

The Microwave Assisted Reclamation of the Phenolic Cured Butyl Rubber

Hosein Amoli Vatan Dust and Saeed Ostad Movahed*

Faculty of Sciences, Ferdowsi University of Mashhad, Iran

*Corresponding Author

Saeed Ostad Movahed, Faculty of Sciences, Ferdowsi University of Mashhad, Iran..

Submitted: 2023, June 06; Accepted: 2023, June 29; Published: 2023, July 07

Citation: Dust, H. A. V., Ostad Movahed, S. (2023). The Microwave Assisted Reclamation of the Phenolic Cured Butyl Rubber. *Adv Envi Wast Man Rec*, 6(2), 416-427.

Abstract

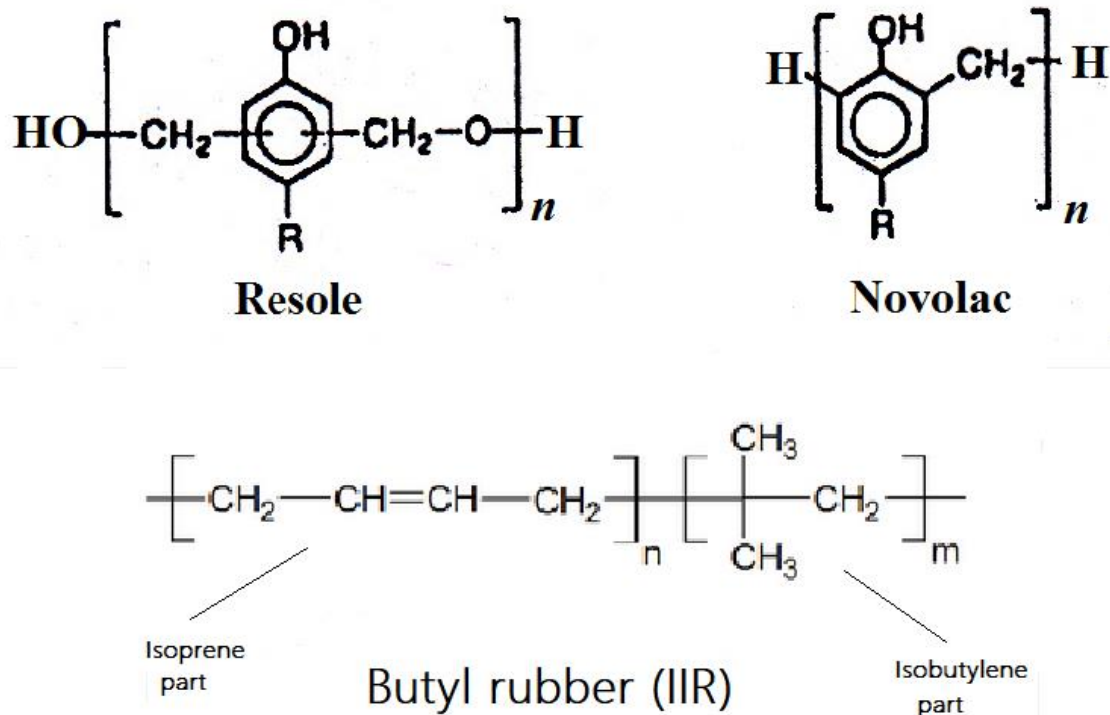
The cured rubber made bladder is used for curing the green tire because, it is thermally stable and has low gas permeability. The bladder is made with the cured butyl rubber as a base rubber under a phenolic curing system. To save the resources and also the environmental protection, it should be de-crosslink to gum rubber. Several waste bladder rubber compounds prepared with different de-crosslinking agents in formulation. Subsequently, the prepared compounds irradiated in a microwave oven. The de-crosslinking parameters including the sol fraction and de-crosslinking % measured using traditional rubber swollen tests. It revealed that the Fourier transform infrared spectroscopy (FTIR) graphs were suitable evidences to prove the existence of the phenolic resin in the structure of the used waste bladder butyl rubber powder. Efficient de-crosslinking observed for all studied de-crosslinking agents. It was evidenced by the SEM micrographs and also, the measured crosslink densities of the reclaimed rubbers. The highest and the lowest de-crosslinking %'s belonged to the compounds with DPDS and DCBS in formulation, respectively. Based on the obtained de-crosslinking parameters, the DPDS selected as a suitable de-crosslinking agent for the used waste bladder butyl rubber. In accordance with Horikx analysis, the microwave irradiation broke down the crosslinks and rubber main chains simultaneously. The increasing in microwave irradiation temperature had positive effect on the de-crosslinking process but it had equal effect on crosslink and main chain scission.

Keywords: Butyl Rubber, De-Crosslinking, Microwave Irradiation, Reclamation, Bladder

1. Introduction

Butyl rubber (IIR) or poly-isobutylene-isoprene (Scheme 1) is a synthetic rubber developed by cold cationic copolymerization of 2-methylpropene (isobutylene) with about 1 to 3 % cis- 2-methyl-1,3-butadiene (isoprene) using Lewis catalysts in organic solvents by Sparks and Thomas in 1937 [1]. The isoprene provides the necessary unsaturation sites for curing (crosslinking). Butyl rubber has outstanding characteristics including, resistance to attack by oxygen and ozone, good chemical resistance to organic and inorganic materials, heat resistance, stability in dilute acids and bases, and low gas and moisture permeability [2]. The most of above mentioned properties are due to the low unsaturation (double bond) in chemical structure of the butyl rubber. Butyl rubber uses in manufacturing of wide variety of rubber articles,

