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Devulcanization of natural rubber industry waste in supercritical carbon dioxide combined with diphenyl disulfide

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ABSTRACT

The elimination of rubber wastes without affecting the environment is one of the most important challenges of the 21st century waste management. Accordingly, the present work is focused on the recycling of natural rubber (NR) industry waste by means of devulcanization in supercritical carbon dioxide (scCO₂) atmosphere. With that aim, a novel device allowing to perform rubber devulcanization was developed. It consists of a triaxial compression reactor integrated into a dynamic hydraulic universal testing machine with a heating chamber. NR industry waste was devulcanized in the mentioned device at different temperatures, in scCO₂ by using diphenyl disulfide (DD) as devulcanizing reagent. The devulcanization degree and quality of the treated materials were evaluated by the swelling test combined with the Horikx theory. It was appeared that a successful devulcanization, with almost no degradation, was obtained, and the devulcanization degree reached maximum value of ~90 %. Thermogravimetric tests and scanning electron microscopy

(SEM) images strengthened these results. Finally, it was concluded that the developed device is appropriate to perform rubber recycling, which contributes to the progress in the environmental protection.

Keywords: Recycling, natural rubber, devulcanization, supercritical CO₂, diphenyl disulfide.

1. Introduction

Rubber parts and tires manufacturing industries are constantly growing. Nonetheless, the inability to recycle their wastes represents a thorny environmental problem (Liu et al., 2020; Sabzekar et al., 2016). Because of the three dimensional structure of rubbers and their composition that includes numerous components, these kinds of polymers are indeed difficult to recycle (Hejna et al., 2020; Sienkiewicz et al., 2017; Kroll and Hoyer, 2019). One of the alternatives to throw off the mentioned wastes is to accumulate them in stocks, however they are focus of illnesses and fires (Adhikari et al., 2000; Mohajerani et al., 2020). Another way is to use them as an energy source by burning them, but this route is hazardous and generates environmental contamination (Molino et al., 2018; Pehlken and Müller, 2009). Consequently, these two methods are not recommended. With the aim of reducing this environmental problematic, several processes have been developed, especially the devulcanization technique (Asaro et al., 2018; Cheng et al., 2018 ; Bockstal et al., 2019), during the last few decades. Rubber devulcanization is a process where poly-, di-, and mono-sulfidic bonds, formed during vulcanization, are totally or partially broken, i.e. a process that causes a selective breakup of sulfidic bonds without degrading the polymer (Isayev, 2013; Asaro et al., 2018). Performing a successful devulcanization is a great

challenge, since the energies needed to break the sulfur-sulfur (S-S) and carbon-sulfur (C-S) bonds (227 and 273 kJ/mol, respectively) are not so different from the energy required to break the rubber main chains i.e. the carbon-carbon (C-C) bonds (348 kJ/mol) (Rooj et al., 2011; Karger-Kocsis et al., 2013; Ramarad et al., 2015; Sabzekar et al., 2015). Several devulcanization processes have been developed and presented in the literature, including: biological (Yuanhu et al., 2011; Yao et al., 2013), chemical (le Beau, 1967; Zhang et al., 2018; Anu Mary et al., 2016), ultrasound (Isayev et al., 2014; Mangili et al., 2015; Hon et al., 2015), microwave (Novotny et al., 1978; Aoudia et al., 2017; Simon et al., 2020), mechanochemical (Jana et.al, 2005), and thermo-mechanical (Seghar et al., 2019b; Diaz et al., 2018). Usually, the mentioned devulcanization processes are followed by the characterization of the obtained materials. The aforementioned characterization serves both to evaluate the devulcanized material as well as the devulcanization process. Because direct access to the changes generated by the devulcanization process at molecular level is non-trivial for rubber materials, an indirect method is required. The most widely used indirect technique is based on the swelling test (ASTM D 6814-02). Through this test, it is possible to measure the crosslink density of the devulcanized material and compare it with that of the virgin vulcanized rubber. Then, the degree of devulcanization is estimated as the decrease in the material crosslink density. Other characteristics such as the degree of swelling, the soluble and gel fractions, etc., are also obtained by this technique. It is worth mentioning that the swelling test results are also used to perform the Horikx chart (Horikx, 1956). This diagram allows analyzing the quality of the performed devulcanization by means of crosslink scission versus main chain scission. The development of the Horikx theory (Horikx, 1956) is based on the work of Charlesby (Charlesby, 1953). It is well developed in the literature (Verbruggen et al., 1999), and its application in the rubber

devulcanization analysis was recently experimentally proved (Verbruggen et al., 2016; Seghar et al., 2019a).

