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Research Article

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Investigation of Effect of Resin Content on the Stress Relaxation of Poly (epichlorohydrin-*co*-ethylene oxide) Based Elastomers

Poli (epiklorohidrin-*ko*-etilen oksit) Temelli Elastomerlerin Gerilim Gevşemesine Reçine İçeriğinin Etkisinin Araştırılması

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ABSTRACT

In this study, the effect of C9/C10 aromatic hydrocarbon resin and resin amount on the curing and stress relaxation properties of poly (epichlorohydrin-co-ethylene oxide) based elastomers was investigated. The curing parameters of elastomers that did not contain resin and elastomers containing resin in different proportions were examined. When examining the curing parameters, it was observed that with increasing resin content, the scorch time (ts2) and optimum curing time (t90) increased, and the curing rate decreased. Moving die rheometer studies showed that as the amount of resin increased, cross-link formation reactions were delayed or negatively affected. Additionally, temperature scanning stress relaxation (TSSR) studies were conducted to evaluate the effect of the resin on the stress relaxation behavior, service temperature, and cross-link density of the prepared vulcanizates. TSSR studies were carried out in two stages: first, after curing and then after removing the resin from the vulcanizates by extracting the test samples in toluene. TSSR data showed that when the resin ratio was above 5 phr, the stress relaxation behavior of poly(epichlorohydrin-co-ethylene oxide) elastomers increased with the increase in the amount of resin, which had an effect on reducing the service temperature.

Key Words

Resin, curing parameter, temperature scanning stress relaxation (TSSR), poly (epichlorohydrin-co-ethylene oxide) elastomer.

ÖZ

Bu çalışmada, C9/C10 aromatik hidrokarbon reçinenin ve reçine miktarının, poli(epiklorohidrin-ko-etilen oksit) bazlı elastomer karışımlarının kürlenmesi ve termo-mekanik özellikleri üzerindeki etkisi araştırılmıştır. Hazırlanan karışımlarda reçine içermeyen ve farklı oranlarda reçine içeren karışımların kür parametreleri incelenmiştir. Kürlenme parametrelerinin incelenmesinde artan reçine içeriğiyle birlikte ön kürlenme süresi (ts2), optimum kürlenme süresi (t90) değerlerinin arttığı ve kürlenme oranının ise azaldığı görülmüştür. Hareketli kalıp reometresi çalışmaları reçine miktarının artmasıyla birlikte çapraz bağ oluşum reaksiyonlarını geciktirdiği veya olumsuz yönde etkilediğini göstermiştir. Ayrıca, reçinenin hazırlanan vulkanizatların gerilim gevşemesi davranışı, servis sıcaklığı ve çapraz bağ yoğunluğu üzerindeki etkisini değerlendirmek için sıcaklık taramalı gerilim gevşemesi (TSSR) çalışmaları yapılmıştır. TSSR çalışmaları iki aşamada gerçekleştirilmiştir: ilk olarak kürlendikten sonra ve test örnekleri toluen içerisinde ektraksiyon yapılarak reçinenin uzaklaştırılmasından sonra yapılmıştır. TSSR verileri, reçine oranında 5 phr 'ın üzerinde çıkıldığında, poli(epiklorohidrin-ko-etilen oksit) elastomerlerin gerilim gevşemesi davranışının reçine miktarının artmasıyla arttığı bunun da servis sıcaklığını azaltıcı yönde etki yaptığı görülmüştür.

Anahtar Kelimeler

Reçine, kürleme parametreleri, sıcaklık taramalı gerilim gevşemesi (TSSR), poli(epiklorohidrin-ko-etilen oksit) elastomer.

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INTRODUCTION

Rubbers serve many purposes across an extensive array of products, encompassing essential items such as automobile tires, conveyor belts, hoses, seals, gaskets, gloves, footwear, and sports equipment [1-8]. Their versatile applications extend into the manufacturing and construction sectors, where they are employed for insulation and as a fundamental material for the fabrication of various components.

Each type of rubber possesses distinct advantages and disadvantages based on its main chain structure and side groups. The continuous evolution of elastomeric materials has led to the development new-generation options tailored for emerging industrial applications. Among these innovative elastomers is poly epichlorohydrin (PECH), a synthetic polymer derived from the epichlorohydrin monomer. PECH is defined as a polyether with a functional alkyl chloride side group. Different epichlorohydrin-based polymers are used to increase the requested performance and demand specific requirements in the final product. These include the use of poly(epichlorohydrin) homopolymer (CO), poly(epichlorohydrin-ethylene oxide) (ECO), and poly(epichlorohydrin-ethylene oxide-allyl glycidyl ether) (GECO) [9,10]. These modifications aim to optimize the properties of the elastomers for diverse applications. Epichlorohydrin polymers, in general, exhibit notable resistance to factors such as high oil, fuel oil, ozone, and thermal stress. This unique set of properties has positioned them as compelling alternatives to traditional rubbers like chloroprene, butyl, and nitrile. Consequently, the utilization of epichlorohydrin-based polymers has significantly increased in various industrial sectors in recent years [11]. This trend underscores these elastomers' adaptability and expanding utility in response to evolving industrial needs. In the rubber industry, fillers or compounds are used to increase the properties of rubber products such as tensile strength, modulus, and abrasion wear or to give different properties to the final product.

