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RESEARCH ARTICLE

Investigation of the effects of compound content and vulcanization temperature on the physical and mechanical properties of EPDM and NBR rubbers

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Abstract

In the industrial field, different kinds of parts are produced from rubber. Ethylene Propylene Diene (EPDM) and Acrylonitrile-butadiene Rubber (NBR) are the preferred rubbers in various fields. In this research, different types of hardness recipes were designed by using EPDM and NBR rubbers. Physical and mechanical values were analyzed. The results showed, with the increasing of carbon black amount, the hardness was increased, vulcanization time decreased and flow properties deteriorated. With increases carbon black, tensile strength changed from 8.8 MPa to 9.4 MPa for EPDM and 8.9 MPa to 13.7 MPa for NBR compounds. Opposite of tensile strength, the elongation values decreased 615% to 304% for EPDM and 511% to 221% for NBR. Tear resistance and density values increased with the increases in carbon black and the highest values belonged to N70 compounds for both. When physical values were analyzed, it was found that dimensional change decreased with the increasing of carbon black. With the polymer changes, it was seen that the dimensional change in NBR rubber is higher than EPDM rubber. When the effect of temperatures was examined, it was observed that high vulcanization and exposure temperature affects the dimensional changes badly for both rubber types.

1. Introduction

Polymers that show very high elongation under tensile force and return to their original state as soon as the force is removed are called elastomers. This polymer, referred to as elastomer in the scientific literature, is so named because of its high elasticity [1]. Rubbers are elastomeric polymers that are not cross-linked but can be cross-linked [2]. The rubbers are used for comfort and safety in homes, workplaces, playgrounds and sports fields, airplanes, automobiles and trains. In the industrial field, there are tapes, hoses, belts, vehicle tires, gaskets (sealing) and various parts are produced from rubber. Rubber has an important place in the developed world. Rubber is obtained in two different ways, naturally from the sap taken from plants considered as tree class grown in Asia, Africa and America, and synthetically from fuel derivatives and other minerals [3]. With developing technology and changing demands, many types of synthetic rubbers have been developed in laboratory circumstances. Nowadays, use of synthetic rubbers is quite common. In the automotive sector, Ethylene Propylene Diene (EPDM) and Acrylonitrile-butadiene Rubber (NBR) are the popular synthetic rubbers that can meet the needs in usage areas.

EPDM rubber has very good resistance to ozone, oxygen, heat and radiation, aqueous systems, polar environments and a wide range of acidic and alkaline chemicals. EPDM has a very low glass transition temperature as -45°C. It can also be used in outdoor applications and at high temperatures. It has a wide working range between -45°C to +150°C. NBR rubber has strong resistance to oils and fuels. It has good mechanical properties. It has strong air sealing properties. It has the ability to work between -40°C to +130°C. NBR is sensitive to ozone and oxygen degradation [4].

Rubber compounds are expressed in rubber recipes. A recipe is also called a material tree formed by mass ratios calculated to ensure that the rubber meets the desired properties. All additives are added to the recipe as phr. Phr is expressed as "parts per hundred of rubber" [5]. Rubber recipes consist of rubber, fillers, plasticizers (lubricants), activators, auxiliary chemicals and vulcanization systems.

Fillers are classified as black and white. Black fillers are carbon blacks. White fillers are such as calcium carbonate, silica, clay, talc. Among these various fillers, the most important reinforcing agent is used in the rubber industry is carbon black [6]. Carbon black is obtained by collecting elemental carbon in hydrocarbon vapor obtained by decomposition at high temperature on a surface [7]. Different carbon blacks are defined depending on the production technique. Fef N-550 carbon black is the most widely used carbon black type in injection molded parts. Carbon blacks directly affect the mechanical and physical properties of the compound.

The mechanical and physical properties of rubber compounds are not only affected by the compound recipe. The vulcanization conditions of the compound can also play a role on mechanical values. Vulcanization is the process of irreversibly imparting elastic properties as a result of cross-linking reactions in the structure of rubber materials. Vulcanization is the process applied to improve the mechanical properties of rubber compounds and increase its resistance to heat or solutions. There is no optimum degree of cross-linking that optimizes all mechanical properties. Optimum properties are obtained by selecting appropriate vulcanization systems and vulcanization chemicals while preparing rubber compounds [8].