The chemical devulcanization in a pressurized reactor in scCO₂ atmosphere has been already analyzed. It is expected that, during the devulcanization process, the supercritical carbon dioxide swell the polymer, facilitating the sulfidic bonds scission. A more detailed explanation of the devulcanization mechanism in scCO₂ is given afterwards. Kojima et al. (Kojima et al., 2003; Kojima et al., 2004; Kojima et al., 2005a; Kojima et al., 2005b) have devulcanized different kinds of rubbers in an autoclave, with supercritical carbon dioxide (scCO₂) and diphenyl disulfide (DD) as devulcanizing reagents, among others. The most suitable conditions for the devulcanization of the sulfur-cured unfilled natural rubber (NR) were 60 min at 10 MPa and 180 °C, with 11wt% of DD. They concluded that scCO₂ helps in the penetration of the devulcanizing reagent DD into the rubber. In order to understand the reaction that occurs between DD and the rubber during the devulcanization, they proposed a reaction mechanism scheme that is presented in Fig. 1. It was pointed out that, firstly, the DD molecules solvated by the scCO₂ will get into the NR previously swollen by the scCO₂. Then, these molecules will contribute to the sulfur bonds cleavage, giving as a result a devulcanized rubber.

The analysis of the devulcanized material was based on the variation of the sol fraction content. Jiang et al. (Jiang et al., 2013) studied the devulcanization of sulfur-cured butyl rubber in a high-pressure reactor with scCO₂, by using DD as a devulcanizing reagent. The devulcanization degree of the treated materials was evaluated with respect to the sol content. It was pointed out that the revulcanization of the recycled material could be affected by the amount of DD used during the devulcanization process, and therefore excessive quantities of DD should be avoided. A devulcanization mechanism, including the

reaction between the butyl rubber and DD, and the material thermal degradation, was proposed. Mangili et al. (Mangili et al., 2014a; Mangili et al., 2014b) analyzed the devulcanization of ground tire rubber (GTR) with scCO₂, using DD as devulcanizing reagent. DD fraction, temperature and their interaction were identified as the main parameters affecting the devulcanization process. The devulcanization degree, obtained by swelling measurements, was about 50 %. It was shown that the use of DD in the devulcanization process favors the increase of the strain at break and the decrease of the Young modulus of the blends containing the devulcanized GTR. The devulcanization of waste tire rubber (WTR) by using scCO₂ as reaction medium and DD as devulcanizing reagent, in an autoclave, was also performed by Liu et al. (Liu et al., 2015). The effect of time, temperature, pressure, and DD concentration on the devulcanization process were studied. In each case, the quality of the devulcanization was analyzed using the Horikx theory (Horikx, 1956). It was found that the concentration of DD was the most important variable during the process. It was also shown that an excessive increase in time and temperature just lead to polymer degradation. A combination between sulfur bond scission and main chain scission, commonly named regeneration, was obtained in all cases, with a maximum devulcanization degree near to 50 %.

Despite the several works carried out on the static devulcanization of rubber in scCO₂ atmosphere with DD, to the best of our knowledge, no published paper has reported the achievement of a high devulcanization degree of good quality, i.e. mostly scission of the sulfur-sulfur bonds with little degradation of the polymer.

The aim of this work is to develop a novel device allowing to perform successful rubber devulcanization, under scCO₂ atmosphere. In fact, a triaxial compression reactor was developed. It is integrated into a dynamic hydraulic universal testing machine equipped

with a heating chamber. Rubber devulcanization tests were carried out in this reactor, under scCO_2 atmosphere, using DD as devulcanizing reagent, at different temperatures. The degree and quality of devulcanization of the treated materials were evaluated by the swelling test combined with the Horikx theory. The mentioned theory was recently proved as efficient and as one of the fundamental tools in the devulcanization analysis (Seghar et al., 2019a). It was found that, when optimal conditions are used in the developed device, the degree of successful devulcanization reaches 90 %. To the extent of our knowledge, no similar results have been reported in the literature, using a comparable devulcanization process.

2. Experimental

2.1. Devulcanization equipment

A novel laboratory device allowing to perform rubber devulcanization tests was developed (Fig. 2). It mainly consists of a cylinder and a piston that let to apply triaxial compression on the rubber to be devulcanized. This reactor is integrated into a dynamic hydraulic universal testing machine (100 kN) equipped with a heating chamber. It allows to contain the rubber particles in a controlled atmosphere, in our case CO_2 , and all necessary devulcanizing reagents. The compression loading generated by the testing machine results in an increase of the pressure of the gas surrounding the rubber particles, turning the CO_2 from the gaseous state to the supercritical state. The main idea is to devulcanize the rubber by submitting it to an equi-triaxial mechanical stress (hydrostatic pressure loading) and by using small quantities of CO_2 (less than 5 wt%).

As shown in Fig. 2, the pressure inside the reactor is controlled by the movement of a piston (38 mm diameter), using the testing machine software (WaveMatrix). This movement has to be gradual, during unloading, to avoid the rapid gas decompression that could damage the rubber seals and the studied rubbers. The procedure followed to obtain a desired pressure by moving the piston can be summarized as follows.

First two compression loading tests were carried out, with and without CO₂ in the reactor. From the difference between the obtained force-displacement curves, the experimental gas pressure-full load curve was deduced. Then, the force to be imposed to reach a desired gas pressure was obtained from the mentioned curve. Finally, the piston was moved till reached the mentioned force, and the gas pressure was satisfactorily achieved.

It was assumed that, during the fast compression of the CO₂ and the rubber particles, the rubber remained unchanged.