Fillers play a significant role in the formulation of rubber compounds, contributing to both the performance and cost-effectiveness of rubber-based products. Carbon black (CB) or silica is the most used filler in elastomeric materials [12]. However, the compounding process to add fillers and other additives into rubber via mechanical kneading requires a high energy consumption due to the high viscosity. Various process facilitators are commonly integrated into rubber formulations to enhance manufacturing efficiency. Also, resins are used as processing aids by improving flow properties, reducing viscosity, and promoting even distribution of ingredients during mixing, facilitating easier molding or shaping of rubber [14]. They can enhance the adhesion between rubbers and other materials, such as metal, fabric, and even other rubber compounds. This improved bonding contributes to the overall strength and durability of the final product [15,16]. They also enhance the thermal stability of rubber compounds, making them more resistant to heat, oxidation, and aging. This improves durability and performance under various temperature conditions [17,18]. Certain resins, like phenolic or rosin esters, act as reinforcing agents, increasing rubber compounds' strength, stiffness, and toughness [19,20]. This reinforcement helps in better load-bearing capabilities and resistance to wear and tear. Resins can be classified under many groups such as reinforcing, curing, tackifying, and adhesive. The choice of resin and its concentration in rubber compounds is critical for designing the final properties and performance of the rubber product. Manufacturers carefully select resins based on the specific requirements of the final product to achieve the desired balance of properties.

In a previous study conducted by Sen et al., the energy dissipation, stress-softening properties, mechanical, and dynamic-mechanical properties of epichlorohydrin-based elastomers were compared. This study, the effects of poly(epichlorohydrin) homopolymer and poly (epichlorohydrin-co-ethylene oxide-co-allyl glycidyl ether) (GECO) terpolymers on the energy dissipation behavior and physical properties depending on the curing chemical type, resin type, and filler amount were examined [21]. In this study by Sen et al., the contribution of the resin type to the energy absorption properties of the elastomer was demonstrated.

In this study, it was also proved that the damping property of hydrin-based polymers such as ECO and GECO could be significantly increased if resins as high as 20 phr were used. While the use of resin gives hydrin polymers this high energy damping property in the range of -40 to 40 ^{o}C, no study has been conducted on the negative effects of the melting of the resin, especially at high temperatures, on the mechanical properties, relaxation properties and energy absorption properties of elastomers.

In another study conducted by Tonka, the curing properties of ECH ter- polymer elastomers containing 20 phr aromatic hydrocarbon resin with GECO in different curing systems and the radiation stability of vulcanizates were examined [22]. Tonka have proven that the mechanical, dynamic-mechanical, and damping properties of GECO-based polymers can be changed in a controlled manner depending on the composition of the curing system used in the preparation of elastomers and the ionizing radiation dose. Both studies determined the resin ratio as 20 phr in the elastomers prepared with different resins.

In this study, it was aimed to examine the effect of resin amount on the curing properties, stress relaxation behavior, and cross-link density of poly (epichlorohydrin-co-ethylene oxide) based elastomer. After curing of elastomeric compounds containing different amounts of resin, the resin in the vulcanizates was removed by washing process and the effect of the resin amount on stress relaxation behavior and cross-link density was compared. All these studies will provide clarification on the extent to which elastomers, whose damping properties are improved by using a high proportion of resin, can maintain these properties at high temperatures.

MATERIALS and METHODS

Materials

The monomer content of the Poly (epichlorohydrin-co-ethylene oxide) (ECO) is 72% epichlorohydriın (ECH), and 28% ethylene oxide (EO) (C2000) was obtained from Zeon Co. (USA). The Mooney viscosity value of polymers is (@100°C ML (1 + 4)) and a viscosity range of 90-102. C9/C10 Aromatic Hydrocarbon Resin (ARResC9/10) used as the resin was supplied from HM Royal, Inc. (USA). The vulcanization system was based on trimercaptotriazine (Zisnet F-PT) and Diphenyl Guanidine (DPG). Other ingredients are commercial chemicals commonly used in the rubber and tire industry.

Preparation of Blends:

Table 1 shows the compositions of the elastomer. All the compounds were prepared by kneading in a Thermo-Haake Rheomix OS lab mixer at 40 ^{o}C at 50 rpm for 30 minutes. Vulcanization was carried out in a pressurized hot press (Brabender, Germany) at 190 ^{o}C and for respective optimum cure times of all the compounds.