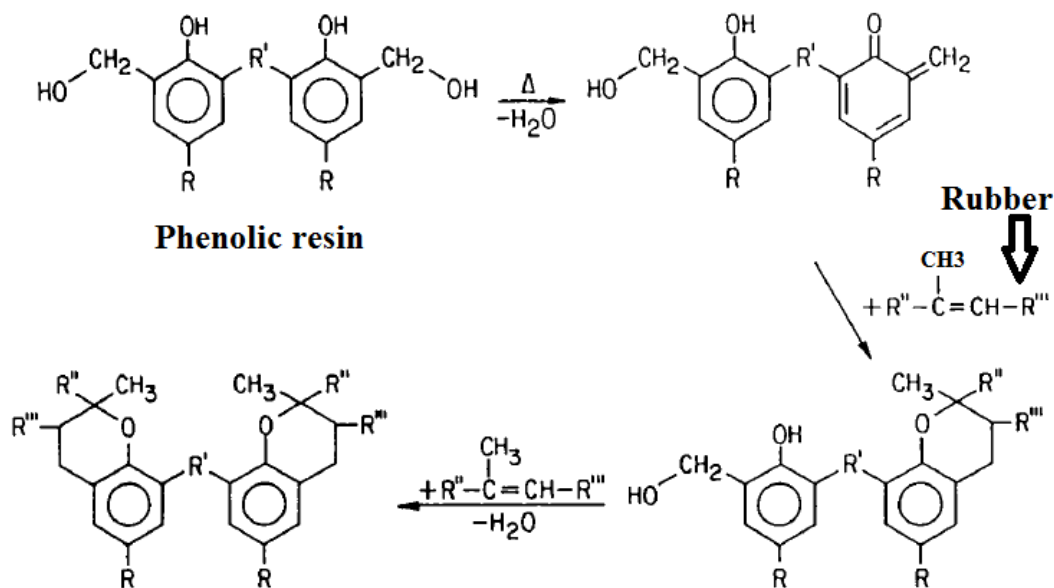
i.e., inner tubes of tires, inner layer of tubeless tires, rubber insulators, seals, gaskets, and etc.. The macromolecules of the green (un-crosslink) rubber should be crosslink with each other to make a rubber made article for industrial and commercial use. Among of several crosslinking (curing) methods, three methods are widely use, including sulphur (vulcanization), peroxide, and resin (phenolic) crosslinking [3]. The final properties of a rubber made article depends to the method of curing directly. As an illustration, for producing a heat and pressure resistance rubber made article, the peroxide and phenolic methods prefer because, in these curing methods, unlike of the sulphur vulcanization with relatively unstable formed C-S and S-S bonds, the stable C-C bonds form between two macromolecules of the rubber [4, 5].



Scheme 1: Butyl Rubber (IIR) and Traditional Phenolic (Phenol-Formaldehyde) Resins, Resole and Novolac.

The cured rubber made bladder is used for curing the green tire because, it is thermally stable, and has low gas permeability. For curing the tire, after assembling the different parts of the un-cured rubber compounds (green tire), it put inside of a hollow mold to cure the whole tire under the heat and pressure. The internal side of the green tire shapes and cures by a filled bladder with superheated steam. Normally, the bladder is made with cured butyl rubber as a base rubber under a phenolic curing system [6]. Scheme 1

represents the traditional used phenolic (phenol-formaldehyde) resins for the cured rubber made bladder. As observed, dimethylene ether and methylene groups connect the phenolic units in Resole and Novolac, respectively. The known phenolic rubber curing mechanism (Chroman mechanism) shown in Scheme 2 [7]. As observed, the phenolic resin makes a bridge between two rubber macromolecules and subsequently, a crosslink network forms in the whole rubber matrix.



Scheme 2: Chroman Mechanism of Rubber Curing by Phenolic Resin [7].

It is interesting to note that unlike of the other curing methods (vulcanization and peroxide crosslinking), in phenolic curing, the unsaturated double bond in IIR structure (isoprene part) attacks by the phenolic resin. Hence, the cured rubber has less double bonds when compared with the uncured rubber.

The cured rubber made bladders reach to their end of use and life service. To save the resources (raw materials) and also, environmental protection, it should be recycled to useful materials [8]. There are four main strategies in rubber recycling, primary or re-using, secondary or mechanical, tertiary or chemical, and quarter nary or energy recovery [9]. The former is the most desirable strategy because, the recycling product is uncured (reclaim) rubber which it may re-cure for producing the new rubber made articles.

In rubber recycling, the re-using strategy refers to de-vulcanization for sulphur curing and de-crosslinking for the rest rubber curing methods, including phenolic curing. In rubber de-crosslinking, three dimensional rubber networks which produced during crosslinking (curing) reactions, are broken. The ultimate goal of de-crosslinking process is the break of all crosslinks without main rubber chain scission [10, 11]. Different techniques for de-crosslinking of the cured rubbers were proposed, including chemical, mechanical, biological, thermo-chemical and irradiation techniques [12]. Among of the irradiation techniques, the microwave irradiation is desirable because microwaves enable to break the chemical bonds in cured rubber network selectively [13].

Numerous researches were done on recycling of butyl rubber, including Khavarnia et al., Kuan Jiang et al., Liliame et al., Feng et al., Karmanova, Polvoy et al., Vagizova et al., and Tao Zhang et al. [1, 14-21]. They used different techniques to de-vulcanize sulphur cured butyl rubber. However, based on the knowledge of the authors, there is not any paper in the literature for de-crosslinking of phenolic resin cured butyl rubber (the cured rubber made bladder).

In this study, the effects of microwave irradiation on the waste bladder butyl rubber powder compounded with different de-crosslinking agents at various operative conditions studied and the results discussed.

2. Experimental

2.1. Materials: Waste butyl rubber and de-crosslinking agents

The cured rubber made waste bladder supplied by Yazd rubber industrial complex, Yazd, Iran. To evaluate the rubber powder composition, a thermo gravimetric analysis (TGA) instrument (STA 1500, Scinco Co. Ltd., and South Korea) was employed. The TGA performed in an inert (nitrogen) atmosphere below 410oC and in the air above 410 °C, respectively [Figure 2]. Table 1 summarizes the TGA results.

| Property | | Amount |
|---|--------------------------------------|--------|
| Sol content (%) | | 7 |
| Density (g/cm ³) | | 1.10 |
| Crosslink Density (CLD)* (mol/m ³) | | 281 |
| TGA | Unburned minerals (%) | 23 |
| | Carbon black (%) | 17 |
| | Rubber (%) | 54 |
| | Light materials including oil (%) | 6 |

* CLD was calculated using the Flory-Rehner equation [1].

Table 1: Physical Properties and Composition of the Used Cured Rubber Made Waste Bladder Powder

The used de-crosslinking agents were Diphenyl disulfide (DPDS, Sigma-Aldrich, Germany), tetramethylthiuram disulfide (Perkacit TMTD, Flexsys, Belgium), Benzothiazyl-2-Dicyclohexyl Sulfenamide (DCBS, ACroChem, USA), Dipentamethylenethiuram (DPTT, Western Reserve chemicals, USA), N- tert-butyl-benzothiazole sulphonamide (TBBS, Western Reserve chemicals, USA), and 2-mercaptobenzothiazole (MBT, NOCIL limited, India). 2-2'-Dithiobis(benzothiazole) (MBTS, NOCIL limited, India). The aromatic oil (processing aid) had a boiling point of 270 °C and supplied by local market.