The initial CO₂ pressure, before moving the piston, was calculated using the CO₂ state law proposed by Duschek et al. (Duschek et al., 1990), knowing the mass of CO₂ and the empty volume of the reactor cavity. Hypotheses of rubber incompressibility and isothermal transformations were considered.

The parameters that can be controlled during a test are: CO₂ content, CO₂ pressure, temperature (up to 160 °C), amount of rubber, amount of devulcanizing reagent, time, and compression loading and unloading steps.

2.2. Materials and devulcanization process

Sulfur-cured NR was obtained from the production waste of an industry. The requirements for the parts made with this material were high elasticity and high resistance to fatigue. The global formulation of the mentioned NR is detailed in Table 2. The rubber was used as

received, in the form of particles. The average size of the particles, determined by sieving was less than 4 mm. Because of confidentiality reasons, no further details about the studied material were provided by our industrial partner.

Diphenyl disulfide (DD) purchased from Sigma Aldrich Corporation was used as devulcanizing reagent in a proportion of 15 wt% in all cases. This proportion is similar to those already considered in the literature (Kojima et al., 2004; Kojima et al., 2005b; Jiang et al., 2012; Mangili et al., 2014b). The amount of DD actually consumed during the devulcanization process was not measured. Taking into account that excessive DD could affect the revulcanization process, measurement and recovery of the eventual unused DD reminds as a challenge to be addressed. DD chemical structure is given in Fig. 3.

The reaction medium during the devulcanization was CO₂ because its supercritical state is easy to reach (31.1 °C and 7.38 MPa), it is non-toxic, cheap, easily removable from the rubber, and non-flammable (Kojima et al., 2003; Ikeda, 2014). In the supercritical state the CO₂ has high diffusivity and acts as an excellent swelling agent in the rubber, which helps in the devulcanization process (Mueller et al., 2005; Liu et al., 2015; D'hooge et al., 2016). The purity of the used CO₂ was $\geq 99.7\%$.

Before running the devulcanization test, 20 g of NR were mixed with 3 g of DD and placed in the cavity of the reactor, at room temperature. Then the device was closed, positioned in the machine chamber and heated to the desired temperature. The air inside the reactor was removed using a vacuum pump, and then the CO₂ was injected. Afterwards, the compression ramp (Fig. 4) was executed in order to increase the pressure and allow the CO₂ to become supercritical. The imposed force was equal to -18 kN, inducing a pressure of 12 MPa inside the reactor. Tests were carried out at 120, 140 and 160 °C, during 1 hour. At the

end of each test, the system was slowly decompressed and allowed to cool to room temperature. The treated rubber was extracted from the device in the form of one agglomerate piece. The devulcanization parameters (temperature and time) were chosen based on the data found in the literature (Kojima et al., 2004; Kojima et al., 2005b; Jiang et al., 2012; Mangili et al., 2014b), seeking to perform a satisfactory devulcanization by consuming a reasonable amount of energy. Greater temperature and/or time were supposed to be harsh conditions to be used in NR devulcanization tests, as they would induce material degradation.

2.3. Characterization

2.3.1. Evaluation of the devulcanization degree

The devulcanization degree was evaluated by the swelling test, according to the Standard ASTM D 6814-02. Conforming to the procedure, the swelling was carried out after washing the rubber with acetone, in a 250 ml soxhlet extractor, during 16 h (Standard ASTM D297-02). Afterwards, the specimen was dried and weighed (m_i), and then swelled in toluene at ambient temperature, during 72 h. The solvent was refreshed every 24 h. The sample impregnated with toluene was weighed (m_t) and then dried in an oven at 70 °C during a night. The dried sample was finally weighed (m_d). Three samples were tested for each material.

The soluble fraction %S was evaluated using the following equation:

$$\%S = \frac{m_i - m_d}{m_i} \times 100 \quad (1)$$

The Flory-Rehner equation was used for the evaluation of the crosslink density ν_e :

$$v_e = \frac{-[\ln(1-V_r)+V_r+\chi V_r^2]}{[V_1 (V_r^{1/3}-V_r/2)]} \quad (2)$$

where V_r is the rubber volume fraction in the swollen sample, χ is the rubber-solvent interaction parameter, and V_1 is the molar volume of toluene. It is worth noting that $\chi=0.391$ for NR-toluene interaction. This value was obtained from the Standard ASTM D 6814-02 and it has been already used in the literature (Kojima et al., 2004; Lamminmaki et al., 2006; Ghorai et al., 2016). $V_1=106.3$ mL/mol.

V_r was calculated using the following equation:

$$V_r = \frac{m_d/\rho_d}{m_d/\rho_d + m_s/\rho_s} \quad (3)$$

where m_d is the mass of the dried rubber, ρ_d is the density of this rubber, m_s is the mass of the solvent absorbed by the sample, in our case toluene, and ρ_s its density at room temperature. The density of the dried rubber (ρ_d) was evaluated by hydrostatic weighing procedure as explained in the Standard ASTM D 6814-02.

Finally, the devulcanization degree, %*Devulc.*, was evaluated for each sample, as follows:

$$\%Devulc. = \left[1 - \frac{v_f}{v_i}\right] \quad (4)$$

where v_i is the crosslink density of the rubber before devulcanization, and v_f is the crosslink density after devulcanization. v_i and v_f were estimated with Eq. 2.