Cure characteristics:

The optimal cure time and curing parameters of the elastomer were determined using a moving die rheometer (Alpha 2000 MDR) at 190^{o}C according to ASTM D5289-12. Tests were carried out by taking three samples from 3 different points of each blend series. The rheometer (MDR) device provides valuable insights into the curing process's parameters. Curing curves obtained from rheometer tests offer crucial information about the behavior of rubber materials during curing. The ML value serves as a significant indicator of viscosity and resistance to deformation. A lower ML value indicates decreased viscosity, making the material less resistant to flow. Additionally, parameters such as ts2 (scorch time) and t90 (optimum curing time) play pivotal roles in controlling the curing process, ensuring timely and effective vulcanization. The curing Rate Index (CRI) calculated as (CRI=100/(t90-ts2)), provides a measure of the curing rate, facilitating the adjustment of processing parameters. Furthermore, the delta torque value, reflecting cross-link density, influences the critical mechanical properties of the final product.

Differential Scanning Calorimetry:

Differential scanning calorimetry (DSC) analysis was carried out on a Perkin Elmer brand Pyrsis II DSC device. The melting point of the resin sample was investigated under the N$_2$ environment. The analyses were carried

Table 1. The compositions of the elastomer.

Component	ECO	ECO-R5	ECO-R10	ECO-R15	ECO-R20
Poly (epichlorohydrin-co-ethylene oxide)	100	100	100	100	100
C9/C10 Aromatic hydrocarbon	3			3	3
Resin (ARResC9/10)	0	5	10	15	20
Magnesium oxide (MgO)	3	3	3	3	3
Stearic acid	1	1	1	1	1
Diphenyl guanidine (DPG)	0.5	0.5	0.5	0.5	0.5
2.4.6- Trimerkapto-s-triazin (TMT)	0.8	0.8	0.8	0.8	0.8

out using heating rates of 10.0°C min^{-1} for an experiment with approximately 10.0 mg sample.

Temperature Scanning Stress Relaxation Properties: The stress relaxation of test samples was determined according to ASTM D 8363-20 standard by using the Temperature Scanning Stress Relaxation instrument (TSSR instrument by Brabender, Germany). The TSSR testing method involves a two-step procedure. The first step was to subject the test sample to a constant tensile stress of 50% to minimize the effect of thermal expansion at 23°C. After applying the extension, the sample was preconditioned for isothermal relaxation at 23°C for 2 h without heating. The second step, called isothermal relaxation, begins immediately after the first step and is heated up to 300°C at a heating rate of β = 2 K/min until complete relaxation or rupture of the sample. [23].

RESULTS and DISCUSSION

Cure Characteristic:

The torque difference in an MDR test is proposed to be theoretically linked to the shear dynamic modulus, and this modulus is associated with the total crosslink density in rubber compounds [28]. The rheometer curves of the rubber compounds with various resin content are given in Figure 1.

As can be seen from Figure 1, the torque value of the elastomers decreases as the resin content increases. The lowest ML value (3.88±0.06 dNm) was obtained for the elastomer containing 20% resin. The cure parameters obtained from the cure curves are given in Table 2.

Table 2 shows M_L values decrease with an increase in resin content. For the ECO rubber compound, the M_L value is measured at 1.61±0.03 dNm. In contrast, the compound ECO-R20, containing 20 phr resin, shows a 41% reduction, as 0.95±0.05 dNm. The resin, added to the blends demonstrates a plasticizing effect, reducing the compound viscosity [14,24]. Additionally, the ts2 duration, considered the initiation of vulcanization, increases proportionally with the resin content. Specifically, ts2 duration increases by 93%, and t90 duration increases by 28.9%. This indicates that the resin has a retarding or negative effect on cross-linking reactions. It may indicate that the resin used reduces the intensity of the interfacial interaction between the cross-linking components and the rubber and the degree of cross-linking at the interface. As a result, the curing rate index values decreased as the amount of resin increased. The delta torque value is calculated as the difference between MH and ML and is generally considered a measure of crosslink density. The delta torque value for ECO-R20 is 45.8% lower than the resin-free ECO elastomer. This indicates that as the amount of resin increases, the crosslink density of the elastomer decreases. The delta torque value, which is the difference between M_L and M_H of the curing curves, is considered a measure of cross-link density [27]. The Δtorque value of the ECO compound was measured as 7.16±0.001 dNm, and the Δtorque value of the ECO-R20 coded compound was measured as 3.88±0.06, decreasing by 46.7% compared to ECO.

Before examining the TSSR behavior depending on the amount of resin, the melting temperature of the Novares TL 100 resin used was determined. As seen in Figure 2, the melting point of Novares TL 100 resin was found to be 62.4±2.3 °C.

