

The Effect of Microstructure on the Mechanical Properties of Devulcanized Waste Tires

Hamid Yazdani * , Hoorah Fakhari Ahad Ghaemi

1. Department of Chemical engineering, Payame Noor University. E-mail: h.yazdani@pnu.ac.ir
2. Department of Chemical engineering, Payam Noor University, Tehran, Iran. E-mail: hoorah.fakhari@yahoo.com
3. Department of Chemical, petroleum and Gas Engineering, Iran University of Science and Technology, Tehran, Iran. E-mail: aghaemi@iust.ac.ir

ARTICLE INFO	ABSTRACT
<p>Article History: Received: 16 April 2023 Revised: 02 July 2023 Accepted: 04 September 2023</p> <p>Article type: Research</p> <p>Keywords: Waste Tires, Rubber, Mechanical Properties, Microstructure, Devulcanization</p>	<p>With the development of the automotive industry, waste tire production has also increased significantly. And their accumulation causes many problems and occupies a large space. One of the most effective solutions is recycling waste tires by devulcanization, which is more economical than re-production. This paper aims to investigate the relationship between microstructure and physio-mechanical properties of devulcanized rubber samples. For this purpose, mechanical tests and identification tests including FTIR (Fourier-transform infrared spectroscopy) and NMR (Nuclear Magnetic Resonance) were performed on the samples. It was determined that; there is an inverse relationship between sol weight fraction and NR/SBR ratio; the lower the ratio, the higher the vinyl bonds, tensile strength, and swelling index of the specimens, and the better the curing. On the other hand, increasing this ratio decreases the percentage of devulcanization. Therefore, the low NR/SBR ratios will increase the process efficiency and improve the mechanical quality of the devulcanized rubbers.</p>

Introduction

Increasing the amount of polymer waste, including an end-to-life tire, is a real danger to the environment and living things [1]. Due to the large space the waste tires take, they are mostly hard to dispose [2]. So, more than 70 gigawatts of energy would be wasted annually to grind them. Also, this type of waste often comes out of the landfill surface or destroys the anti-leakage cover of the landfill. And it causes the accumulation of bacteria and insects in the presence of moisture. Even burning them produces harmful gases such as dioxin, which can cause serious air pollution problems [3, 4]. Therefore, recycling tires is an effective solution to all the mentioned problems and also has less cost and energy consumption than its re-synthesis [5]. There are various methods for recycling tires, including crushing and decomposing rubber into raw materials (devulcanization). devulcanization destroys the three-dimensional network of cross-links of waste tires. The methods of this process are mechanical, heating, chemical, biological, ultrasonic, etc. [6, 7]. The devulcanization agents selectively break crosslinks (S-S and C-S); with the least degradation in the main polymer chain (C-C) [8]. devulcanized tire

* Corresponding Author: H. Yazdani (E-mail address: h.yazdani@pnu.ac.ir)



rubber can be mixed with raw rubber or other mixtures to reuse, without significant change in its physical and mechanical properties. It can be used as a substitute for conventional concrete aggregates [9] which increases the cement's ductility and strain control and also makes concrete structures more resistant to heat and cold [10]. It can also be used in modified asphalt to improve properties, increase softness and viscosity and reduce penetration [11]. Examination of scattered electrons in SEM and ATR-FTIR tests performed on polystyrene rubber particles by Lanzón et al. Showed that expanded polystyrene has several external cavities to which the curing mortar can easily adhere [12].

One of the important issues in rubber recycling is the quality of the final product and the efficiency of the process; Therefore, it is very important to examine the mechanical and microstructural properties of tires and define a relationship between them. This knowledge about the properties helps a lot to find which rubbers are more suitable to devulcanize and get better quality in order to reuse. So far, no complete research has been done regarding the relationship between the microstructure and all physio-mechanical properties of devulcanized tires. Because, the devulcanization process, performing chemical and physical tests on different samples and re-curing them is not simple and requires a lot of time and precision. Therefore, the current research has comprehensively addressed all the mentioned cases. For this purpose, 15 samples similar to waste tires were devulcanized in a continuous process. To investigate the relationship between properties, first NMR (nuclear magnetic resonance spectroscopy) and FTIR (Fourier-transform infrared spectroscopy) analyses were performed on the samples. Then, they were re-cured and physio-mechanical tests were performed on them. In the current study, the main mixture (NR/SBR) was prepared at a temperature of 160°C and pressure of 150 bar, while George et al. Prepared the same mixture according to ASTM D15-627 standard at a temperature of 150°C [13].

