

# Waste Recovery and Sustainable Development. A Case Study of Material Development from Used Tires in Africa

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## Abstract

This paper outlines a mechanical transformation process for rubber recycling, demonstrating the development of a new material from used tires. With the crumbs obtained using a crusher-compactor, a novel material for the manufacture of O-rings has been developed, with properties close to those found on the market. The process includes an experimental methodology of a sulfur vulcanization system choice and the quantification of ingredients, as well as the experimental determination of cure parameters. Mechanical tests on the samples completed the work by providing the mechanical characteristics of both unaged and aged (thermo-oxidative ageing) novel material. This process has a high potential for sustainable development and industrialization, making it a valuable contribution to the recycling of rubber in African developing countries.

## Keywords

Sustainable Development, Circular Economy, Used Tires, Elastomers Recycling, Vulcanization Parameters

## 1. Introduction

### 1.1. Motivation

Raw material scarcity and waste pollution require sustainable use of existing resources, especially in sub-Saharan African countries. Sustainable development

involves balancing economic growth, social responsibility, and environmental conservation. However, modern amenities such as cars and computers rely on non-renewable resources that cannot reproduce themselves once used [1] [2] [3] [4]. Automobile tires, made from non-renewable resources and designed for single use, do not fit well into the “reduce, reuse, recycle” waste treatment strategy. As the number of cars increases globally, so does the number of used tires. These tires can be transformed or disposed of, but the process is complicated due to the challenges of working with vulcanized rubber, which loses its ability to flow when heated, unlike plastics [5]-[10].

Recycling tires has benefits despite the technical challenges, including elimination of non-biodegradable rubber products, finding interesting civil engineering applications for whole tires [10] [11], and creating job opportunities. In addition, recycling requires less energy than producing new rubber [12]. End-of-life tires can also be used as fuel in the cement industry because of their high calorific value, which is equivalent to that of coal. Indeed, some studies [13] [14] claim that three tons of tires are equivalent to two tons of fuels. However, this method of energy recovery is generally inefficient and limited. Many tires end up in landfills where they can contaminate the environment, highlighting the need for effective recycling techniques.

Due to their rubbery characteristics, used tires are difficult to recycle. Current recycling methods for automobile tires include shredding, devulcanization using chemicals to break down tires, and treating rubber with liquid nitrogen to make a fine powder [5] [14] [15] [16]. The rubber industry faces challenges in recycling production waste and end-of-life products. Developed countries have increased regulations against landfilling or incineration of waste, while developing countries like Africa can benefit from developing a used-tire recycling industry to create jobs and improve waste management strategies. The aim of the present work is to contribute to the improvement of industrial recycling of rubber, similar to materials such as metals, plastics, glass, and paper.

## 1.2. Local Context

In Africa, material recycling industrial development is limited, and used tires are often only used for specific purposes like wells, ports, and roundabouts [17] [18]. Rubber recycling is mostly done by craftsmen who make various objects for local commercial value, and the only significant industrial activity is making wheelbarrow tires. It's crucial to develop and provide basic knowledge and feasible solutions to support the local economy and the environment.

### 1.2.1. Tire Retreading

Used tires are often retreaded or repaired, especially for heavy duty vehicles, which can lower the cost per kilometer of the tire. Developing countries practice this technique more often due to less regulations and standards. Retreading is a laborious process that is carried out manually, but it limits the consumption of raw materials and energy and reduces waste [12] [16]. Passenger vehicle tires are

generally only retreaded once, while truck tires can be retreaded and reused more than six times. In some developing countries, retreading is a well-established activity. For example, in Nairobi, Kenya, more than 10,000 tires are received each week for retreading [17] [18]. In Cameroon, a Chinese company established in the two largest cities of Yaoundé and Douala carries out retreading activities and competes in the tire market with second-hand tires imported from overseas.