2.2. De-Crosslinking Procedure

The de-crosslinking agent dissolved in aromatic oil with the aid of heat. Subsequently, it added to the waste rubber powder in a litre glass beaker and allowed the rubber powder to soak in the treated oil in an oven for 24 hours at 80 °C. This operation guarantees the maximum penetration of the oil in the rubber powder. In the next stage and for de-crosslinking of the rubber compound, the beaker put inside a laboratory microwave oven and irradiated at the selected temperatures with the highest output power. The used microwave oven (GMO-530, Gosonic, Turkey) had the output power, frequency, and capacity of 900 watt, 2000 megahertz,

and 30 litres, respectively. It also equipped with a temperature regulator to control the inside temperature of the microwave oven. Subsequently, the irradiated compounds were processed on a two roll mill with cylinders diameter and length of 14 and 32 cm, respectively, to produce rubber sheets for further tests.

2.3. Measurement of the Crosslink Density (CLD), Sol Fraction, De-Crosslinking %, the Identification, and Morphology of the Rubber Compounds

To distinguish the availability of the phenolic resin in waste rubber powder the attenuated total reflectance Fourier transform infrared spectroscopy (ATR-FTIR, AVATAR 370FT-IR, Termo Nicolet, USA) with a resolution of 4cm⁻¹ was used.

Toluene used as solvent in rubber swelling tests to determine the crosslink density and sol fraction of the rubber compounds. To do rubber swelling test, five grams of the rubber compound placed inside of a tapped glassware filled with 400 ml toluene and allowed to reach the ultimate weight. After weighing the swollen gel, it was dried in an oven to remove the solvent for 24 hours at 80 °C. The CLD was then calculated using the Flory–Rehner equation [1]:

$$V = -\frac{1}{V_s} \frac{\ln(1 - \nu_r^0) + \nu_r^0 + \chi(\nu_r^0)^2}{(\nu_r^0)^{1/3} - \nu_r^0/2} \quad (1)$$

Where V is the CLD (mol/m³), is the volume fraction of rubber (polymer) in the swollen gel (Equation 2), V_s is the molar volume of toluene (1.069×10^{-4} m³/mol at 25 °C), and χ is the interaction parameter ($\chi=0.557$).

$$\nu_r^0 = \frac{1}{1 + \frac{d_r}{d_s} \left(\frac{1}{1 - f_{fil}} \right) \left(\frac{w_s}{w_0} - 1 \right)} \quad (2)$$

where d_r , d_s , f_{fil} , w_s , and w_0 were rubber density (1.10 g/cm³, Table 1), solvent density (0.867 g/cm³), the initial weight fraction of the filler (fillers) in the butyl rubber waste powder, the weight of the swollen gel, and the weight of the dried sample, respectively. The de-crosslinking % and sol fraction % calculated using Equations 3 and 4.

$$\text{De-rosslinking (\%)} = 100 \times (\text{Initial waste rubber CLD} - \text{Final rubber CLD}) / \text{Initial waste rubber CLD} \quad (3)$$

$$\text{Sol content (\%)} = 100 (w - w_0) / w \quad (4)$$

Where w was the initial weight of butyl rubber waste power (5 grams). The initial waste rubber CLD was measured as 281 mol/m³ (Table 1).

The relationship between sol fraction and CLD may express by Horikx analysis [1]. The relative decrease in CLD when main chain scission occurs [22]:

$$1 - \frac{\vartheta_f}{\vartheta_i} = 1 - \left[\frac{(1 - s_f^{0.5})^2}{(1 - s_i^{0.5})^2} \right] \quad (5)$$

And when only crosslink scission takes place during de-crosslinking:

$$1 - \frac{\vartheta_f}{\vartheta_i} = 1 - \left[\frac{\gamma_f (1 - s_f^{0.5})^2}{\gamma_i (1 - s_i^{0.5})^2} \right] \quad (6)$$

Where s_i , s_f , ϑ_i , ϑ_f , γ_i , and γ_f are sol fractions before and after de-crosslinking, CLD's before and after de-crosslinking, and the average number of crosslinks per chain in the remaining insoluble rubber network before and after de-crosslinking, respectively. In

$$S = \frac{[(2+\gamma) - (\gamma^2 + 4\gamma)^{0.5}]}{2\gamma} \quad (7)$$

By substituting the s_i (0.07) in equation 7, the value of γ_i was calculated as 3. Also, by replacing the calculated values of γ_i and s_i in equations 5 and 6 and with the help of equation 7, the Horikx curves were drawn as solid and dotted curves in correspondent figures.

The surfaces of the de-crosslink rubber compounds assessed by a LEO 1530 VP field emission gun scanning electron microscope (SEM). Small pieces of the rubber samples, with the approximate dimensions of 10 by 10 mm coated with gold and subsequently photographed in the SEM. The obtained SEM micrographs used for the morphology study of the rubber compounds.

3. Results and Discussion

3.1. The Used Waste Butyl Rubber Composition

As mentioned, the employed waste butyl rubber was the end of life

this study, s_i and ϑ_i were measured as 0.07 and 281 mol/m³ (Table 1), respectively. Equation 7 expresses the relationship between γ and s .

rubber made bladder which used for curing the tire in the mould. The size distribution and mean size of the rubber powder particles are key factors for assessment of the de-crosslinking efficiency [13].

For particles with larger diameter than 0.5 cm, the hot spots appear in rubber sample during microwave irradiation. The above mentioned phenomena makes a temperature profile in the bulk of the sample resulting in heterogeneous de-crosslinking of the rubber sample. To avoid of the mentioned difficulty, the waste rubber grinded to produce powder with a median size of 371 microns (Figure 1). As observed, 25, 43, 64, and 86 volume % of the waste rubber particles had diameters less than 183, 293, 469, and 750 microns, respectively. Also, the minimum and the maximum particle diameter were 28 and 1518 microns, respectively.