Based on microstructural and mechanical research; The three-dimensional grids in the structure of tires improve their mechanical properties, chemical resistance, and thermal stability [14,15]. According to NMR analyses performed in various articles; The type of filler used will affect the structure and mechanical properties of the rubber's network. The carbon black-filled mixture has a stronger surface interaction with more lattice chains and fewer dynamics than the silica-filled mixture and other fillers [16,17]. SEM analysis, X-ray diffraction (XRD), and FT-IR spectroscopy by Gwon et al. Showed that the addition of dual cement has a favorable effect on the structural strength and compressive strength of sulfur polymer composites, and also can prevent them from cracking [18]. Evaluating the mechanical properties of devulcanized rubber in some research; showed that the mechanical properties are affected by the devulcanizing agent and the tensile strength is a function of the crosslink density and is directly proportional [19]. In Valentine and Dorigato's research mechanical properties including; Elongation at break and tensile strength were measured by the 50WDT global electronic tester at a cross-sectional head speed of 500 mm/min [20]. Colom et al.'s strategy to prevent possible changes in the crosslink density of specimens during experiments; was microstructural analysis of the samples in powder form; without any additional processing [21].

Devulcanization is usually recognized as a solution to major environmental problems. And the subject of this study is to examine the parameters related to the product of this process. It leads to discover which kind of tires are suitable for devulcanization and which are not. Also, under what conditions the efficiency is the highest, that helps to improve the final product and save more time and energy. Actually, this paper aims to improve the solution of a problem. Not just solving a problem. Since this important issue seems to be small, it has not been dealt with in a very comprehensive and specific way.

Experimental

Materials

Tire materials including natural rubber (SMR20 grade) and styrene-butadiene rubber (grade SBR1500) were obtained from Malaysia and Bandar Imam Petrochemical, respectively. Additives and fillers were provided by Pars co (Iran), Behran co (Iran), and Bayer (Germany). And Di Xylene Di Sulfide (described in Table 1) was used as devulcanizing agent. Since knowing the basic structure of the material is very important; mixtures similar to waste rubber were prepared. Then they were devulcanized by a twin-screw extruder of the TSE 20 model made by the Brabender Company. the formulation of the original mixture is shown in Table 2 (based on ASTM D15-627). And the condition of processing the derived samples is shown in Table 3.

Table 1. Details of the devulcanizing agent used

Properties	Value
Flashpoint	165°C
Value of Sulfur	27.5 wt.%
Boiling point @ 1 atm	120°C
Specific weight @ 30°C	118.5 Kg/cm ³

Table 2. NR/SBR devulcanized compound formulation

Material	Phc
Devulcanized NR/SBR	100
ZnO	5
Oil 250	10
Stearic acid	2
CBS	50
MBT	0.7
Sulfur	1

Table 3. Devulcanization condition and ratios of the samples

Sample	C	T (°C)	D (wt.%)	NBR/SBR ratio
2	-1	210	10	1.754869
3	-1	190	5	2.062787
5	0	190	0	1.885286
6	0	210	5	2.040742
8	0	230	0	2.27654
10	1	190	5	1.730654
12	0	230	10	1.730003
14	0	210	5	1.512373
15	-1	230	5	2.156566

Tests

The methods mentioned in Table 4 have been used to investigate the physio-mechanical and microstructural properties of the samples.

Table 4. Structural identification and physio-mechanical tests

Test	Standard	Device/ Brand
NMR	Iso 21561	Bruker Avance DPX
FTIR	Iso 12965	Bruker IFS48
tensile	ASTM D412	Instron 6025
Hardness shore	ASTM D2240-02	Zwick Analog shore A
Mooney viscosity	ASTM D1646	Zwick viscometer 4309
Rheometry	ASTM D2084	Zwick rheometer B-4308
Compression Set	ASTM D395	-
	ASTM D1054	Frank Tripsometer Dunlop
Resilience	ASTM D 5963	Frank

Abrasion	ASTM D 6814-02	-
Sol swelling	ASTM1505D	-

The Amount of Devulcanization

The amount of devulcanization is determined by measuring the weight percentage of the gel (an insoluble component of rubber) and determining the cross-links density (the number of moles of connected parts in the rubber network per unit volume). And it is calculated according to ASTM D6814-02 using Eq. 1.

$$\text{Devulcanization}\% = \frac{V_1 - V_2}{V_1} \times 100 \quad (1)$$

V_1 and V_2 are the density of the cross-links before and after the devulcanization.