### 1.2.2. Artisanal Material Recovery

A common way of recycling in African countries is to cut out materials from used items to create new objects. Rubber from tires is easy to work with and can be treated like leather. Basic tools like knives, shears, pliers, and hammers are used to make new products from used tires, as well as improvised tools for specific purposes. (See **Figure 1**)

Products from this rubber recovery method are of medium to low quality and sold to low-income populations. These include sandals, shoe soles, doormats, water containers, pots, dustbins, and even automobile components like silent-blocks, motor mounts, seals, and washers (**Figure 2**). Some parts are customized by mechanics, and workshops are usually located near autoshops. Consumers generally appreciate the long-lasting effectiveness of these products, and due to the good price/quality ratio, there is a strong demand for these automotive spare parts. This consumer demand proves the great commercial outlet of the industrial production and the interest of having a good mastery of the mechanical properties of the parts thus produced.

This paper will focus on creating and characterizing a material made from used tires for producing engine sealing components, specifically O-rings (**Figure 3**).

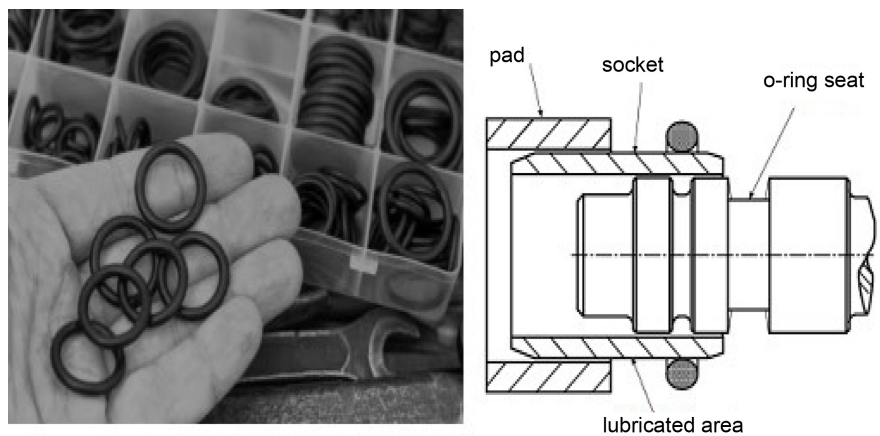
An O-ring is a popular elastomer-based molded part. It is made by filling an



**Figure 1.** Typical tools used by rubber artisans (photo credit from authors).



**Figure 2.** Auto parts made from used tires include silent-blocks ((a), (b), (d)), an exhaust support clamp (c), a wedge for reinforcing the tie rod (e), and the brake pedal and footrest of a motorcycle (f). (Photo credit: authors)



**Figure 3.** Rubber O-rings with a schematic description of their use in pneumatics/hydraulics (Image courtesy of Google).

elastomer compound into a mold of the required shape of the part to be made [19] [20] [21]. There are two main molding processes: compression molding and injection molding. The choice of process depends on the component dimensions and the quantity to be produced.

Commercial tires are mainly composed of elastomeric rubber, carbon black, metal, textile fibers, and other additives. The composition varies depending on the type and model of the tire and its road application. Natural rubber, styrene-butadiene rubber, and butadiene rubber are commonly used elastomers, while butyl elastomer is used for inner tubes due to its impermeability to gases [12] [22] [23] [24] [25]. To make new material from used tires, the first step is to crush them using mechanical equipment to create rubber granulates or crumbs. Crumbs are particles smaller than 1 mm, while granulated aggregates range from 1 to 10 mm in size. (See **Figure 4**)



**Figure 4.** Semi-finished products obtained from used tires [10].

Industries are interested in using semi-finished products made from used tires to create molded parts. Indeed, crumbs can be re-agglomerated under heat and pressure to make items such as wheels for wheelbarrows or lawnmowers, as well as more technical spare parts like O-rings, which we will discuss in this study. Vulcanization is the process that allows the properties of the elastomer article to be suitably adjusted for proper functionality.