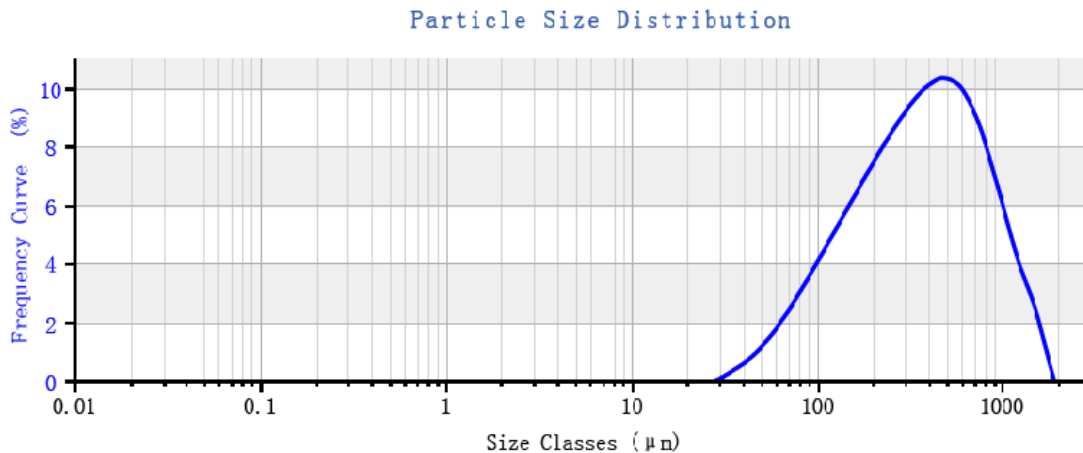


Figure 1: The Cured Rubber Made Waste Bladder Powder Size Distribution. The Particles Dispersed in the Water Before Size Distribution Measurement.

Table 1 represents the composition of the waste butyl rubber obtained from TGA curves (Figure 2). As shown, the oil content and other light hydrocarbons begin to degrade at ambient temperature to 340 °C with about 6% mass loss of the rubber sample. Also, the rubber began to degrade from 340 °C and ended at 455 °C just

before changing the atmosphere from nitrogen to air. The mass loss for rubber was 54%. The attributed mass loss for carbon black (filler) was 17% at the temperature range 455 to 545°C. The left mass beyond this temperature was unburned residues including minerals with 23% mass loss.

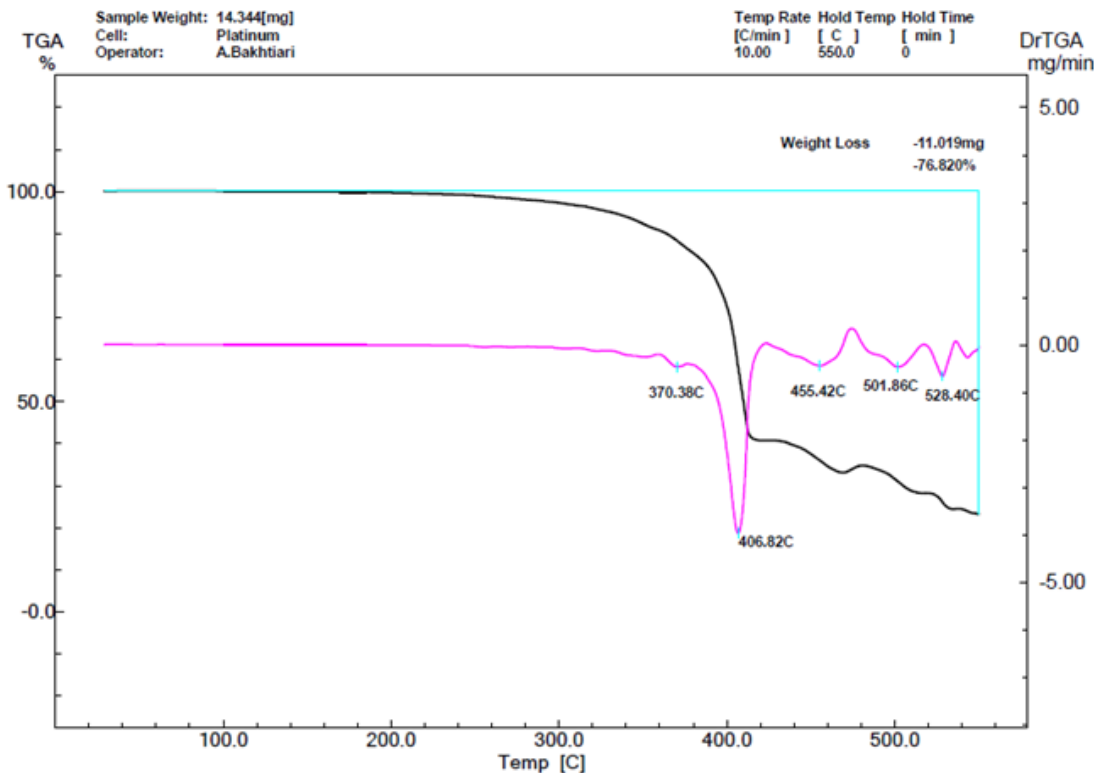


Figure 2: The Thermo Gravimetric Analysis (TGA) Curves for the Cured Rubber Made Bladder Waste Powder.

The Fourier transform infrared spectroscopy (FTIR) graphs used to identify the existence of the phenolic resin in the structure of the waste bladder butyl rubber powder (Figure 3). As observed, the appeared peaks at 3412, 1539, 1621, 1180, and 715 cm^{-1} attributed to phenolic hydroxyl (OH), C=C in aromatic ring, the original IR

band of the aromatic ring, C-O-C (ether crosslink), and the C-H bending in the aromatic ring of the phenolic resin, respectively [3]. However, the intensities of the latter two peaks were extremely lower than the former peaks.