Ideal devulcanization is a process that only breaks the cross-links and does not break the main chain of the polymer. The Horikx theory (1956) can be used to study the degree of degradation in the polymer main chain. Also, the relationship between the sol weight fraction and the density of cross-links is determined by the statistical equations [22].

$$1 - \frac{v_{e_2}}{v_{e_1}} = 1 - \frac{(1 - S_2^{\frac{1}{2}})^2}{(1 - S_1^{\frac{1}{2}})^2} \quad (2)$$

$$1 - \frac{v_{e_2}}{v_{e_1}} = 1 - \frac{\gamma_2(1 - S_2^{\frac{1}{2}})^2}{\gamma_1(1 - S_1^{\frac{1}{2}})^2} \quad (3)$$

Indices 1 and 2 in the equations above mean before and after devulcanization. v_e symbolizes cross-link density, S is the sol fraction, and γ is the lattice index or average of the number of units connected along the chain. If only crosslinks break; the equation between the amount of sol and the density of cross-links (2) is correct. and if both the crosslinks and the main chain are broken in devulcanization; Eq. 3 will be correct.

Results and Discussion

Relationship between Physical and Mechanical Properties by Combining the Percentage of the Microstructure of the Sol Section of Devulcanized Samples

Fig. 1 is a diagram of the ratio of NR/SBR and the percentage of weight fraction for devulcanized samples using Table 5 results. with increasing NR in devulcanized samples; an almost decreasing trend is observed for the sol weight fraction. When the NR/SBR ratio is high, the sol weight fraction of the devulcanized sample is low. In this case, the NR/SBR ratio indicates that the NR chains are not broken or less broken during the devulcanization.

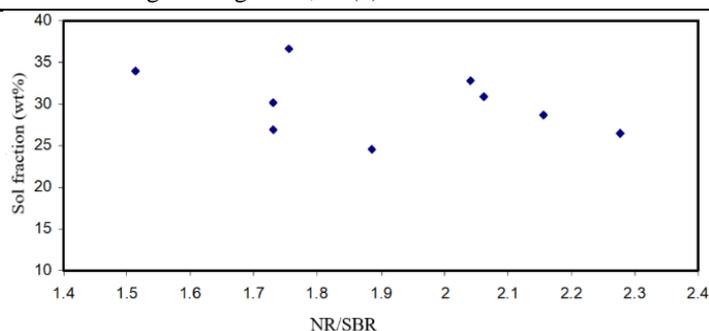


Fig. 1. Diagram of changes in weight percentage of sol fraction to NR/SBR ratio

Table 5 shows the values obtained from the physio-mechanical tests, and the percentage of components obtained from the $^1\text{H-NMR}$, $^{13}\text{C-NMR}$, and FTIR spectra (spectra with less overlap and error). Which gives us a more accurate value for analyzing the results.

Table 5. The ratio of data obtained from microstructure and physio-mechanical properties of devulcanized samples