Vulcanization is a process that creates a three-dimensional network in raw rubber by reacting it with sulphur. This chemical reaction is slow at room temperature, so it is typically done at around 150°C to 180°C [23]-[30]. The most used vulcanization system is based on sulphur and applies to dienic elastomers such as NR, SBR, BR, IR, NBR, EPDM, and IIR (see Appendix for the nomenclature of elastomers). Other vulcanization systems are sometimes called crosslinking or ratification [7] [8] [9] [23] [25]. The process involves activators like zinc oxide and stearic acid, which facilitate the combination of sulphur and rubber, and accelerators that determine the kinetics of the reaction [23]. The length of the sulphur bridges created by the reaction affects the behavior of the resulting vulcanizates and can be adjusted by changing the doses of sulphur and accelerators [26]-[30].

The choice of vulcanization system depends on the type of base elastomer for this chemical reaction, which can be either natural rubber derived from the latex sap of the Pará rubber tree (*hevea brasiliensis*) or synthetic rubbers derived from hydrocarbons.

Vulcanization is an irreversible chemical reaction that must be done on virgin elastomer, so used tire crumbs must be reinforced with virgin elastomer to allow them to vulcanize again. For this study, natural rubber (NR) associated with inner tube crumbs (IIR) is selected as the base elastomer, because natural rubber is abundantly produced in Africa and has a positive carbon balance as a renewable biopolymer [23]. Tire treads are not used in this study because they are made of



SBR or BR elastomers, which have undesired damping characteristics for sealing [19]-[24]. However, tire treads could be a good choice as part of base elastomer for other applications that require strong damping, such as engine mounts or silent-blocks, and the methodology developed in this study for O-rings could then be used for any other technical rubbery components.

The rest of the paper is organized as follows. The framework of the investigation is depicted in Section 2. The third section describes the compounding method of masterbatches. Section 4 contains experimental tests and the results. Section 5 summarizes the findings of this paper.

## 2. Research Methodology

Developing a rubber material involves choosing a base elastomer, then consequently a vulcanization system and appropriate additives to tailor the mechanical properties required for good performance of the finished product. It is important to ensure that the selected mixture can maintain its properties over time despite mechanical stress and environmental damage, which can be tested using thermo-oxidative ageing technique. For this study, the material will be developed for O-rings from used tires, and the properties will be modeled on commercial O-rings. A preliminary investigation is carried out on commercial O-rings, including practicality, mechanical properties, and performance in different environments [19] [20] [21]. The mechanical properties of the material will be based on the properties of commercial O-rings as provided by manufacturers' catalogs (see summary in Table 1).

The approach in this study, depicted by Figure 5 below, can be applied to the development of any elastomer material from used tires.

It consists initially in identifying the required characteristics for the material that one wants to develop, and then the approach comprises 3 stages:

- The first stage is the preparation of masterbatches: here it is a question of defining 1) the base elastomer, *i.e.*, the proportions of used tires' crumbs and a virgin elastomer for the vulcanization reaction; 2) the most suitable vulcanization system to obtain the desired characteristics; and 3) the right additives in the right quantity to better modulate the characteristics of the finished product.
- The second stage consists of vulcanization aptitude tests, which will make it

**Table 1.** Mechanical properties of O-rings material as provided by manufacturers.

	Minimum value	Maximum value
Hardness (Shore A)	50	90
Elongation at break (%)	300	1000
Stress at break (MPa)	3	20
Modulus 100 <sup>a</sup> (MPa)	1	4

a. The modulus at 100% elongation.