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In order to get the final product to operate at the desired performance, the physical properties as well as its mechanical values must be well calculated at the beginning of the project. The calculation of physical properties is such an equation with many variables. The application mold should be designed by considering the content of compound, vulcanization conditions and exposure temperature. In order to get the product to be of appropriate dimensions, it must be manufactured in an appropriate way. This is why shrinkage allowance is so important in rubber products. Shrinkage allowance or shrinkage amount is the dimensional change of the part after produced [9]. Shrinkage allowances cannot be known precisely; however, it was known that compound content and temperature factor are the factors affecting the shrinkage allowance.

In the literature, there are some studies which shrinkage allowances are considered while designing the molds of plastic materials. In İçer's study, computer-aided modeling of plastic parts and female-male mold parts were investigated in accordance with the injection molding process. Since the shrinkage rate will vary between the types and the change in processing conditions will definitely cause different shrinkage rates, he completed his studies by giving the material type and shrinkage allowance to the software program used [10].

In Can's study, the modeling of a plastic part manufactured by the injection molding process and the modeling of female and male molded parts were investigated. The plastic part was designed by a CAD program and the process deficiencies were analyzed in prediction and analysis of defects on the surface of the plastic part in a CAE program. ABS and PP thermoplastics were selected for the materials that were used in the plastic part. While modeling the molds, the effects of both temperature changes and material changes on mold shrinkage were mentioned and modeling was done in line with this information [11].

In Zhao and colleague's research, on the development of micro molds and micro tool designs. According to the results, it was found that for different polymer materials and product designs, different gating systems and mold structures should be used for improved melt flow and cavity filling [12].

In this research, the effects of carbon black filler, vulcanization conditions and exposure temperature on the physical and mechanical properties of compounds that formed from two different rubber types were examined. There are not many studies in the literature comparing the mechanical and physical properties of rubber compounds for mold and recipe design. Carbon black effects on rubber compounds or shrinkage allowance calculations for plastic materials studies can be seen in literature in general. However, in this study, the content of recipe and also exposure and vulcanization temperature effects on mechanical and physical values were investigated for two different types of rubber. In this way, with this research, it is aimed to provide guidance for optimum calculation while designing product molds and rubber formulation to meet the customer requirements in rubber industrial applications.

2. Materials and methods

2.1. Raw materials

EPDM and NBR rubber types were used in this study. Both types of rubbers were supplied from Versalis company. Dutral 4038 rubber with Mooney Viscosity ML 1+4(125 °C) is 60 MU, Propylene content is 27% and ENB content is 4.4% was used as EPDM rubber and Europrene N3345 (ACN Content is 33%, Mooney Viscosity ML 1+4(100 °C) is 45 MU) was preferred as NBR rubber. Fef N550 (BET Surface Area 40 ± 5 m^2/g) carbon black was used as filler. Carbon black was supplied by Orion company. Active zinc oxide (ZnO, 95% of zinc oxide) from the activator group was purchased from Melos company and stearic acid (acid value is 45%) supplied from Evyap. In the plasticizers group, paraffinic oil (paraffinic value > 65%) was used for EPDM compounds that purchased from Petrol Ofisi and diisononyl phthalate (DINP value < 99.5%) was used for NBR compounds that purchased from Plastay. For accelerator groups in vulcanization chemicals, ZBEC (Zinc bis(dibenzyldithiocarbamate)), TBBS (N- tert-Butylbenzothiazole-2-sulfenamide), MBTS (2,2-Dithiobis(benzothiazole)), TMTM (Tetramethylthiuram monosulfide), CBS (N-Cyclohexylbenzothiazole-2-sulfenamide) were supplied by Univar company. Sulfur vulcanization system was preferred for both types of rubber and S-80 (sulfur) was purchased by Univar. All ingredients in the recipes were calculated as phr and they were named based on the rubber types and hardness.

2.2. Preparation of compounds

EPDM and NBR recipes were designed by changing the proportion of carbon black in its content. The formulations of the rubber compounds are given in Table 1.