To remove the resin in the vulcanizates prepared according to optimum curing times, they were immersed in toluene (>99.5%) and the samples waited for 24 hours to reach equilibrium swelling. The test samples taken out at the end of 24 hours were washed with toluene three times and kept in an oven at 25 °C until they reac-

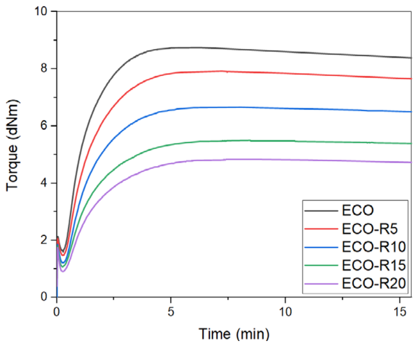

Figure 1. Curing curves of elastomers.

Table 2. Cure characteristics of R series elastomers.

	Compound Code				
Cure Paremeter	ECO	ECO-R5	ECO-R10	ECO-R15	ECO-R20
ML (dNm)	1.61±0.03	1.47±0.01	1.22±0.01	1.09±0.03	0.95±0.05
ts2 (min)	0.71±0.01	0.83±0.01	0.97±0.01	1.16±0.02	1.37±0.03
t90 (min)	2.71±0.11	3.17±0.10	3.39±0.06	3.72±0.02	3.83±0.02
Δ torque (dNm)	7.16±0.01	6.41±0.06	5.30±0.22	4.43±0.01	3.88±0.06
CRI (min-1)	50.2±2.7	42.8±1.8	41.3±0.7	39.1±0.01	40.7±0.12

hed a constant weight. The weight decrease in the test samples before and after washing on the test samples that came to a constant weight is shown in Table 3. The letter "T" was added to the end of the label of samples that were subjected to the washing process. TSSR studies of rubber compounds prepared before and after washing were carried out.

As stated in Table 3, the mass reduction of the ECO-labeled resin-free elastomer was determined as 1.55±0.16%. This result was attributed to the dissolution of process aid components such as stearic acid used in the prepared elastomer, and polymer chains that were not involved in cross-linking in toluene. Considering the mass reduction of the resin-free ECO elastomer, it can be stated that the resin in the vulcanizates is completely removed by the washing process. This observation also confirms that the resin does not participate in the cross-linking process.

Temperature scanning stress relaxation behavior

The normalized stress versus time isothermal relaxation curves for the elastomers are presented in Figure 3a before washing and Figure 3b after washing with toluene. The results show that the ECO elastomer without resin only lost 5% of its initial strength after 120 minutes. However, the strength of the EC0-R5 and ECO-R10 samples decreased by 7% and 8%, respectively, like the sample without resin. In contrast, the strength of the ECO-R15 and ECO-R20 samples decreased by 12% and 13%, respectively, which is considerably higher than that of the samples without resin. The study observed that the relaxation properties of the elastomer were negatively affected, particularly at 15% and 20% resin ratios. This is attributed to the lower mechanical strength of the resin compared to the elastomer, which creates weak points in the polymer and reduces its overall mechanical strength.

To better understand the effect of resin amount on polymer relaxation properties, the resin was removed from the polymer using the extraction method. The TSSR was used to analyze the relaxation properties of the polymers (Figure 3b). The results show that there is no significant change for the polymer containing 5% resin after resin removal by washing. On the other hand, it is seen that the stress-relaxation behavior of the polymers decreased after washing in samples containing 10%, 15%, and 20% resin. This suggests that using more than 5% resin has a negative impact on the stress relaxation properties of the polymer.

Table 4 provides the isothermal relaxation values for the ECO-R series before and after washing with toluene.

In studies on resin cross-linking reactions and curing processes, it was determined that the aromatic hydrocarbon resin used did not participate in cross-linking. It can also be said that the resin is trapped in a three-dimensional network that forms after curing. It was predicted that the resin reduces the cross-link density during curing, causing a negative effect on cross-linking reactions.

It was stated that TSSR studies consist of two stages. The first stage involves 2 h of isothermal relaxation at initial stress (σs) below 23 °C. It was observed that, depending on the resin content before washing, σs values were higher than the resin-free ECO elastomer. This is because the resin, which is solid at room temperature, gets trapped inside the vulcanizates after curing.

When the σs values of the vulcanizates in Figure 4a were examined before and after washing, a decrease in the initial force values was observed after removing the resin trapped in the three-dimensional network after washing. A similar trend was observed in Figure 4b, where the σ0 value marking the onset of non-isothermal relaxation was given, for all elastomers with more than 5 phr, a decrease in σs and σ0 values was observed in isothermal relaxation experiments conducted before and after washing.