The reported values are the averages of the results obtained with the three tested samples.

2.3.2. Horikx analysis

The Horikx diagram (Horikx, 1956) allows to carry out an accurate analysis of the performed devulcanization. In fact, in this chart, the devulcanization degree (Eq. 4) and the

soluble fraction (Eq. 1) of the rubber are related, so that both the degree and quality of devulcanization, in terms of crosslink against main chain scission, can be qualitatively estimated.

The theoretical variation of the soluble fraction is shown in Fig. 5, for the tested NR, as a function of the devulcanization degree. The dashed line corresponds to only main chain scission, and the solid line corresponds to only selective crosslink scission. The initial soluble fraction of the studied vulcanized NR was evaluated by the swelling test and was found to be equal to 1.17 %. When carrying out the analysis, it is considered that the experimental points located near the solid curve correspond mostly to the scission of sulfur bonds, and those positioned near the dashed curve correspond to the rupture of the carbon-carbon chains i.e. the polymer degradation. If there are points located between the two curves, they are considered to undergo regeneration i.e. a combination between devulcanization and degradation of the rubber.

2.3.3. Thermogravimetry

Thermogravimetric analyses (TGA) were performed in a Perkin Elmer Diamond TG/DTA. Samples of about 10 mg were heated from room temperature up to 800 °C, at a heating rate of 10 °C/min and under nitrogen atmosphere, after soxhlet extraction and swelling in toluene (see section 2.3.1). The mass variation of the samples was gradually measured during the thermal degradation of the material.

2.3.4. Scanning electron microscopy

Morphological analysis of the virgin and devulcanized NR was achieved by scanning electron microscopy (SEM) performed in a TESCAN MIRA3 FEG-SEM. The working accelerating voltage was 20 kV. Sample surfaces were prepared by cryofracture.

3. Results and discussion

The evaluation of the variation in the soluble fraction, before and after devulcanization treatment, is not enough to conclude about the devulcanization quality, because it cannot distinguish between crosslinks breaking (devulcanization) and rubber main chains breaking (degradation) (Li et al., 2005; Yazdani et al., 2011). In this work, the evaluation of the devulcanization quality was performed using the Horikx diagram (section 2.3.2.). Swelling tests results and the Horikx theoretical curves corresponding to the studied material are reported in Fig. 6. This figure shows that all the experimental points are near the lower curve corresponding to sulfur bond scission. Thus, the devulcanization was effective, with a selective bonds breaking and little or no polymer degradation. Fig. 6 also highlights that, as the temperature increases, the experimental points are moved to the right side of the chart i.e. to higher devulcanization levels. The devulcanization degree reaches a maximum value of ~90 %. To the extent of our knowledge, there are no similar results in the literature dealing with NR static devulcanization in scCO₂.

Mangili et al. (Mangili et al., 2014a) has proposed a devulcanization mechanism for sulfur-cured rubber, in the presence of DD. It is explained that the devulcanization process starts with the DD decomposition, which leads to the creation of two active radicals that are capable to open the sulfur bonds. The allylic hydrogen from the rubber is subtracted by the benzene-sulfide radical creating intermediate products (Rajan, 2005). Different combinations can take place during the reaction, afterwards new active crosslinking sites

are created making the devulcanized rubber ready to be revulcanized (Vega et al., 2008; Anu Mary et al., 2016; Asaro et al., 2018).

According to the preceding explanation, it is not possible to obtain a high devulcanization degree if the temperature is not enough to dissociate the DD, which was the case of the test carried out at 120 °C (Fig. 6). It can be concluded that the reactivity of DD increases at high temperature, and that 160 °C was the optimum one in this study. In addition, all the above results prove that the developed device works well and that it can be used to carry out rubber devulcanization under different experimental conditions. As stated in the literature, generally, the initial mechanical properties of the rubbers are not entirely recovered after the devulcanization and revulcanization processes, whatever the used devulcanization technique (Li et al., 2005; Mangili et al., 2014a; Isayev et al., 2014). As shown in Fig. 6, the experimental points obtained in this study are located on the solid curve of the Horikx chart, which means that the material has underwent mostly sulfur bonds scission. This suggests that the proposed devulcanization process would not have a significant influence on the mechanical properties of the revulcanized NR.

Although a satisfactory devulcanization was confirmed by the Horikx analysis, TGA and SEM tests were made to strengthen this result.

As explained by Kleps et al. (Kleps et al., 2000), the thermogravimetric analysis is useful to control and evaluate the devulcanization process. Therefore, thermal degradation of the virgin NR and the most devulcanized NR were studied by thermogravimetry, and the results are presented in Fig. 7. It is noticeable that the devulcanized material starts to degrade slightly before the virgin one. This could be related to the breaking of the tridimensional network of the treated rubber, during the devulcanization process, which is in accordance with the swelling test results presented in the Horikx diagram (Fig. 6).