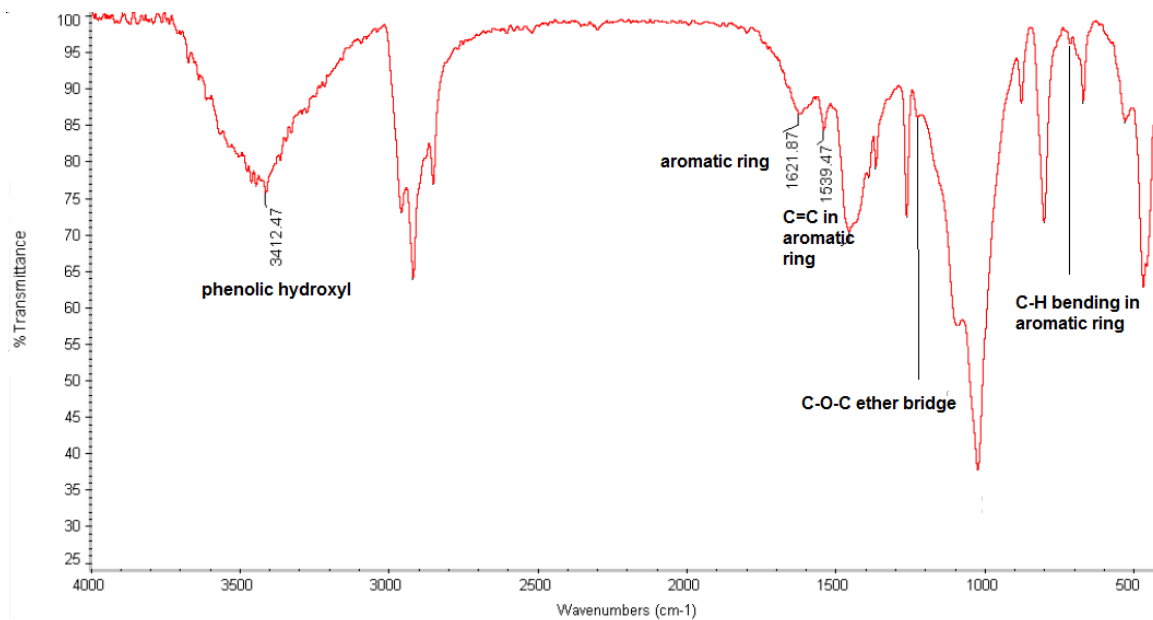


Figure 3: The FTIR Graph for the Used Waste Butyl Rubber Powder.

3.2. Devulcanization of the Waste Butyl Rubber Powder

During microwave irradiation, the apparent state of the rubber powder changed from a solid particle to a bulk and sticky state. It was a good evidence for de-crosslinking of the rubber powder.

It was in conformity with the measured de-crosslinking %'s for the treated compounds (Table 2) and also, the SEM micrographs in Figure 4.

| Compound | De-crosslinking agent | De-crosslinking temperature (°C) | Sol fraction (%) | CLD (mol/m^3) | De-crosslinking (%) |
|----------|-----------------------|----------------------------------|------------------|-------------------|---------------------|
| RC | --- | 180 | 18.0 | 108.1 | 62.0 |
| A | MBT | 180 | 22.0 | 83.4 | 70.3 |
| B | MBTS | 180 | 20.6 | 80.1 | 71.5 |
| C | TMTD | 180 | 20.8 | 84.1 | 70.1 |
| D | TBBS | 180 | 17.4 | 95.9 | 65.8 |
| E | DCBS | 180 | 17.6 | 110.7 | 60.5 |
| F | DPTT | 180 | 24.6 | 88.7 | 68.4 |
| G | DPDS | 180 | 28.0 | 61.4 | 78.1 |
| H | DPDS | 180 | 20.4 | 85.1 | 69.7 |
| I | DPDS | 180 | 27.6 | 60.4 | 78.5 |
| J | DPDS | 180 | 31.6 | 57.8 | 79.4 |
| K | DPDS | 180 | 28.0 | 56.4 | 79.9 |
| J-1 | DPDS | 200 | 24.6 | 47.2 | 83.2 |
| J-2 | DPDS | 220 | 25.2 | 49.4 | 82.4 |
| J-3 | DPDS | 240 | 24.4 | 50.0 | 82.2 |
| J-4 | DPDS | 250 | 24.4 | 58.9 | 79.0 |

Table 2: De-Crosslinking Parameters, Sol Fraction, Crosslink Density (CLD), and De-Crosslinking % of the Microwave Irradiated Compounds.

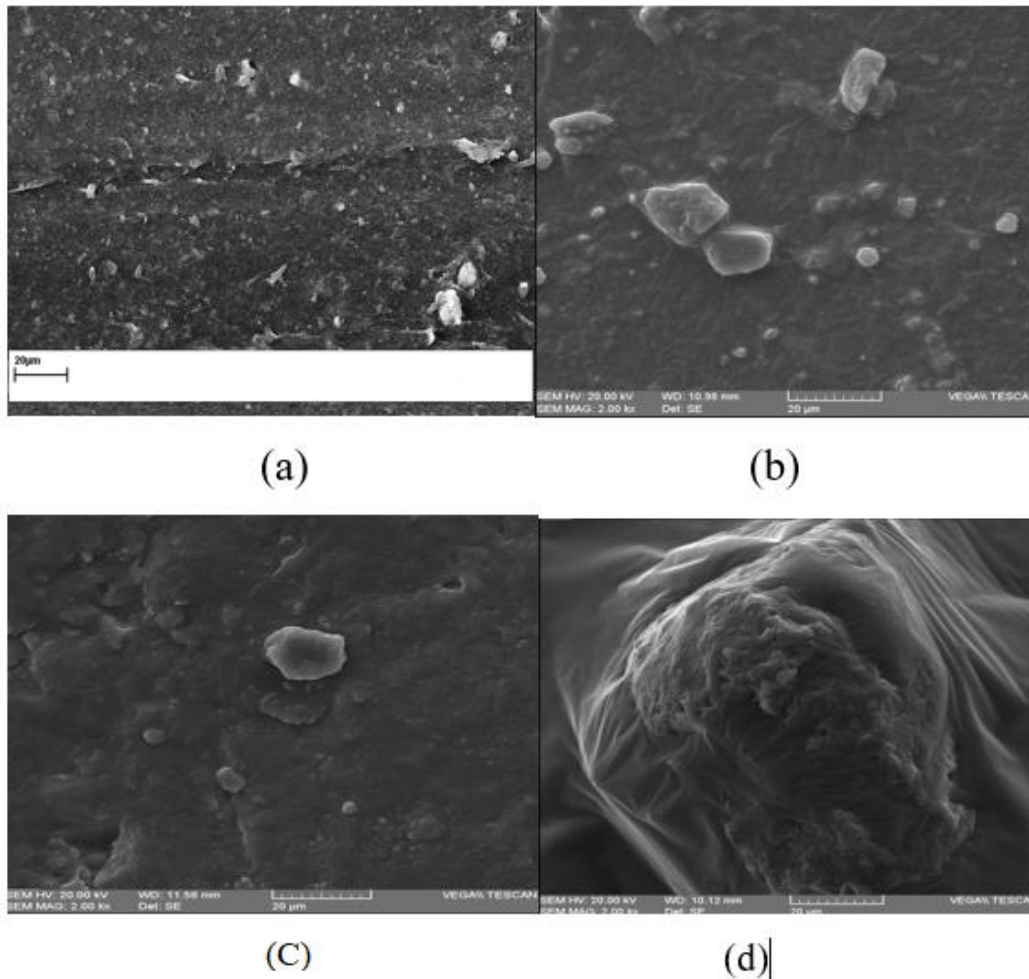


Figure 4: The SEM Micrographs Showing the Interior of the
 (a) Waste butyl rubber powder
 (b) Reference compound, RC
 (c) Compound E with the lowest de-crosslinking %