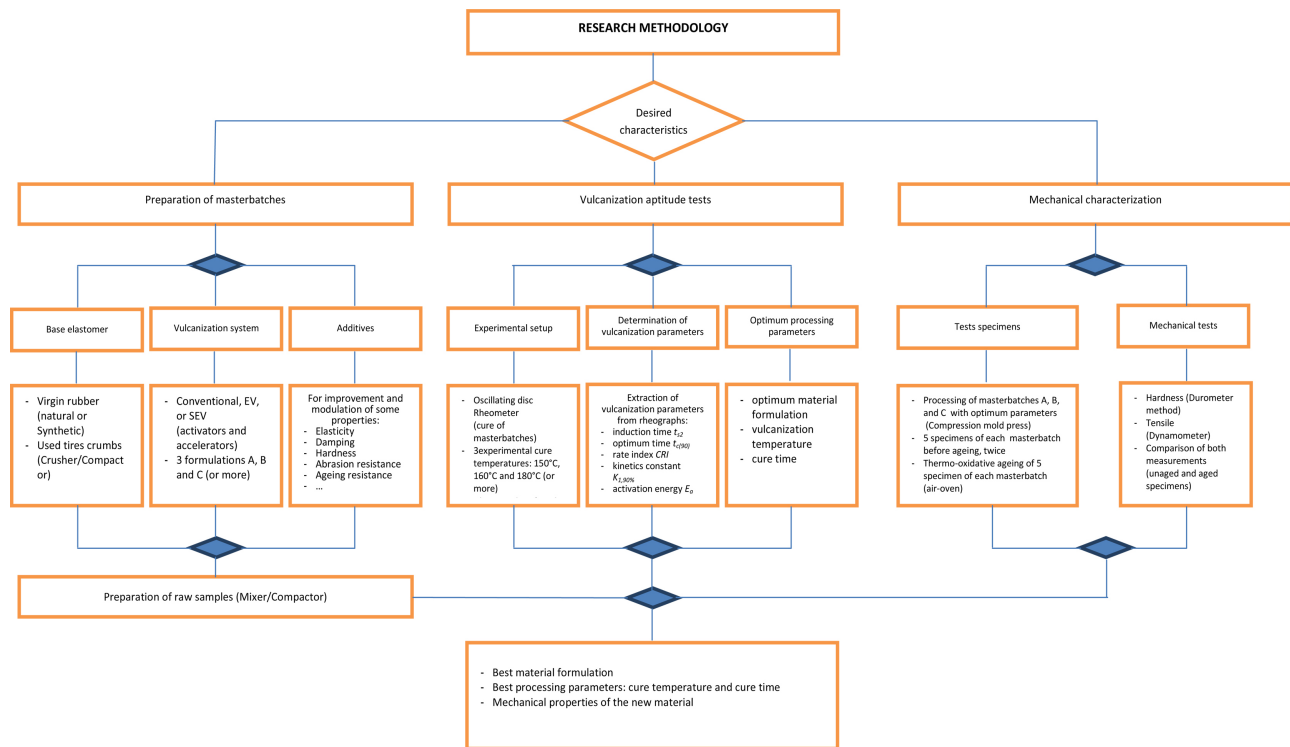


Figure 5. Research methodology framework.

possible to 1) experimentally extract the optimal vulcanization parameters, which allows to 2) determine the best formulation of raw masterbatches that leads to the optimal processing of the finished product.

- The last stage is the mechanical tests, comparing resulted samples from the previous stages to determine the best formulation with the best vulcanization parameters which will make it possible to obtain the new material having the characteristics closest to the desired finished product, as well than to estimate its relative resistance to deterioration due to ageing over time. Mechanical tests will include hardness and tensile before and after thermo-oxidative ageing.

Three formulations (A, B, and C) will be used for the vulcanization aptitude tests, and five specimens will be tested for each formulation before and after ageing.

### 3. Preparation of Masterbatches

#### 3.1. The Base Elastomer

For this study, natural rubber associated with inner tube crumbs is selected as the base elastomer, in a specific ratio of 1/4 natural rubber, NR, and 3/4 inner tube crumbs, IIR. Consequently, the vulcanization system and other additives must be carefully selected to fine-tune the properties needed for the best performance of O-rings. Finally, the mixture must be able to maintain its performance despite wear and tear from mechanical and environmental factors.