Table 1. Recipes

Raw Materials	E50 phr	E60 phr	E70 phr	N50 phr	N60 phr	N70 phr
Dutral 4038	100	100	100	0	0	0
EuropreneN3345	0	0	0	100	100	100
Fef N550	100	140	180	30	50	70
ZnO	5	5	5	5	5	5
Stearic Acid	1	1	1	1	1	1
Paraffinic Oil	120	120	120	0	0	0
DINP	0	0	0	20	20	20
ZBEC	2	2	2	0	0	0
TBBS	2	2	2	0	0	0
MBTS	2	2	2	0	0	0
TMTM	0	0	0	0.3	0.3	0.3
CBS	0	0	0	2	2	2
S-80	1.5	1.5	1.5	2	2	2

In this study, all compounds were made under the same process parameters. The compounds were made in 3L laboratory type banbury and two roll mill (Werner Pfeidener, Germany) machines. All the compounds were prepared according to ISO 2393 norm.

The ingredients were added as follows;

- The polymer group (EPDM rubber and NBR rubber) was put into the banbury and the rubber disintegrated for 30 seconds.
- Carbon black, ZnO and stearic acid were added to the polymer and plasticizer (paraffinic oil for EPDM, DINP for NBR) was added on top. The materials were mixed at a speed of 50 rpm for 5-6 minutes to get homogeneous mixing of all materials.
- Accelerator groups were added to the mixture that it reached to 120°C. The mixture was mixed again for 15-20 seconds and the dough mixture (compound) was taken to the two-roll mill.

- S-80 was added to the compound in the two-roll mill and mixed for 5 minutes.

After preparation of rubber compounds, in order to perform the tests, the test plates were produced in a compression press with compression production method under 170°C 15 minutes and 190°C 15 minutes vulcanization parameters.

2.3. Mechanical, rheological, flow and physical properties

Rheological properties for all compounds were performed using Alpha MDR 2000 (USA) tester at 190 °C 2 minutes parameters and vulcanization structures were examined. Viscosity values were performed using Ektron (Malaysia) Mooney Viscometer at 125°C (1+4) criteria. One test sample was used for determination of rheological properties and viscosity results. Samples for mechanical measurements were prepared according to DIN 23529. Hardness was performed according to ISO 48-4. Zwick Roell Z010 (Germany) Shore meter was used for a hardness test. Tensile strength, elongation at break tests were performed on S2 dumbbell specimen according to DIN 53504 standard and tear test was performed on Type-C specimen according to ASTM D624 standard using Zwick Roell Z010 device. For the aging tests, Memmert (Germany) ventilated drying ovens were used and the test was performed according to DIN 53508 standard. Air aging tests were performed at 100°C and 168 hours. Density test was checked using Precisia (UK) brand density meter according to ISO TS 2781 specification. These test specimens were used for determination of mechanical properties. Determination of physical properties and measurement of sample dimensions were performed using Mitutoyo (Japan) calipers. One specimen was used for the determination of physical properties. A 200*200 test plates were used to calculate the mold shrinkage allowances based on the structure of the material. For this intention, the dimensions of the test plate were measured with the help of calipers and the perimeter was calculated with the measurements of all edges. Accordingly, the perimeter of the mold was compared with the perimeter of the vulcanized rubber compounds. Cold measurements were taken as basic for the comparison. Care was taken to ensure that the mold and the rubber plates were cold.

3. Results and discussion

3.1. Mechanical properties

3.1.1. Hardness, tensile strength and elongation values

Hardness, tensile strength, elongation at break and tear resistance results are shown in Table 2.

The hardness values of all EPDM and NBR compounds enhanced as the carbon black in the recipe increased. The increases of carbon black amount in recipes also improves the tensile strength and tear resistance values. This may be due to the fact that the type of carbon black used is semi-activated carbon black. Based on BET surface area of Fef N550, it is called semi-activated filler [13]. Activated carbon blacks (high and semi), which are used as filler and additive material in natural and synthetic rubbers, improves the strength of the material [14]. The usage of high proportion carbon black in rubber compounds, due to the large surface area and zigzag structures of carbon black, creates strong network structures which increase the mechanical properties of the compound. For this reason, when a suitable amount is added to the compound, carbon black increases the tensile strength of the compounds [15]. However, there is a radical decrease in elongation values. It is expected that elongation decreases with increasing hardness in rubber materials [16]. Elongation decreases with increasing amounts of carbon black and the reason for this is the increase in carbon black that reduces the amount of the basic rubber material surrounding carbon black granules which increases the hardening rubber compound and then lower elongation at break [15].