The cross-link density of thermoplastic vulcanizates (TPVs) and conventional elastomers can be determined from TSSR curves using classical rubber elasticity theory [29,30]. Assuming an ideal rubber network, mechanical stress is simply proportional to absolute temperature. When the stress relaxation test is performed at a constant elongation, the slope of the temperature–stress curve—essentially the derivative of stress concerning temperature—is assigned as the temperature coefficient κ, as depicted in Equation (1). In a linear relationship, the cross-link density can be calculated by multiplying the temperature coefficient κ by the absolute temperature, as given in Equation (2).

$$\kappa = (\,(1)\ \partial\sigma / \partial T)\lambda \qquad \text{Equation 1}$$

According to the well-known theory of rubber elasticity, the mechanical stress σ is proportional to the absolute temperature, as expressed by Equation (2).

$$\sigma = (\rho RT/Mc)(\lambda - \lambda^{-2}) \qquad \text{Equation 2}$$

Here, ρ represents the density, λ stands for the strain ratio of the sample (where λ = l/lo), and R denotes the universal gas constant. Mc is defined as the average molar mass between cross-links, which is proportional to the reciprocal value of the cross-link density ν of the network.

The cross-link densities of the prepared elastomers were determined by TSSR studies before and after washing, and the effect of the resin amount on the cross-link density was examined.

After a 2-hour relaxation period, the samples were heated at a rate of 2 °C/min, as outlined in the test procedure. The normalized stress versus temperature of non-isothermal relaxation curves for vulcanizates, both before and after washing with toluene, are presented in Figures 5a and 5b, respectively.

Non-isothermal relaxation values of the elastomers series before and after washing with toluene are provided in Table 5.

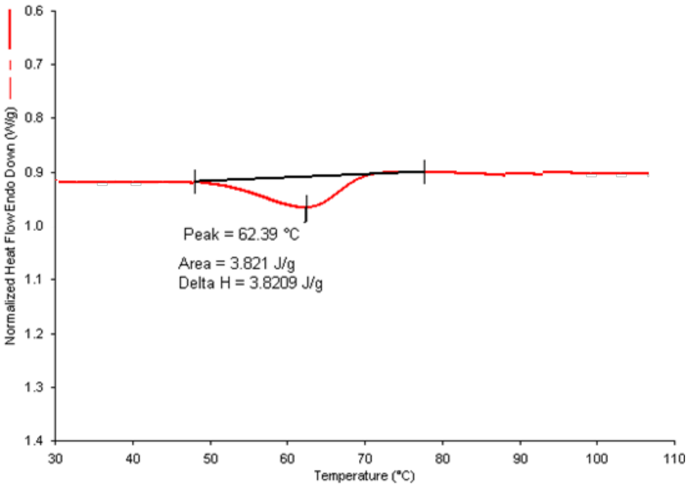

Figure 2. DSC thermogram of resin with a heating rate of 10 °C/min.

Table 3. Resin content in elastomers and mass percentage reduction after washing.

Elastomer Code	The resin content in the vulcanizate (%w)	The weight reduction in the vulcanizate after washing (%w)
ECO	0.00	1.55±0.16
ECO-R5	4.53	5.71±0.21
ECO-R10	8.67	9.21±0.18
ECO-R15	12.47	13.26±0.16
ECO-R20	15.96	17.57±0.14

As shown in Table 5, in the non-isothermal relaxation curves that commenced after isothermal relaxation, the initial stress value of the ECO sample was 0.46 MPa, with the initial stress values decreasing as the amount of resin increased. The lowest initial stress value was observed in ECO-R20, measuring 0.29 MPa. While the σ0 value of ECO before washing was 0.41 MPa, this value increased by 10% to 0.46 MPa after washing. However, the σ0 value of the sample coded ECO-R20, containing 20 phr resin, was 0.44 MPa before washing. This value decreased by 34.1% to 0.29 MPa after washing. The primary reason for this decrease is that the resin used is in a solid form at room temperature, thereby introducing additional resistance to vulcanizates under 50% tension. In Table 5, at the T_{10} service temperature, where the initial strength value of ECO series vulcanizates decreased by 10% before washing, the strength values decreased in direct proportion to the resin content. The T_{10} temperature of the ECO vulcanizate, which did not contain resin, was 201.4±4.5 °C. With the addition of 20 phr resin, the T_{10} value decreased by 63.5% and was recorded as 73.4±2.9 °C in the ECO-R20 coded vulcanizate. This resulted in a decrease in the T_{10} service temperature of the vulcanizates with the amount of resin. The T_{10} temperature of the ECO-R5 elastomer containing 5 phr of resin was 193.0±2.6 °C, which was very close to the temperature of the ECO elastomer. However, in blends with resin content above 10 phr, the T_{10} service temperature decreased by approximately 116 °C. As seen in Figure 4a, the relaxation curves of ECO-R series vulcanizates start around 70 °C. This service temperature drop is directly proportional to the amount of resin. As shown in Figure 2, depicting the DSC melting curve of our resin, the resin melts at 62.4±2.3°C.