### 3.2. The Vulcanization System

Vulcanization is the essential process that transforms raw elastomer into a finished product, maintaining its elasticity and shape. Three vulcanization systems are possible by adjusting the amounts of sulfur and accelerators, [26]-[30] (see **Table 2**): classical or conventional, efficient (EV), and semi-efficient (SEV). The conventional system creates polysulfide bridges with good mechanical properties and resistance to fatigue but poor resistance to aging. The efficient system creates monosulfide bridges with improved aging resistance but decreased mechanical properties and fatigue resistance. The semi-efficient system creates disulfide bridges with intermediate characteristics, providing a compromise between mechanical properties, fatigue resistance, and aging resistance.

The choice of vulcanization system depends on the desired specifications. For the base elastomer used in this work (consisting of 1/4 NR and 3/4 IIR), the conventional system is chosen because it provides good mechanical properties and fatigue resistance.

### 3.3. Additives

Besides activators and accelerators, other ingredients can improve or modulate properties of the elastomeric material, such as hardness, compression set, shear and tensile strength, abrasion resistance, or ageing resistance. To increase the hardness of the material for high service pressures (50 to 90 degrees shore), carbon blacks, specifically N330 of SRF type are added. This ingredient type is more suitable for O-rings compared to HAF type, which provides good damping but is not ideal for seals' material [26]-[31]. Finally, an antioxidant, Vulkacit H/30 (hexamethylenetetramine), is added to the vulcanization system to protect against ageing [26]-[31].

### 3.4. Raw Samples Compounding

To conduct experiments, it is needed to mix together different ingredients in varying quantities to create different formulations adapted for the chosen vulcanization system. Three different formulations are created as summarized in **Table 3** one with the minimum amount of ingredients required (formulation A), one with the maximum amount (formulation C), and one with the average amount (formulation B). These ingredients will be mixed using an open two-roll

**Table 2.** Sulphur based vulcanization systems.

Ingredients (pphr*)	Conventional	EV	SEV
Zinc oxide	3 - 5	3 - 5	3 - 5
Stearic acid	0.5 - 2	0.5 - 2	0.5 - 2
Sulphur	2 - 3	0 - 0.25	0.4 - 1.5
Accelerator	0.5 - 1	3 - 5	1.2 - 2

\*pphr is parts per hundred of rubber, *i.e.* relative to 100 parts of natural rubber.



**Table 3.** Compounding of masterbatches.

Formulation	Ingredient, *pphr							
	NR	Inner tube crumb	Zinc oxide	Stearic acid	Sulphur	TMTD	N330	Vulkacit H/30
A	100	300	12	2	8	2	200	12
B	100	300	16	5	10	3	200	12
C	100	300	20	8	12	4	200	12

\*pphr is parts per hundred of rubber, *i.e.* relative to 100 parts of natural rubber.

mill of 1/1.35 friction ratio mixer/compactor (**Figure 6**) to create raw samples for testing.

First, natural rubber (NR) is softened and made more pliable through a process called plasticizing. Then, used tire crumbs (IIR) and other ingredients needed for vulcanization are gradually added to the rubber until the mixture is well blended. The plasticizing process involves heating and shearing the rubber with mixer cylinders, which also prevents premature vulcanization by cooling the cylinders with circulating water. The mixing process takes a certain amount of time to properly blend the ingredients without causing vulcanization (**Table 4**). Once the raw mixtures are finished, they are stored in plastic paper for testing.

## 4. Experimental Tests and Analysis

### 4.1. Vulcanization Aptitude of Raw Materials

Finally, in order to obtain the best properties of the product, the proportions of the mixture of materials, the temperature and the vulcanization time must be determined and optimized. For that, tests will be performed to determine these optimal parameters.