3.2. Density values

As the proportion of carbon black in recipes increased, the hardness of the compounds increased and the density also enhanced accordingly. The reason for this is the carbon black with high density added into the recipe. Carbon black increases the density of EPDM and NBR and as shown in figure 1, and this is attributed to the carbon black which has a density 1.8 g/cm^3, NBR rubber has 0.98 g/cm^3 and EPDM rubber had 0.88 g/cm^3.The densities of NBR compounds were higher than EPDM compounds. Figure 1 shows the density values of the compounds.

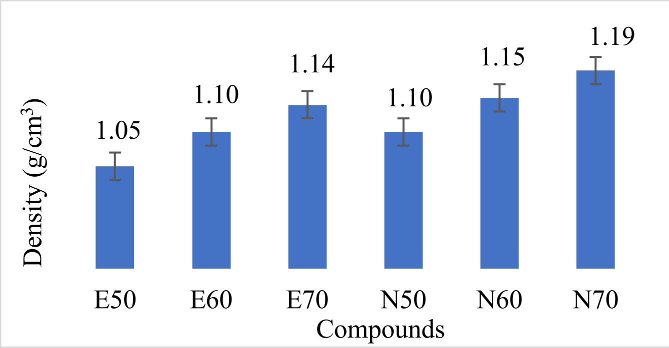

Figure 1. Effects of filler content and rubber type on density values

3.3. Rheological and flow properties

Rheometer device was used for the rheological values of the compounds and the values are given in Table 3.

For rheological values, each rubber type needs to be interpreted in its own way. For each rubber type, the minimum resistance of flow (ML) and maximum resistance of flow (MH) was enhanced with increasing amount of carbon black. On the other hand, the time to start vulcanization (scorch time) (TS_2) and the time to complete vulcanization (T90) values decreased. In this case, it can be concluded that the increase of carbon black in the recipe combination, increasing the resistance of the rubber material rotational movement under pressure and temperature for a certain period of time. The similar results can be seen in the literature. The MH and ML increases with the increase of the carbon black proportion in the compounds. The difference between MH and ML could be used as an indirect indication of the network formation in the crosslinking system. Both TS_2 and T90 times are decreasing with increasing the active filler content in the compounds [17]. It shows that the hardness increases with the increase in the carbon black ratio in the same recipes, shorten the vulcanization times and therefore the compounds are vulcanized earlier. This situation provides information about the time required to fully fill the mold while the product is being produced and guides the mold design. A viscometer was used for the flow values of all the compounds and the values are given in Table 4.

Table 2. Mechanical values of the compounds

Tests	E50	E60	E70	N50	N60	N70
Hardness (Sh-A)	50±1	60±1	70±1	50±1	60±1	70±1
Tensile strength (MPa)	8.8±0.2	9.3±0.3	9.4±0.2	8.9±0.3	12.9±0.3	13.7±0.1
Elongation at break (%)	615±6.3	446±5.9	304±6.1	511±7.1	447±,6.8	221±6.4
Tear resistance (N/mm)	29.2±0.8	30.8±0.7	31.6±0.5	29.0±0.9	37.1±0.9	42.6±0.2

Table 3. Effects of filler content and rubber type on rheological values

Criterias	E50	E60	E70	N50	N60	N70
ML (Ib:in)	0.36	0.73	1.61	0.36	0.57	0.83
MH (Ib:in)	5.47	6.33	8.57	9.09	12.32	22.41
TS_2 (mm:ss)	01:06	00:57	00:49	00:51	00:45	00:31
T90 (mm:ss)	01:37	01:35	01:34	01:23	01:17	00:52