In addition, when examining the cross-link densities determined through TSSR studies of ECO and ECO-R20 coded test samples before and after washing, the cross-link density of ECO-coded samples increased by 5.3%, rising from 118.6±2.4 mol/m³ before washing to 130.0±2.6 mol/m³ after washing. On the other hand, the cross-link density of the ECO-R20 coded samples decreased by 59.3% before washing, measuring 75.9±1.9 mol/m³. After isothermal relaxation at 23 °C for ho-

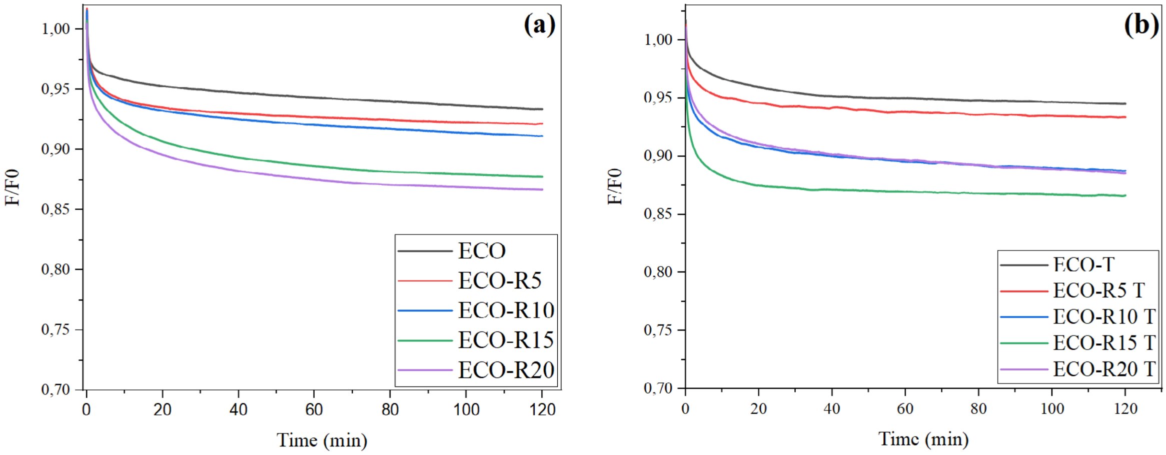

Figure 3. The normalized stress versus time isothermal relaxation curves for the ECO-R series.

Table 4. The isothermal relaxation values for the ECO-R series before and after washing with toluene.

	Before washing		After washing	
Elastomer Code	σs (MPa)	σ0 (MPa)	σs (MPa)	σ0 (MPa)
ECO	0.44±0.04	0.41±0.02	0.49±0.03	0.46±0.02
ECO-R5	0.40±0.02	0.37±0.03	0.43±0.02	0.41±0.01
ECO-R10	0.45±0.01	0.41±0.01	0.39±0.04	0.35±0.04
ECO-R15	0.52±0.03	0.46±0.02	0.38±0.03	0.33±0.02
ECO-R20	0.51±0.02	0.44±0.04	0.33±0.02	0.29±0.03

Table 5. The normalized stress versus temperature of non-isothermal relaxation values for elastomers before and after washing with toluene.

		Compound Code				
	Paremeter	ECO	ECO-R5	ECO-R10	ECO-R15	ECO-R20
Before washing	σ_0 (MPa)	0.41±0.02	0.37±0.03	0.41±0.01	0.46±0.02	0.44±0.04
	T_{10} (°C)	201.4±4.5	193.0±2.6	97.5±3.5	79.2±4.4	73.4±2.9
	T_{50} (°C)	233.2±6.4	230.7±5.9	219.1±4.9	201.4±4.3	184.4±5.6
	T_{90} (°C)	249.3±6.6	248.2±6.2	247.2±5.8	245.5±5.9	244.0±4.4
	v (mol/m^3)	118.6±2.4	111.7±2.1	125.6±3.8	139.3±2.8	113.8±2.5
After washing	σ_0 (MPa)	0.46±0.02	0.41±0.01	0.35±0.04	0.33±0.02	0.29±0.03
	T_{10} (°C)	201.8±4.2	200.9±2.9	195.9±2.4	184.3±2.6	188.5±3.2
	T_{50} (°C)	236.2±5.9	234.1±5.4	231.0±5.6	196.5±4.7	195.7±5.7
	T_{90} (°C)	251.2±6.4	249.1±6.1	247.7±5.9	196.5±4.6	195.8±4.7
	v (mol/m^3)	130.0±2.6	119.5±2.2	85.3±3.4	94.6±2.1	75.9±1.9