Similar results were found by Valetini et al. (Valentini et al., 2020) when studying the thermal degradation of devulcanized tire rubber. Moreover, the thermal degradation rate is higher for the virgin material, result that could be associated to a higher sulfur content, which plays the role of thermal degradation accelerator. It is worth underlining that the average degradation temperature (375 °C) is the same for both materials, suggesting that the devulcanization process mainly breaks the sulfur bonds without breaking the main chains of the rubber. Finally, the residual mass corresponding to the virgin rubber amounts to about 20 % while that of the devulcanized rubber is about 34 %. The difference between the obtained values could be explained on the basis of carbon black content (Table 2). In fact, both materials had the same carbon black content, however, during devulcanization and washing processes, the treated material was separated into the gel and sol fractions, and only the gel part was used in the thermogravimetric test, the sol fraction was removed during the swelling test and washing (see section 2.3.1). Consequently, the relative carbon black content of the devulcanized rubber is higher, and is observed as a higher residual mass.

The microstructures of the rubber before and after devulcanization were analyzed by SEM. The obtained images are presented in Fig. 8. The microstructure of the virgin NR is continuous and smooth (Fig. 8a), which is a signature of a homogeneous and cohesive material. The material devulcanized at 120 °C (Fig. 8b) presents slight changes compared to the virgin NR, suggesting that the rubber underwent little modifications during the devulcanization process. The microstructure of the NR devulcanized at 140 °C (Fig. 8c) presents two phases, a homogeneous cohesive phase and a discontinuous non-cohesive phase, corresponding respectively to the devulcanized and non-devulcanized zones of the

rubber. This result means that the rubber was only partially devulcanized. Fig. 8d reveals that the microstructure of the material devulcanized at 160 °C is homogeneous, continuous and smooth, confirming that the rubber was highly and effectively devulcanized, as concluded from the Horikx analysis (Fig. 6).

4. Conclusion

A novel device allowing to perform rubber devulcanization was developed. It consists of a triaxial compression reactor integrated into a dynamic hydraulic universal testing machine with a heating chamber. NR industry waste was devulcanized in the mentioned device at different temperatures, in scCO₂ and using DD as devulcanizing reagent. A very high devulcanization degree (~90 %) was reached in the specimen tested at 160 °C. From the Horikx analysis, it was found that the devulcanization was successful, with almost no polymer degradation. It is worth noting that the devulcanization test conditions needs to be optimized to obtain similar results with another type of rubber. Thermogravimetric tests revealed that the material treated at 160 °C begun to degrade before the virgin one, which was an indication of crosslinks breaking in the rubber, confirming its successful devulcanization. In addition, SEM images showed that its morphology is homogeneous, continuous and smooth, which is another proof of its successful devulcanization, in accordance with the results obtained by Horikx and thermogravimetric analyses. The results from this work suggested that the NR industry waste could be highly devulcanized by the proposed process. This means that the developed reactor is appropriate to perform rubber recycling, which contributes to the progress in the waste management and thus in the environmental protection.

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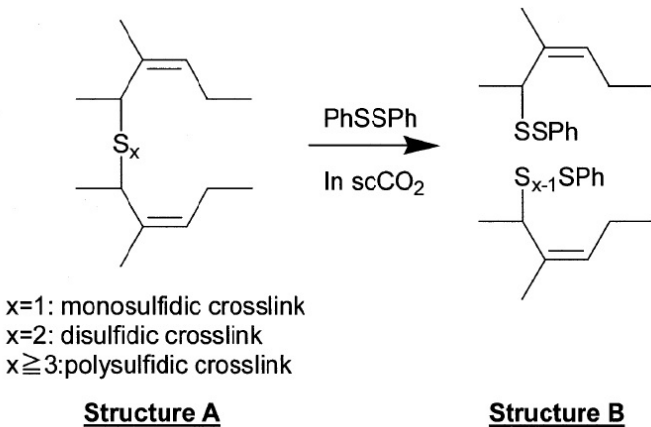


Fig. 1. Crosslink cleavage reaction of NR in the presence of DD and scCO₂ proposed by Kojima et al. (Kojima et al., 2004).