Table 4. Effects of filler content and rubber type on flow properties

Mooney	E50	E60	E70	N50	N60	N70
Viscosity	15.70	25.50	45.61	22.69	27.98	31.94

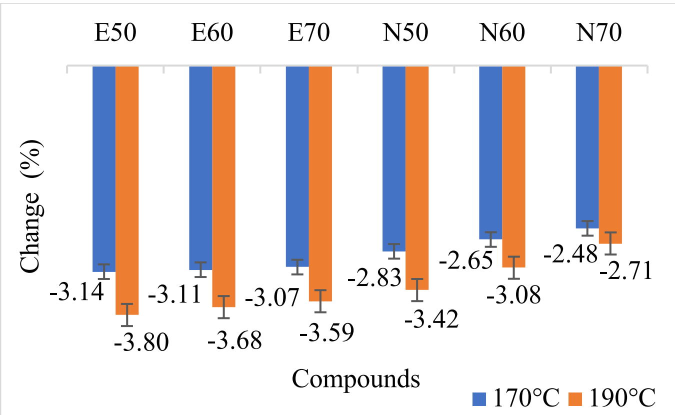

Figure 2. Effects of vulcanization temperature and rubber type on dimension changes

Viscosity also increased with the increase of carbon black ratio of the compound. This is consistent with the ML value, which is the flow indicator in the rheometer. The results are similar to the study by Comez and Ozturk. It was found that the flow properties of the compound deteriorated with increasing hardness and carbon black amount. This was confirmed by the parallel result of ML and viscosity test from rheological results. The increase in ML, MH and viscosity may lead to the conclusion that the processability of the compound in the process would be difficult. At the same time, it can be concluded that the vulcanization times would be brought forward, causing mold filling problems, especially for injection molding manufacturers [18]. The other significant information is flow properties of the compound at molding design. While mold designing, it should be considered to obtain optimum formed product. Based on viscosity results, the designers can consider the hardness for only changed carbon black ratio formulations, during creating flow channels of mold.

Table 5. Effects of vulcanization temperature and rubber type on perimeter dimensions

*	E50 mm	E60 mm	E70 mm	N50 mm	N60 mm	N70 mm
Test plate mold	816.5	816.5	816.5	816.5	816.5	816.5
170°C cure	790.8	791.1	791.4	793.4	794.9	796.2
190°C cure	785.5	786.4	787.1	788.5	791.3	794.3

3.4. Physical properties

3.4.1. Effect of vulcanization temperature

In addition to the change in the structure of the material in the mold shrinkage allowances, the effect of temperature was also examined. The variability in vulcanization parameters was also examined. For this purpose, the vulcanization temperature was changed by keeping the vulcanization time constant (15 minutes). Table 5 shows the plate dimensions obtained as a result of vulcanization of EPDM and NBR compounds at different vulcanization temperatures.

Figure 2 shows the changes in the dimensions of the plates produced at different vulcanization temperatures.

As can be seen from Figure 2, vulcanization temperature affects the dimensions of the final product. It was observed that as the vulcanization temperature increased, the dimensions of the product became smaller, and the shrinkage increased. The effect of carbon black ratio in the same rubber type, the shrinkage decreased as the carbon black increased, that is, in materials with the same content, the dimensional shrinkage of low carbon black amount is higher than the high ones. In the study conducted by Can in plastics, it is stated that mold shrinkage decreases with the increase of filler material in the content of composite materials [10]. When comparing EPDM and NBR rubbers, under the same vulcanization conditionals and based on similar hardness with changing carbon black amount, it was found that EPDM rubber experienced more dimensional shrinkage than NBR rubber.

3.4.2. Effect of exposure temperature

The effect of temperature is not only experienced during vulcanization. Rubber products are also exposed to temperatures in their function areas. EPDM and NBR rubber are generally used in the automotive industry and the products made of these rubbers are used in areas close to engine equipment in automotive. In order to look at the effect of the high temperature while the parts working in the functional area, the rubber plates aged under 100°C for 168 hours. The aging was carried out on a plate vulcanized at 170°C for 15 minutes and dimensions were compared. Figure 3 illustrates a comparison of the dimensions of the plates that aged at 100°C for 168 hours with the plates non-aged.