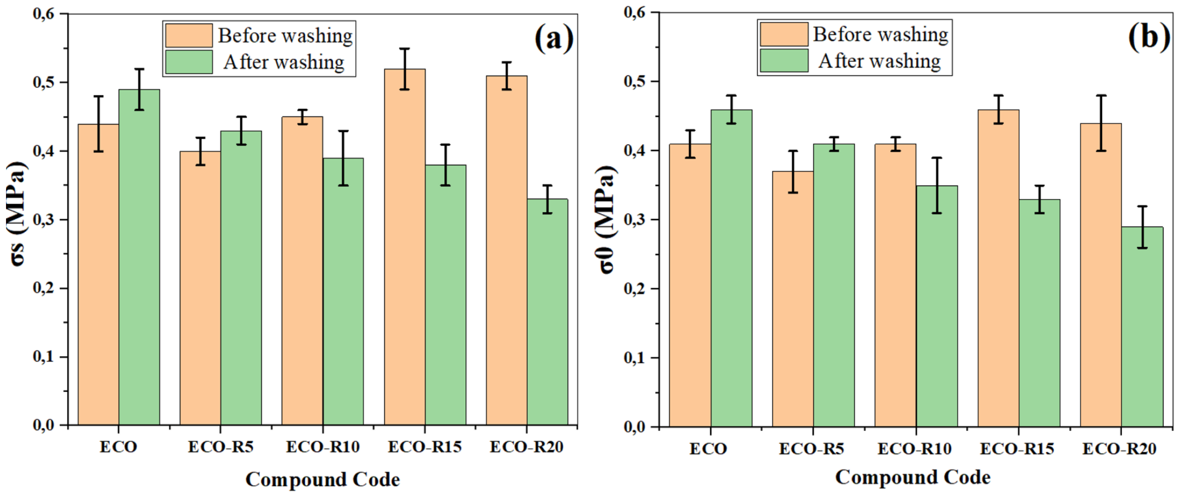

Figure 4. Isothermal relaxation behavior of elastomers before and after washing with toluene.

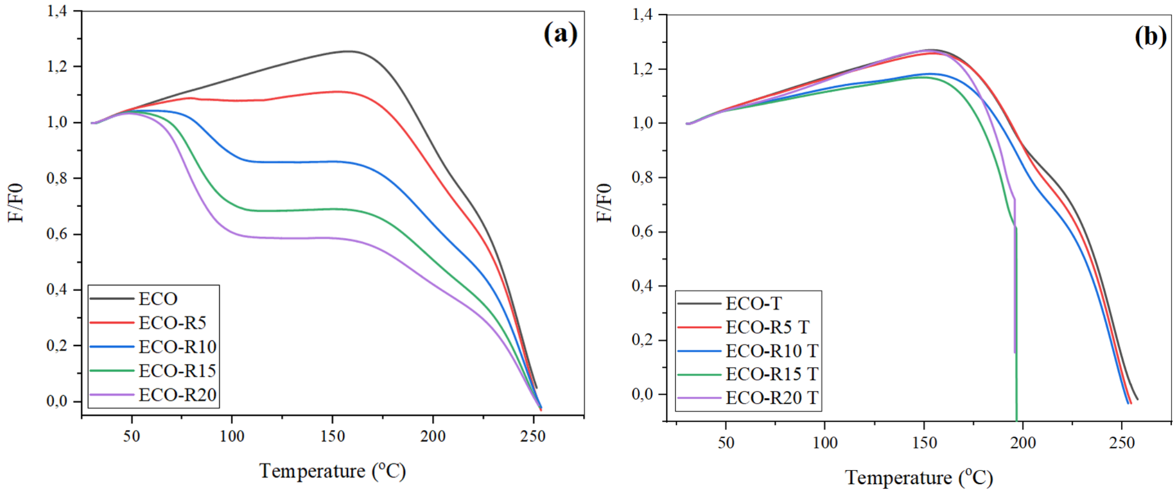

Figure 5. The normalized stress versus temperature of non-isothermal relaxation curves for elastomers.

urs, the cross-link density of elastomers is found by using the slope of the anisothermal relaxation stress-temperature curve using Equation 2. At this temperature, the rigidity of the test samples is higher due to the resin contained in the elastomers. One reason for this is that the initial slope values of the stress-temperature graph used to calculate the cross-link density were found to be higher in resin-containing elastomers compared to before washing.

The influence of Novares TL100 resin quantity on the non-isothermal relaxation behavior of ECO series vulcanizates is depicted more distinctly in Figures 6a, 6b, 6c, 6d, and 6e.

This suggests that the resin utilized does not undergo cross-linking [25] rather, it serves as a filler between polymer chains, melting at elevated temperatures and consequently increasing the stress relaxation behavior of vulcanizates [26].