Figure 4-a shows the internal structure of the waste rubber powder. The carbon black (filler) dispersed in the rubber matrix unproperly with the average diameter of 7 microns. However, after only microwave irradiation without adding any de-crosslinking agent (Figure 4-b, for reference compound, Table 3), the agglomeration of the filler observed and the average size of the filler increased to 15 microns. Also, the shape of the rubber matrix changed from a course to a relatively soft state. The formed de-crosslink rubber covered the surface of the most filler particles better than the available cross linked rubber in initial waste rubber powder. Approximately, the same situation observed for the SEM micrograph (Figure 4-C) of the Compound E with the lowest de-crosslinking % (Table 2). However, the situation was absolutely different for the Compound j-1 with de-crosslinking 83.2 % as the

highest de-crosslinking % among the treated compounds (Table 2). As observed, the border between the formed de-crosslink rubber and the filler disappeared. The rubber covered the filler particles appropriately. It was due to high mobility of the de-crosslink rubber which enable it to make high bound rubber with the filler particles.

The sol fractions versus relative reduction in crosslink densities (CLD's) or $(1-\theta_i/\theta_0)$ for microwave irradiated rubber compounds at 180 °C prepared with different de-crosslinking agents, DPDS, MBT, TMTD, MBTS, DPTT, TBBS, DCBS and reference compound without de-crosslinking agent (Compounds G, A, C, B, F, D, E, and RC, Table 3) shown in Figure 5

| Compound number | Waste rubber (phr) | DPDS* | TMTD* | DPTT* | MBTS* | DCBS* | TBBS* | MBT* | Aromatic oil** |
|-----------------|--------------------|-------|-------|-------|-------|-------|-------|------|----------------|
| Reference (RC) | 100 | | | | | | | | 30 |
| A | 100 | | | | | | | 4 | 30 |
| B | 100 | | | | 4 | | | | 30 |
| C | 100 | | 4 | | | | | | 30 |
| D | 100 | | | | | | 4 | | 30 |
| E | 100 | | | | | 4 | | | 30 |
| F | 100 | | | 4 | | | | | 30 |
| G | 100 | 4 | | | | | | | 30 |
| H | 100 | 2 | | | | | | | 30 |
| I | 100 | 6 | | | | | | | 30 |
| J | 100 | 8 | | | | | | | 30 |
| K | 100 | 10 | | | | | | | 30 |

* De-crosslinking agent (phr).

** Processing aid (phr).

Table 3: The Compound's Formulation for De-Crosslinking of the Waste Butyl Rubber Powder. De-Crosslinking Took Place Inside of Microwave Oven with 100% Output Power at 180 °C .

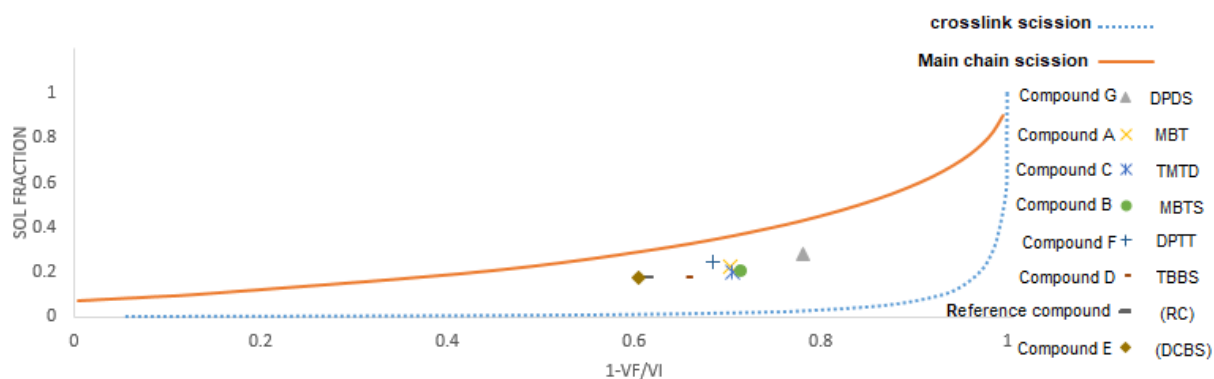


Figure 5: The Relative Decrease in CLD versus the Sol Fraction of Compounds A to G and Reference Compound. De-Crosslinking Took Place Inside of Microwave Oven with 100% Output Power at 180 °C.

For preparing the above mentioned compounds, 4 phr from each de-crosslinking agent along with 30 phr aromatic oil as processing aid were used. Based on the Horikx analysis, the solid (Equation 5) and dotted (Equation 6) curves in Figure 5 represent the complete

main macromolecule chain scission and the crosslink scission, respectively. Lying down the points between the above mentioned curves indicates during de-crosslinking process not only crosslinks but also the main chain of the macromolecule breaks down. The

former is pleasant and the latter is not desired. As much as closer the points to the dotted curve represents the more efficient de-crosslinking process.

Figure 5 shows during de-crosslinking process crosslinks and main chain scission cause for all studied de-crosslinking agents. However, the highest de-crosslinking % with the value of 78.1 % (Table 2) observed for Compound G with the DPDS in composition. Table 2 shows that the de-crosslinking % for the RC was 62%. The de-crosslinking %'s of the left compounds ranged between 60.5% (Compound E with DCBS) to 71.5% (Compound B with MBTS). It concluded, the DPDS was the most suitable de-crosslinking agent among of the studied agents.