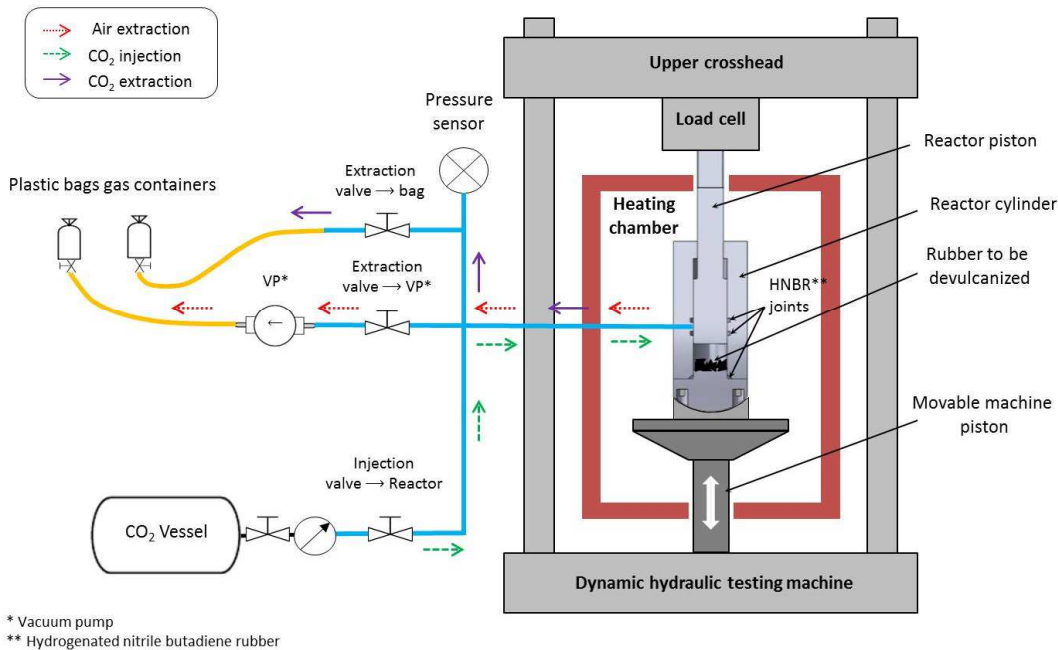


Fig. 2. Devulcanization device scheme.

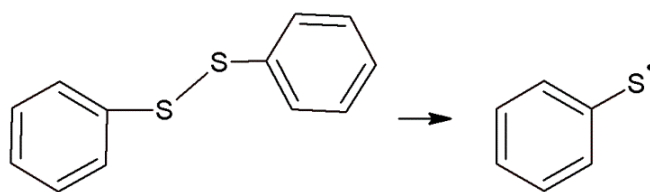


Fig. 3. Diphenyl disulfide chemical structure.

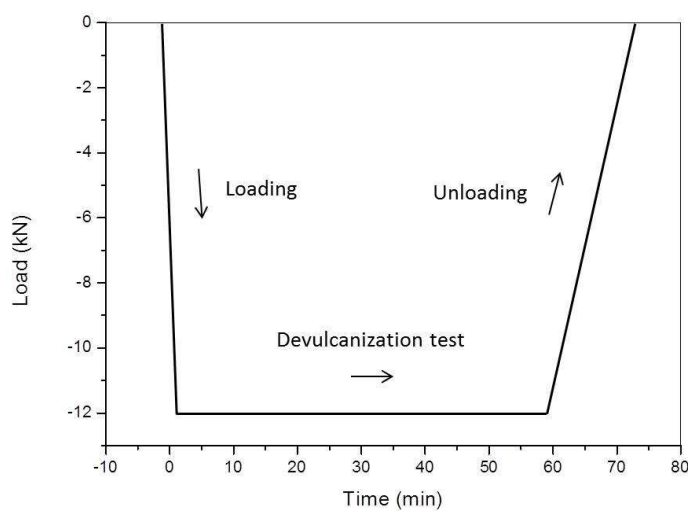


Fig. 4. Loading-unloading cycle carried out during the devulcanization test.

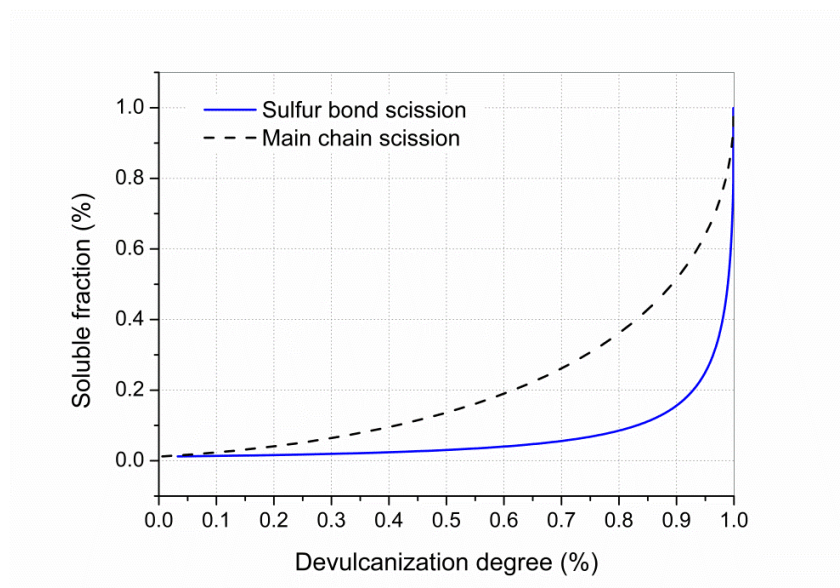


Fig. 5. Theoretical Horikx diagram of the studied NR.