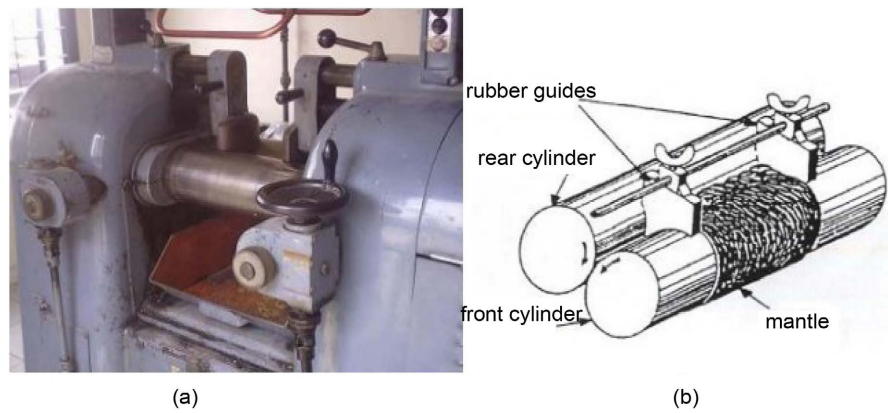
#### 4.1.1. Experimental Setup and Parameters Determination

The Monsanto Model R 100S Oscillating Disc Rheometer (**Figure 7(a)**) was used to conduct experiments, in accordance with international standards for determining the vulcanization characteristics of rubber compounds [26]-[30]. The experiments were carried out at isothermal temperatures of 150°C, 160°C, and 180°C, with rotor oscillating amplitude of 3° and a frequency of 1.67 Hz. Raw samples of masterbatches were compressed in a sealed enclosure and maintained at the chosen vulcanization temperature. A disc embedded in the sample was used to apply a shearing force to the masterbatch, and the required torque was automatically recorded as a function of time (**Figures 7(b)-(d)**). The obtained rheographs allow the determination of various vulcanization parameters of the masterbatches. These include:

- 1) The minimum torque  $M_L$  (reflecting processability), the maximum torque  $M_H$  (corresponding to the ultimate degree of vulcanization), and the difference between the two (which provided the maximum crosslinking density). The safety

**Table 4.** Raw samples of masterbatches processing sequence.

Step	Operation	Duration (min)
1	Heating the cylinders surface to 70°C	1
2	Plasticization of the natural rubber	1
3	Incorporation of the crumb (after spacing the cylinders)	2
4	Incorporation of the activators: ZnO + Stearic acid	1
5	Incorporation of the accelerators: TMTD	1
6	Incorporation of the Carbon blacks: N330	1
7	Incorporation of the antioxydant: Vulkacit H/30	1
8	Incorporation of the sulfur (after reducing the cylinder gap)	2
9	Drawing of the raw sample in slabs	2

**Figure 6.** (a) Open Mixer/Compactor; (b) Descriptive scheme.

limit for optimum vulcanization retained is equal to 90% of the difference between  $M_H$  and  $M_L$ .

2) The induction time  $t_{s2}$  marks the start of crosslinking and serves as an implementation security measure for the vulcanization process.

3) The vulcanization optimum time  $t_{c(90)}$  is the time required to obtain 90% of the maximum torque.

4) The vulcanization rate index  $CRI$  (Cure Rate Index) is determined by calculating the average slope of the ascending part of the curve and used to assess the kinetics of vulcanization. The approximate expression for  $CRI$  is thus given by the formula:

$$CRI = \frac{100}{t_{c(90)} - t_{s2}} \quad (1)$$

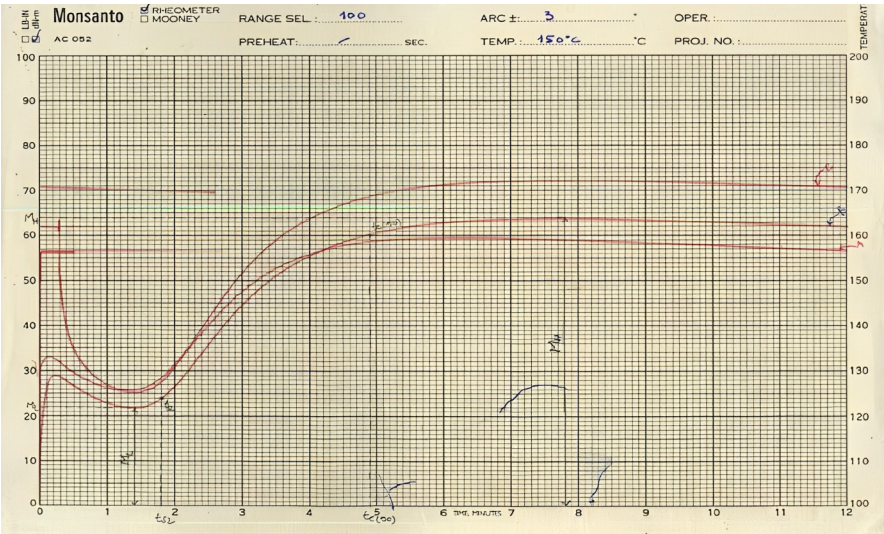
5) It is also noted that the constant of the chemical reaction kinetics is given by the empirical Arrhenius relationship:

$$K = Ae^{-E/RT} \quad (2)$$

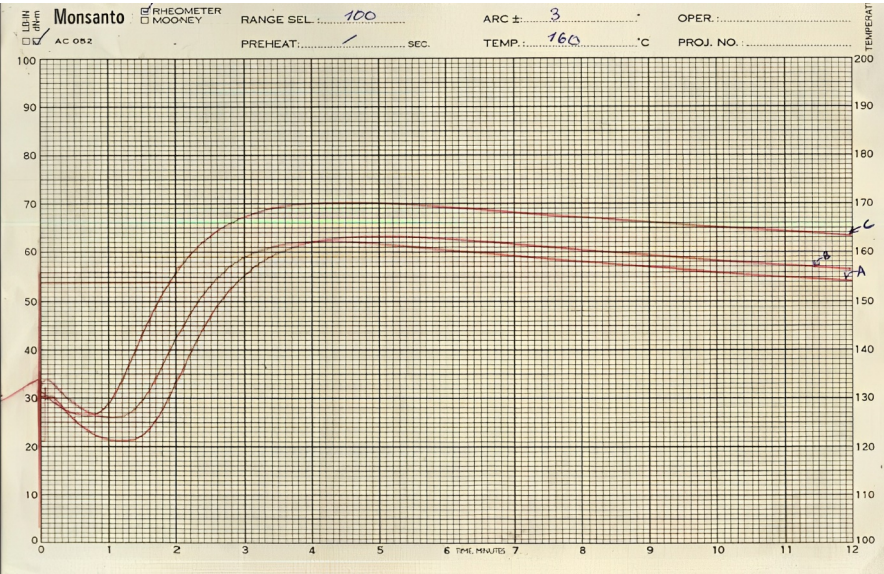
where  $A$  is the action constant;  $R$  is the ideal gas constant; and  $E$  is the activation



(a)