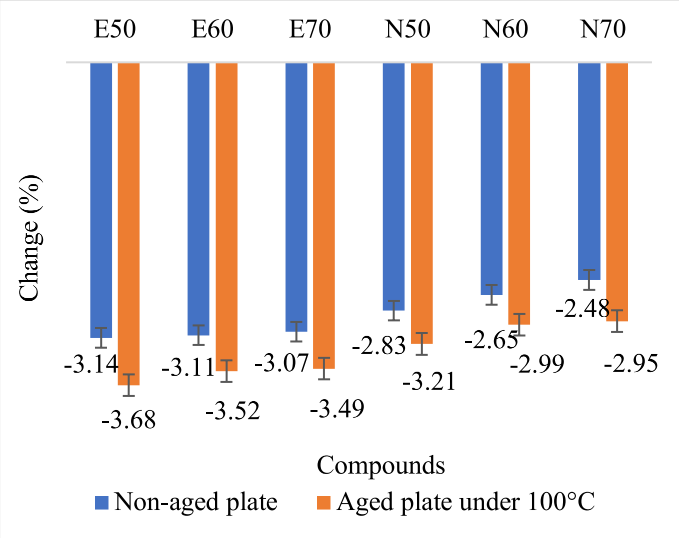

Figure 3. Effects of aging, rubber type and hardness on dimension changes

When looking at the dimensions of the aged-plates, dimensional reductions are noticed. It is possible to see a similar study in literature on PLA materials. PLA samples were aged at 50°C for different durations. According to the results, the shrinkage ratio is not acceptable after 72h and 168h aging. According to the study, it was observed the shrinkage is not strong during aging for short term exposures but it was stated that it should be considered when designing goods manufactured where high accuracy is required [19]. In this study, the conditions of the area were kept stable, only increasing the aging period but it is clear to understand that the environment conditions are vital for shrinkage allowances. This shows that not only the vulcanization temperature, also the temperature that is exposed in the working area, affects the sample's dimensions. Similar results were found for both rubber types.

4. Conclusions

In this study, six different compounds that using different ratio of carbon black and EPDM, NBR rubbers were formed. The rheological, flow, mechanical and physical properties of the compounds were investigated.

When the mechanical values of the compounds were examined, it was observed that the hardness increased as the amount of carbon black in the compound increased. The tensile strength, density value and tear resistance increased while the amount of elongation decreased with increases in carbon black. With these changes, tensile strength changed from 8.80 MPa to 9.40 MPa for EPDM and 8.90 MPa to 13.70 MPa for NBR compounds. Opposite of tensile strength, with increases in hardness, the elongation values decrease 615% to 304% for EPDM and 511% to 221% for NBR. Tear resistance and density values increase with the increases with increases carbon black and the highest values are N70 compounds for both.

When the rheological results were examined, the flow resistance of the compounds had similar results to the viscosity values. As the carbon black ratio of the compounds increased, the ML and MH values in the rheometer also increased. Viscosity value also increased with the changes.

Physical properties were evaluated considering both rubber type, carbon black amount, vulcanization temperature and exposure temperature. When EPDM and NBR rubbers are compared, it is seen that EPDM rubber undergoes more dimensional change than NBR rubber at similar hardness. Based on increasing carbon black, dimensional change decreased. In the measurements made by changing the vulcanization temperature (170°C to 190°C), it was observed that the change in product dimensions increased with the increase in vulcanization temperature. The changes of dimensions were seen among -2.48% to -3.42 for NBR and among -3.07% to -3.80% for EPDM compounds. When the vulcanized plates were aged under the same conditions, it was calculated that the aged-plates had smaller dimensions than their original dimensions. The maximum change is E50 as -3.68% in EPDM compounds and N50 as 3.21% in NBR compounds.

The study showed that varying amount of carbon black, rubber type and also vulcanization temperature and the exposed temperature affect the physical and mechanical properties of compounds. In industrial applications, these properties are extremely significant for product suitability. Therefore, while designing both compound formulation and rubber mold, it is crucial to consider these items before starting the study.

Within this research, EPDM and NBR rubber types were examined but different rubber types are used in various applications so in the future, it is planned to carry out the same studies with different rubber types as AEM, ACM, CR and ECO for comparison.

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Author contributions

Selda Öztürk provided discussion and interpretation of test results. Ergenç Gökay carried out the tests and preparation the compounds. Ezgi Erbek Cömez provided guidance throughout the article, investigated literature and data collection and also Ezgi Erbek Cömez was responsible for writing, control and editing issues.

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