samples	2	3	5	6	8	10	12	14	15
St/Bu	0.362027	0.326964	0.457726	0.501502	0.347709	0.477105	0.309072	0.385042	0.377031
Vinyle-h	16	15	18.3	17	18.2	18.7	25	17	19
trans/cis	1.5	1.4	1.6	1.84	2.2	1.9	1.8	1.75	1.68
Sol weight fraction	36.6	30.9	24.6	32.8	26.5	26.9	30.1	34	28.7
Δ torque	13.09	29.17	28.23	13.18	50.96	24.29	6.62	19.57	20.5
Devulcanizat ion %	81.81	48.85	88.6	76.73	54.98	84.95	59.2	86.57	67.15
swelling index	362.58	412.71	460.19	337.32	275.82	415.94	269.23	417.45	308.13
Mooney viscosity	15.35	56.4	56.12	18.04	90.34	45.99	46.34	29.26	40.12
Tensile strength	2.68	6.4	6.2	2.83	4.75	6.5	2.15	4.89	2.15
Elongation at the rupture point	112.84	112.84	121.84	99.3	10.94	131.83	75.12	129.02	49.89
Rupture strength	14.83	18.18	20.09	10.75	12.91	22.73	9.56	15.07	11.71
Compression set (70°C)	47.4	58.43	36.8	46.2	43.8	54.55	66.86	56.7	49.7
Compression set (25°C)	31.68	12.49	10.1	23.61	36.72	13.69	36.53	18.92	28.81
abrasion	44.01	21	27.12	46.74	39.56	19.13	49.38	35.94	42.84
hardness	63.63	63.3	61.23	62.7	93.7	62.63	66.3	63.97	78.5
resilience	15.75	27.4	25.37	15.9	15	27.35	13.95	21	12
Crosslink density	3.23	2.58	2.03	4.13	7.99	2.67	7.25	2.39	5.84

When the samples are subjected to high shear, the cross-links are opened and the NR chains are broken into smaller chains. Which makes it easier to dissolve in the solvent and increase the Sol weight fraction. according to Fig. 2, with an increasing NR/SBR ratio, the crosslinking density of devulcanized samples (except sample 12 due to degradation) is almost increasing. The loss of dual bonds and vinyl structure in the SBR results in lower SBR and a higher NR amount in the NMR spectrum. which increases the NR/SBR ratio. Due to the high percentage of gel, Sample 8 comes out of the extruder in a powder form. That has the highest Δ torque, the maximum amount of NR/SBR (2.3), the lowest sol weight fraction (after sample 5), and the highest amount of vinyl (Bd1,2). This indicates that the high temperature (230°C) and the average shear rate broke the main chains and the formation of double bonds at the end of the chain increases the amount of vinyl. Also, the high temperature converts a higher percentage of the trans structure to cis. That is the reason sample 8 got the highest trans/cis ratio.

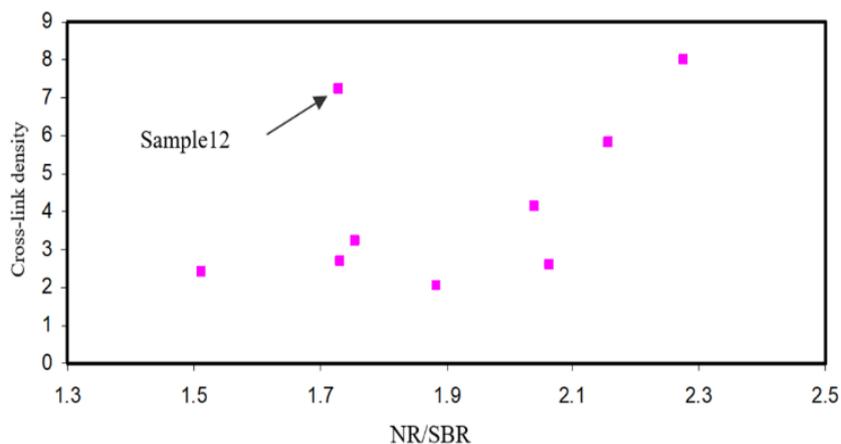


Fig. 2. Diagram of changes in crosslinks density to NR/SBR ratio

The devulcanization process was done at a high temperature, which cause the small molecules, with a low boiling point, to evaporate. resulting in a low sol weight fraction. Sample 2 with the highest amount of sol (36.6%) has one of the lowest amounts of vinyl bonds. As the shear rate increases, the vinyl butadiene bonds and the main chain are broken. which causes more sol weight fraction and low NR/SBR ratio. Fig. 3 shows the approximate decreasing trend of vinyl bonds with increasing sol weight fraction.