In general, the CLD reduction is accompanied with the increase in rubber sol content (%) [1, 23]. The reason refers to the extent to which the rubber main chains and crosslinks are broken. As an illustration, the sol fraction and CLD for the RC were 108.1 mol/m³ and 18%, respectively. They reduced and increased to the values of 61.4 mol/m³ and 28 % for Compound G (Table 2). The relative decrease in CLD versus the sol fraction of the compounds with different amounts of DPDS, 2, 4, 6, 8, and 10phr (Compounds H, G, I, J, and K, Table 3) depicted in Figure 6.

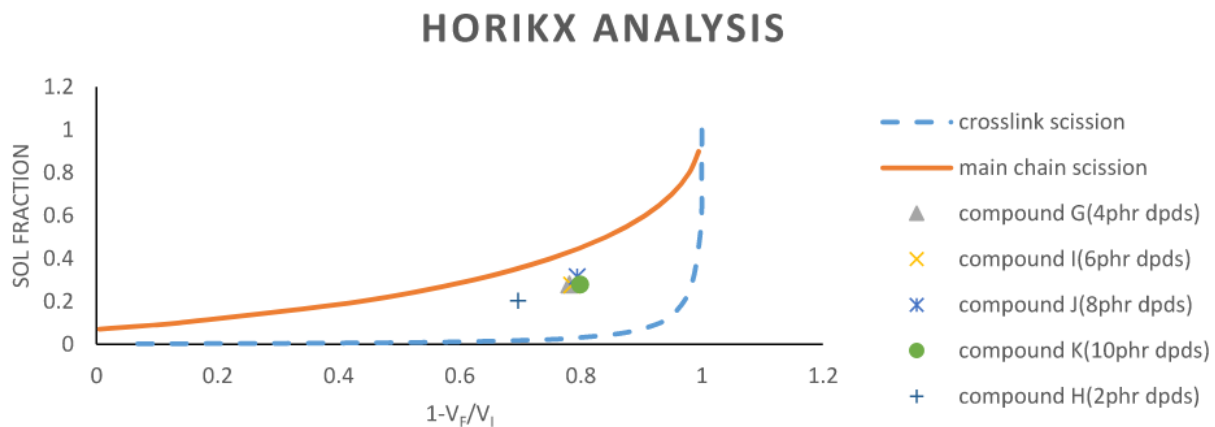


Figure 6: The Relative Decrease in CLD versus the Sol Fraction of the Compounds with Different Amount of DPDS. De-Crosslinking Took Place Inside of Microwave Oven with 100% Output Power at 180 °C.

The de-crosslinking took place inside of the microwave oven with 100% output power at 180 °C. As observed and with the except for Compound H, the points for the left compounds coincided on each other approximately. Also, both crosslink and main chain scission observed for all compounds with different amount of DPDS in formulation. The correspondent de-crosslinking %'s were 69.7, 78.1, 78.5, 79.4, and 79.9% for Compounds H, G, I, J, and K, respectively (Table 2). The increase of DPDS had little effect on de-crosslinking % beyond the DPDS values of 4phr. The same fact was true for the sol fraction of the mentioned compounds. With considering the applicability of the outcomes of the research for commercial use, Compound J selected for further experiments. Table 2 shows the de-crosslinking parameters for Compound J with microwave irradiated at different temperatures 180, 200, 220,

240, and 250 °C (Compounds J, J-1, J-2, J-3, and J-4, Table 2). As observed, increasing the irradiation temperature had positive effect on the de-crosslinking % with the except for irradiated compound at 250 °C (Compound J-4). It seemed beyond this temperature, the crosslinking reactions re-accomplish gradually [24]. The highest de-crosslinking % belonged to Compound J-1 irradiated at 200 °C with the value of 83.2%. Also, increasing the temperature had adverse effect on the sol fraction and reduced it from 31.6 % for Compound J to the values of 24.6, 25.2, 24.4, and 24.4 % for the Compounds J-1 to J-4, respectively. The Horikx analysis showed that there was not remarkable difference between the results for the above mentioned compounds (Figure 7). It concluded, the increasing in microwave irradiation temperature had equal effect on crosslink and main chain scission.

HORIKX ANALYSIS

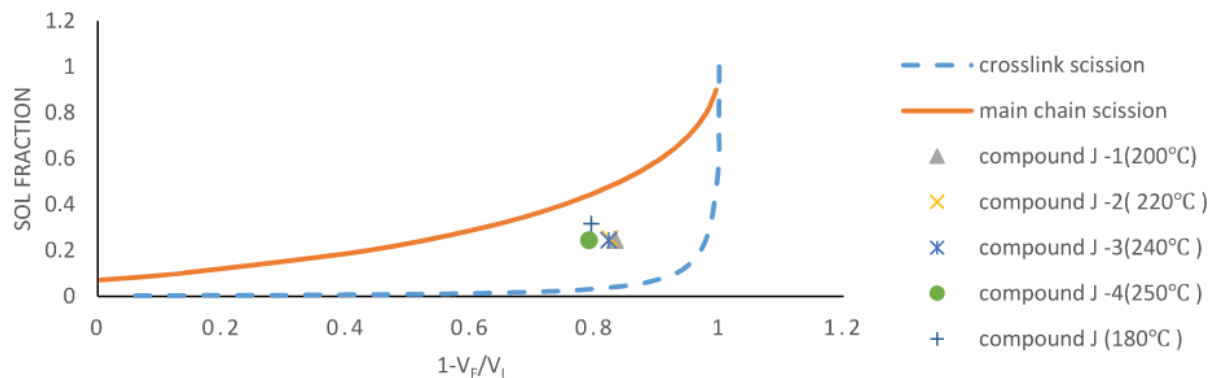


Figure 7: The Relative Decrease in CLD versus the Sol Fraction of the Compounds J and J-1 to J-4. De-Crosslinking Took Place Inside of Microwave Oven with 100% Output Power at Different Temperatures.

4. Conclusions

The Fourier transform infrared spectroscopy (FTIR) graphs were suitable evidences to prove the existence of the phenolic resin in the structure of the used waste bladder butyl rubber powder. Efficient de-crosslinking observed for all studied de-crosslinking agents. It was evidenced by the SEM micrographs and also, the measured crosslink densities of the reclaimed rubbers. The highest and the lowest de-crosslinking %'s belonged to the compounds with DPDS and DCBS in formulation, respectively. Based on the obtained de-crosslinking parameters, the DPDS selected as a suitable de-crosslinking agent for the used waste bladder butyl rubber. In accordance with Horikx analysis, the microwave irradiation broke down the crosslinks and rubber main chains simultaneously. The increasing in microwave irradiation temperature had positive effect on the de-crosslinking process but it had equal effect on crosslink and main chain scission.