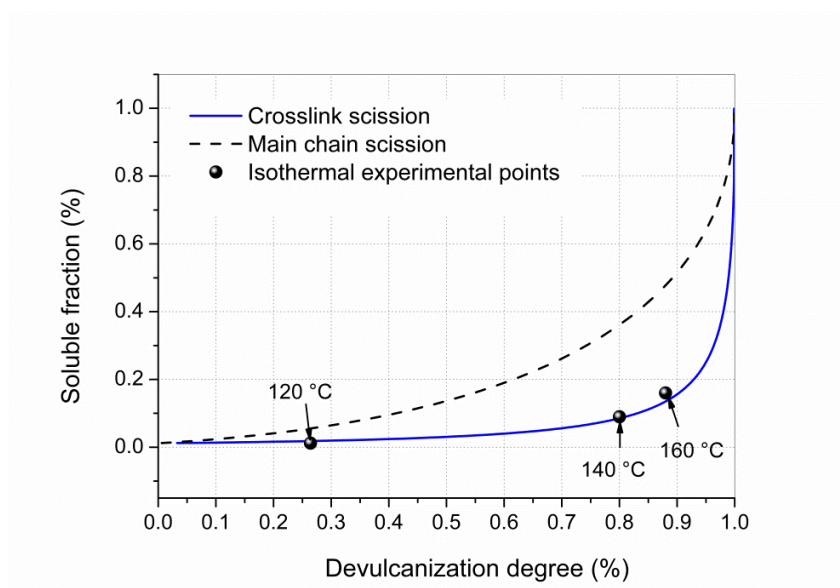


Fig. 6. Theoretical and experimental soluble fractions as a function of the devulcanization degree for the studied NR.

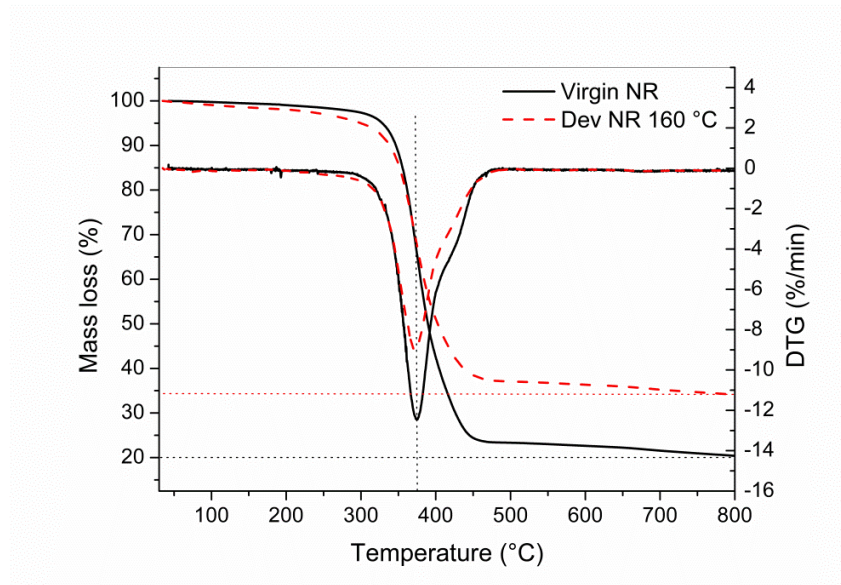
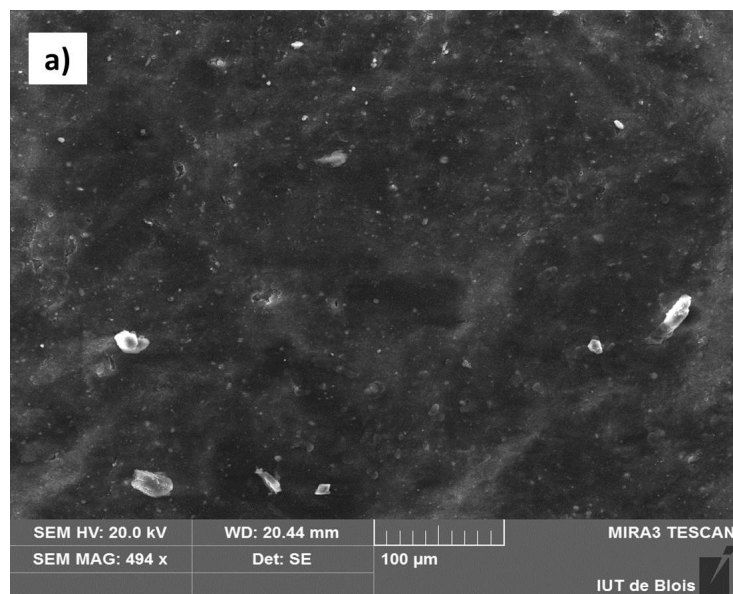
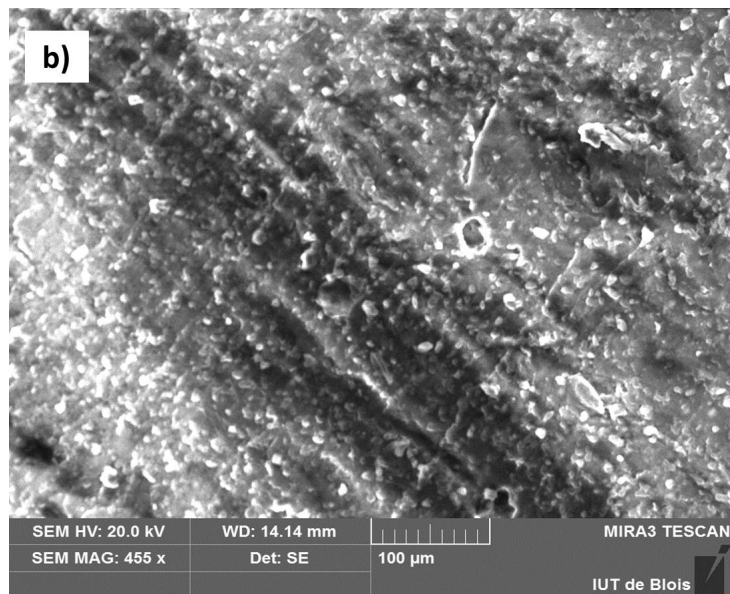