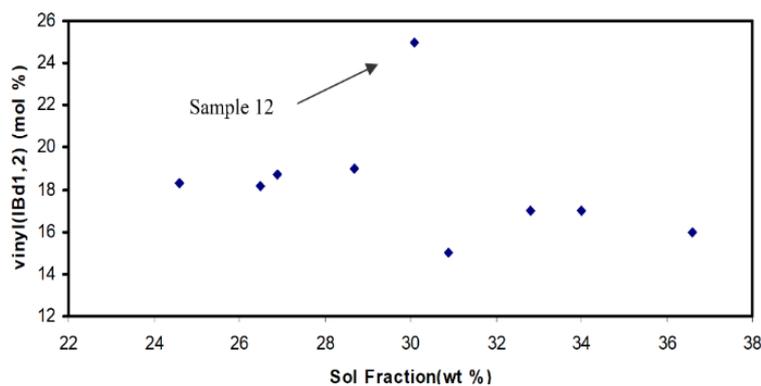


Fig. 3. Graph of vinyl changes to the sol weight fraction

According to Fig. 3, the lower the sol weight fraction, the higher the number of vinyl bonds. that make the sample more suitable for curing. Samples with a higher sol weight fraction, such as samples 2, 6, and 14, have a higher percentage of trans.

Due to the severe degradation of the main chains and in the SBR, the St/Bu ratio increases. When vinyl bonds are lost in the SBR, the number of butadiene decreases. So, we see an increase in styrene in the spectra. So, the percentage of SBR decreases and the NR increases.

Sample 12 got the highest amount of vinyl bonds, the lowest Δ torque, and the average amount of sol weight fraction, in the presence of 10 wt.% of the devulcanizing agent. The main chain is also broken in this sample. after breaking the main chains, a high percentage of vinyl bonds is observed in the spectrum. Which is due to vinyl bonds at the ends of the short chains.

Samples No. 5, 10, and 14, got the highest percentage of devulcanization. And sample 14 has also the lowest NR/SBR ratio at 210°C, medium shear rate, and the presence of 5 wt.% devulcanizing agents. This probably means the sulfur bonds have been broken well without breaking the main chain. Thus, the percentage of SBR and NR will not change much, and the NR/SBR ratio will get closer to 1 (i.e., the main mixture).

As the percentage of devulcanization increases, the swelling index increases, so samples No. 5, 10, and 14 got the highest swelling index.

With increasing sol weight fraction, Mooney viscosity decreases. According to Table 5 and Fig. 1, samples with higher NR/SBR ratio have higher Mooney viscosity. This is due to the long chains with a high molecular weight of NR. That causes high NR/SBR ratio and high Mooney viscosity in the devulcanized samples.

The St/Bu ratio is the highest for sample 6. because the Butadiene structure has become an aliphatic structure; so, the amount of Butadiene has decreased. Instead, an increase in styrene and NR is observed in the spectra. which causes a higher Mooney viscosity.

Among the devulcanized specimens, samples No. 5 and 10 got the highest tensile strength, which results in the highest swelling index, devulcanization percentage, vinyl bonds, the lowest cross-link density, and NR/SBR ratio. at the lowest NR/SBR ratio, the amount of SBR and a number of vinyl bonds are high, which makes the better curing of SBR in the re-curing stage.

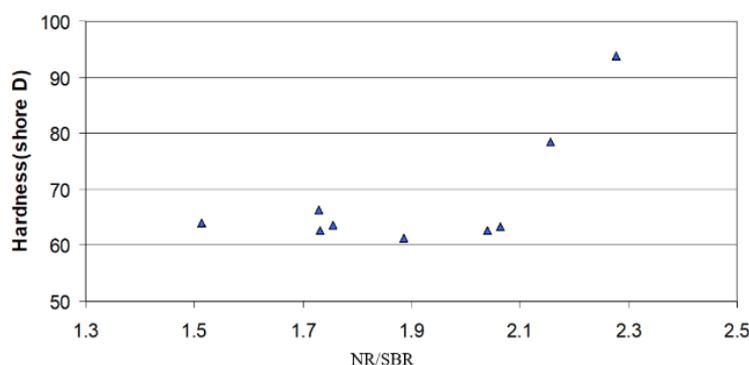


Fig. 4. Graph of changing hardness to the NR/SBR ratio

According to Fig. 2 and 4, the hardness and density of the cross-links cause similar changes in the NR/SBR ratio. Since high temperatures cause higher hardness in samples due to the evaporation of small molecules with low boiling points, sample 8 got the highest hardness. In this sample, the SBR fraction is not devulcanized completely and a large amount of it remains as a gel.