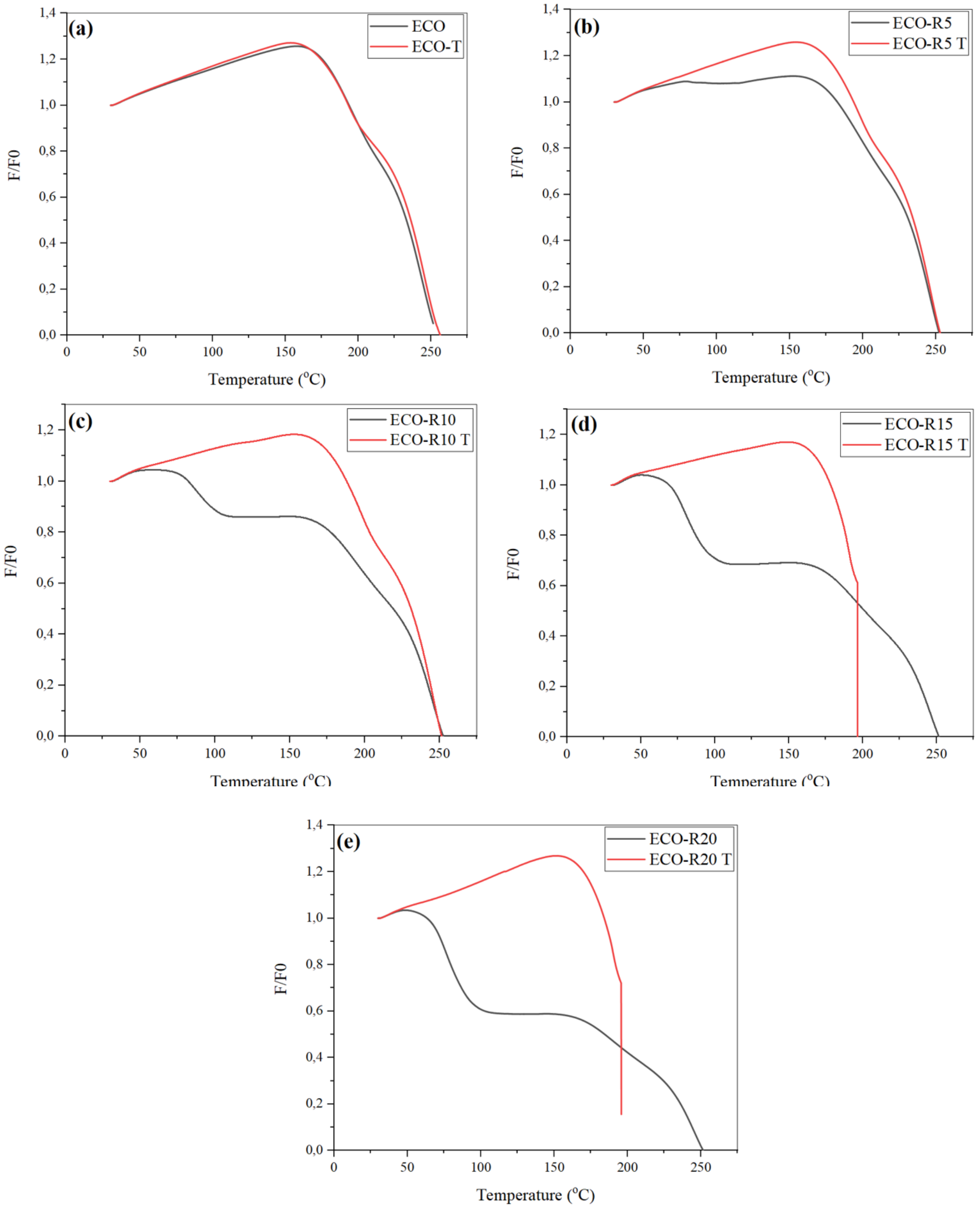

Figure 6. Normalized stress curves versus temperature for ECO (a), ECO -R5 (b), ECO -R10 (c), ECO -R15 (d), and ECO -R20 (e) series vulcanizates are presented before and after washing with toluene.

CONCLUSION

In this research, the effect of the resin used as a process facilitator on the curing parameters and stress relaxation behavior of poly(epichlorohydrin-co-ethylene oxide) based elastomers was examined. It was observed that as the amount of resin increased, both the scorch time (ts2) and the optimal curing time (t90) of the curing reactions increased. It can be stated that this causes a negative effect on the vulcanization reactions of the resin. Moreover, TSSR studies revealed that polymers have a significant effect on stress relaxation behavior, service temperature, and cross-linking density. These findings showed that stress relaxation behaviors increased with resin. The stress relaxation behavior increased in direct proportion to the use of resin above 5 phr. It clearly shows that resin amounts negatively affect the curing reactions of poly (epichlorohydrin-co-ethylene oxide) based elastomer, and the stress relaxation behavior of vulcanizate, service temperature, and cross-link density. The fact that the amount of resin plays a role in reducing cross-link density and increases the stress relaxation, especially in the range of 50-100 °C, has led to the conclusion that it can increase the viscous character of the elastomer and add positive effects to the damping properties. In order to clarify this issue, studies on the examination of damping properties of systems containing high levels of resin at high temperatures have been initiated in our research group.

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