5.59	13.8	172	7.3
Modified EPDM	78.7	1.15	4.71	16.6	214	6.7

Reduction of the hardness resulting from the additives present in the modified EPDM also led to an increase in elongation at break and tensile strength. These results showed that the modified EPDM provides better processability. Ismail et al. compared the effects of mica and talc on the curing, tensile and thermal characteristics of EPDM. Due to the stronger interfacial interaction between filler and matrix, the EPDM/mica composite showed better tensile features than the EPDM/talc composite [15]. Haisheng et al. prepared and vulcanized silica-, nanoclay-, and carbon black (CB)- filled ethylene–propylene–diene terpolymer (EPDM) mixtures. Silica filled EPDM vulcanizates, a tensile strength of 23.5 MPa were achieved, [16]. The tensile strength and elongation at break are the only properties used to evaluate the strength of a rubber compound. As a result of this study, modified EPDM composites showed better tensile properties as compared to available EPDM. The results obtained show that the modified EPDM dough can be used by Rekor Kauçuk company or a different rubber company.

4. CONCLUSIONS

A different recipe was developed from the most commonly used EPDM dough in Rekor Kauçuk company, and experimental results obtained from the changing of the rotor types during mixing showed that the mechanical properties changed.

The rubber industry serves a wide range of industries and has an important area nowadays. In today's conditions, competitive conditions are increasing, and customer satisfaction is coming to the forefront with the industrialization which is gaining momentum. Increasing raw material inputs and production costs led industrialists to find different solutions. They prefer to increase the quality without increasing the cost. The ability to intervene in the quality during the production stage is required rather than the final quality control. Rubber dough preparation, vulcanization and shaping, and final product acquiring mechanism are various and complex processes. In order to achieve the expected properties of the final product, all steps starting from the formulation should be well known, and all points that may have an effect on the properties of the final product should be taken into consideration during the process.

In conclusion, a new EPDM composition with appropriate mechanical and rheological properties was prepared. These recipes can be changed according to different demands, and detailed analysis can be made. In future studies, with different nanoclay filled rubber composites, most suitable mechanical and rheological properties EPDM dough mixture can be obtained.

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