As shown in Fig. 5, with increasing NR/SBR ratio, especially from 2 onwards, the amount of elongation at the rupture point decreases.

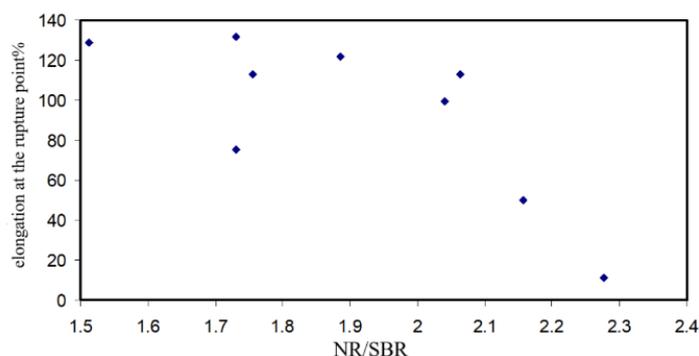


Fig. 5. Graph of changing elongation at rupture point to NR/SBR ratio

If the percentage of crosslink is not high enough, it will cause a lower percentage of NR/SBR, and a greater amount of elongation at rupture. in the high percentages of vinyl bonds, the rupture strength is on its highest level (samples 5 and 10). High percentages of vinyl bonds occur at low NR/SBR ratios. For these samples, the Butadiene vinyl bonds are less damaged. resulting in high rupture strength properties. The more vinyl bonds, the greater the curing, the higher the percentage of cross-links, and the better the mechanical properties.



Samples subjected to high-temperature devulcanization processes, such as sample 12, got the highest values of compression set (at both 25 and 70°C) and a low NR/SBR ratio. which shows that the sample is damaged during devulcanization and the SBR and NR chains are broken and the chain breakage creates a high percentage of vinyl bonds at the end of the chain. which reduces the amount of elasticity and increases the compressive strength.

The lowest compression set is related to sample 3 (about 12). This sample 3 was subjected to the devulcanization process at 190°C with a low shear rate and in the presence of a 5 wt.% devulcanizing agent. Which got a high NR/SBR ratio and elasticity.

Conclusion

In the current study, it was found the sol weight fraction decreases by increasing the $\frac{NR}{SBR}$ ratio. Also, with increasing this ratio, the density of crosslinks shows an increasing trend, similar to hardness. which is the reverse trend of elongation at the rupture point. As the percentage of devulcanization increases, the swelling index increases. The samples with the lowest $\frac{NR}{SBR}$ ratio got the highest swelling index, tensile strength and vinyl bonds. That is similar to the original sample. The results show more crosslink breaks at high shears and lower temperatures. Samples with higher $\frac{NR}{SBR}$ ratio will get a higher percentage of crosslinks through re-curing. It is also been concluded that high temperatures convert more percentage of the trans structure to cis. And samples with higher sol weight fractions got a higher percentage of trans structure and samples with the highest torque got lower $\frac{trans}{cis}$ ratios. As the sol weight fraction increases, the Mooney viscosity decreases. The samples with higher $\frac{NR}{SBR}$ and $\frac{St}{Bu}$ ratios got higher Mooney viscosity. It turned out that the lower the $\frac{NR}{SBR}$ ratio, the higher the compression set and the higher the vinyl bonds.

Nomenclature

NR	Natural rubber
SBR	Styrene-butadiene Rubber
Phc	Per hundred compounds
Ve1	The density of crosslinks before the devulcanization
Ve2	The density of crosslinks after the devulcanization
S	Sol fraction%
γ	The index of unit connection
C	Screw configuration
D	the amount of devulcanizing agent
T	Temperature (°C)

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Data Availability

The data generated or analyzed during the current study will be provided by authors if it is requested. The datasets generated during or analyzed during the current study are available from the corresponding author on reasonable request

Conflict of Interest

The authors declare that they have no conflict of interest.

Author Contributions

All authors contribute to the literature search; concepts, material preparation, design, definition of intellectual content, data acquisition, statistical analysis, manuscript editing, and manuscript review. All authors read and approved the final manuscript.

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