Acknowledgment

The authors sincerely thank the staffs of the polymer chemistry laboratory located at faculty of science, Ferdowsi University of Mashhad for their sincere cooperation. Approval no. 3/53623.

References

1. Khavarnia, M., & Movahed, S. O. (2016). Butyl rubber reclamation by combined microwave radiation and chemical reagents. *Journal of Applied Polymer Science*, 133(17).
2. Ciullo, P. A., & Hewitt, N. (1999). *The rubber formulary*. William Andrew.
3. Imanifar, M., Ostad Movahed, S., & Ahmadpour, A. (2018). Effects of peroxide and phenolic cure systems on characteristics of the filled ethylene-propylene-diene monomer rubber (EPDM). *Journal of Applied Polymer Science*, 135(21), 46213.
4. Pistor, V., Scuracchio, C. H., Oliveira, P. J., Fiorio, R., & Zattera, A. J. (2011). Devulcanization of ethylene-propylene-diene polymer residues by microwave—Influence of the presence of paraffinic oil. *Polymer Engineering & Science*, 51(4), 697-703.
5. Bani, A., Polacco, G., & Gallone, G. (2011). Microwave-induced devulcanization for poly (ethylene-propylene-diene) recycling. *Journal of Applied Polymer Science*, 120(5), 2904-2911.
6. Caron, P. A., Larreteguy, A. E., & Porta, P. F. (2017). Cure kinetics of butyl rubber cured by phenol formaldehyde resin. *Latin American Applied Research*, 47(2), 59-64.
7. Lattimer, R. P., Kinsey, R. A., Layer, R. W., & Rhee, C. K. (1989). The mechanism of phenolic resin vulcanization of unsaturated elastomers. *Rubber chemistry and technology*, 62(1), 107-123.
8. Myhre, M., Saiwari, S., Dierkes, W., & Noordermeer, J. (2012). Rubber recycling: chemistry, processing, and applications. *Rubber chemistry and technology*, 85(3), 408-449.
9. Raju, F. (Ed.). (2017). *Recycling of polymers: methods, characterization and applications*. Wiley-VCH Verlag.
10. Movahed, S. O., Ansarifard, A., Karbalaee, S., & Far, S. A. (2015). Devulcanization and recycling of waste automotive EPDM rubber powder by using shearing action and chemical additive. *Progress in Rubber Plastics and Recycling Technology*, 31(2), 87-116.
11. Movahed, S. O., Ansarifard, A., & Estagy, S. (2016). Review of the reclaiming of rubber waste and recent work on the recycling of ethylene-propylene-diene rubber waste. *Rubber Chemistry and Technology*, 89(1), 54-78.
12. Mohaved, S. O., Ansarifard, A., Nezhad, S. K., & Atharyfar, S. (2015). A novel industrial technique for recycling ethylene-propylene-diene waste rubber. *Polymer Degradation and Stability*, 111, 114-123.
13. Movahed, S. O., Ansarifard, A., Zohuri, G., Ghaneie, N., & Kermany, Y. (2016). Devulcanization of ethylene-propylene-diene waste rubber by microwaves and chemical agents. *Journal of Elastomers & Plastics*, 48(2), 122-144.
14. Jiang, K., Shi, J., Ge, Y., Zou, R., Yao, P., Li, X., & Zhang,

-
- L. (2013). Complete devulcanization of sulfur-cured butyl rubber by using supercritical carbon dioxide. *Journal of applied polymer science*, 127(4), 2397-2406.
15. Landini, L., de Araújo, S. G., Lugão, A. B., & Wiebeck, H. (2007). Preliminary analysis to BIIR recovery using the microwave process. *European Polymer Journal*, 43(6), 2725-2731.
16. Feng, W., & Isayev, A. I. (2004). Continuous ultrasonic devulcanization of unfilled butyl rubber. *Journal of applied polymer science*, 94(3), 1316-1325.
17. Feng, W., Isayev, A. I., & Von Meerwall, E. (2004). Molecular mobility in ultrasonically treated butyl gum and devulcanized butyl rubber. *Polymer*, 45(25), 8459-8467.
18. Karmanova, O. V., Tikhomirov, S. G., Kayushnikov, S. N., Shashok, Z. S., & Polevoy, P. S. (2019). Obtaining and using of reclaimed butyl rubber with the use of ionizing radiation. *Radiation Physics and Chemistry*, 159, 154-158.
19. Tikhomirov, S. G., Polevoy, P. S., Semenov, M. E., & Karmanov, A. V. (2019). Modeling of the destruction process of butyl rubber. *Radiation physics and chemistry*, 158, 205-208.
20. Vagizova, R. R., Khakimullin, Y. N., Makarov, T. V., Vol'fson, S. I., Stepanov, P. A., & Palyutin, F. M. (2008). Thermal stability of compounds based on butyl rubber reclaim obtained under radiation. *International Polymer Science and Technology*, 35(3), 7-9.
21. Zhang, T., Xing, Z., Zhang, L., & Zhang, Y. (2018). Recycling of resin cured IIR-based ground bladder rubber with the assistance of subcritical fluids. *Journal of Elastomers & Plastics*, 50(8), 677-696.
22. Saiwari, S., Dierkes, W. K., & Noordermeer, J. W. (2014). Comparative investigation of the devulcanization parameters of tire rubbers. *Rubber chemistry and technology*, 87(1), 31-42.
23. Molanorouzi, M., & Mohaved, S. O. (2016). Reclaiming waste tire rubber by an irradiation technique. *Polymer Degradation and Stability*, 128, 115-125.
24. Liu, Z., Li, X., Xu, X., Wang, X., Dong, C., Liu, F., & Wei, W. (2015). Devulcanization of waste tread rubber in supercritical carbon dioxide: operating parameters and product characterization. *Polymer Degradation and Stability*, 119, 198-207.

Copyright: ©2023 Saeed Ostad Movahed, et al. This is an open-access article distributed under the terms of the Creative Commons Attribution License, which permits unrestricted use, distribution, and reproduction in any medium, provided the original author and source are credited.