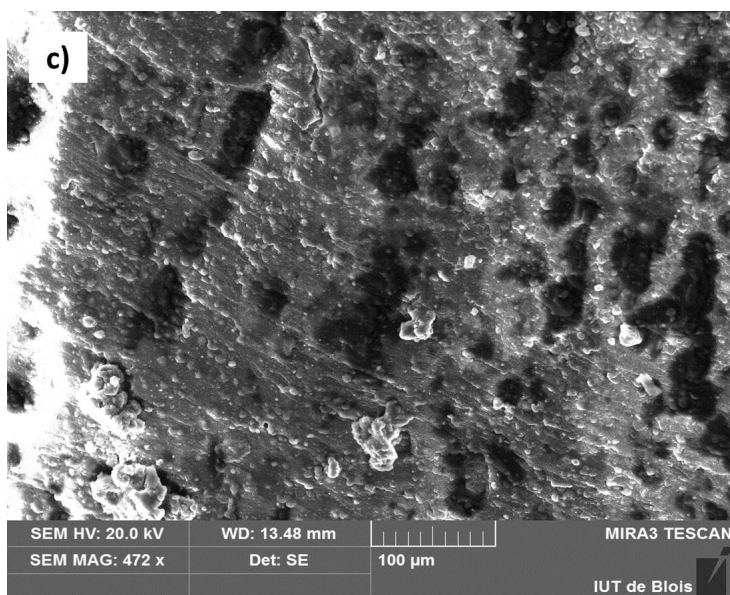
Fig. 7. Mass loss and its derivative (DTG) as a function of temperature for virgin NR and NR devulcanized at 160 °C.



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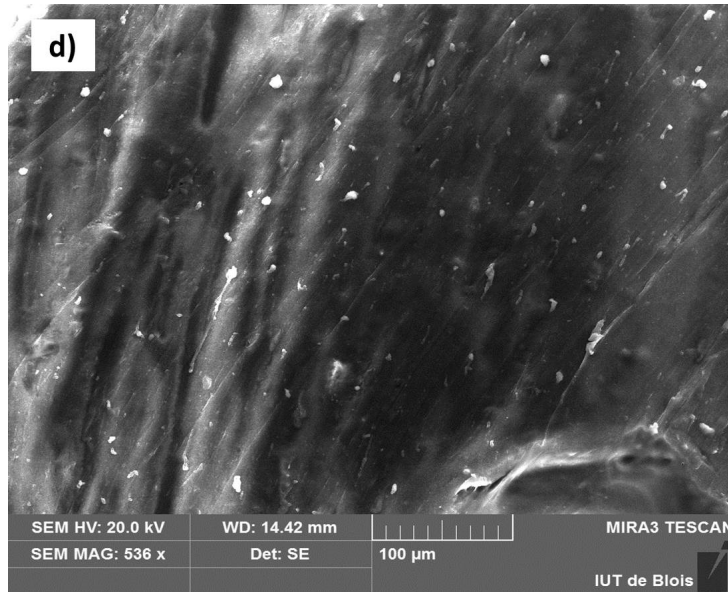


Fig. 8. SEM images of a) virgin NR, b) NR treated at 120 °C, c) NR treated at 140 °C, and d) NR treated at 160 °C.

Table 1 Table of symbols and notations.

Symbols and notations	Explanation
C-C	Carbon-carbon bond
CO ₂	Carbon dioxide
C-S	Carbon-sulfur bond
DD	Diphenyl disulfide
DTG	Mass loss derivative
GTR	Ground tire rubber
NR	Natural rubber
scCO ₂	Supercritical carbon dioxide
SEM	Scanning electron microscopy
S-S	Sulfur-sulfur bond
TGA	Thermogravimetry
m_i	Initial mass

m_d	Mass of the dried rubber
m_t	Mass of the sample impregnated with toluene
%S	Soluble fraction
ν_e	Crosslink density
V_r	Rubber volume fraction in the swollen sample
χ	Rubber-solvent interaction parameter
V_l	Molar volume of toluene
m_s	Mass of the solvent absorbed by the sample
ρ_s	Density of the dried rubber
ρ_d	Density of toluene at room temperature
%Devulc	Devulcanization degree
ν_i	Crosslink density of the rubber before devulcanization
ν_f	Crosslink density of the rubber after devulcanization

559

560 **Table 2** NR formulation.

Component	Sulfur-cured NR (phr)
NR	100
Carbon black	35
Processing oil	3
Stearic acid	1
CBS*	1
Sulfur	1.5

561 *CBS: N-Cyclohexyl-2-benzothiazole sulfonamide

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