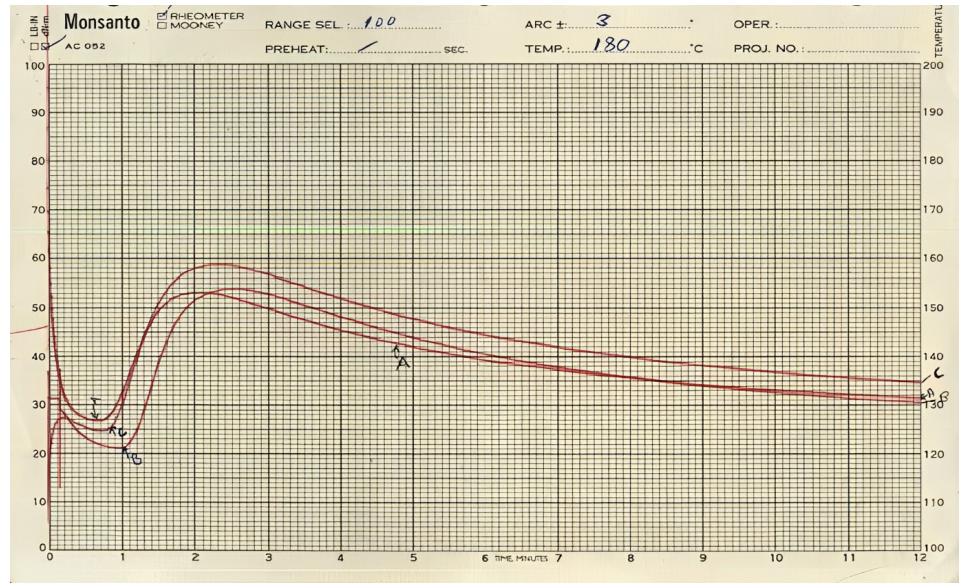


(b)



(c)





(d)

**Figure 7.** (a) Lab oscillating disc rheometer; (b), (c) and (d) rheographs for vulcanization parameters determination, respectively at 150°C, 160°C and 180°C.

energy of the reaction. For rubber technologies, the constant  $K$  corresponding to 90% sulfur consumption is considered to be of first order. It is given by the formula:

$$K_{1.90\%} = \frac{Ln10}{t_{c(90)}} \tag{3}$$

6) Finally, the activation energy of the reaction  $E_a$  can be calculated using the Arrhenius relationship:

$$E_a = R \frac{T_1 - T_2}{T_1 T_2} Ln \frac{K_1}{K_2} \tag{4}$$

where  $K_1$  et  $K_2$  are the reaction constants corresponding to  $T_1$  et  $T_2$  respectively.

#### 4.1.2. Results and Analysis

A model for the analysis of rheographs from **Figure 7** is developed to understand how vulcanization temperature and formulation affect the rubber. **Table 5** shows the values found for the vulcanization parameters. These results are important in the process as they help to compare the processability, the cross-linking density, and the vulcanization kinetics of different formulations (A, B, C) at different temperatures (150°C, 160°C, 180°C). Thus, the analysis can be based on selection criteria such as  $t_{s2}$ ,  $t_{c(90)}$ ,  $CRI$ , and  $K_1$ , 90%.

- To prevent burning of the rubber, the induction time  $t_{s2}$  must be long enough before vulcanization begins. The maximum value for  $t_{s2}$  is 1.8 minutes for all formulations, which is obtained at 150°C (**Table 5**).
- The vulcanization optimum time,  $t_{c(90)}$ , must also be long enough to prevent surface crosslinking. The maximum value for  $t_{c(90)}$  is different for each for-

mulation at each temperature, with the highest value of 4.9 minutes for formulation B at 150°C (Table 5).

- To avoid high vulcanization kinetics parameters  $CRI$  and  $K_{1.90\%}$ , which can lead to problems with thick parts due to poor heat conduction, the minimum values are different for each formulation. Formulation B at 150°C has the smallest values for  $CRI$  (32.3) and  $K_{1.90\%}$  (0.47) (Table 5).

Based on these selection criteria, we conclude that 150°C is the best vulcanization temperature and that formulation B is most suitable for the masterbatch vulcanization. The activation energy for formulation B is also higher than the other formulations, at 15.85  $\text{KJ}\cdot\text{mol}^{-1}$ .

## 4.2. Mechanical Tests

### 4.2.1. Masterbatches and Specimens

To make test specimens, a laboratory mold press (shown in Figure 8) is used to shape samples from pre-made materials A, B, and C. The press plates are heated to 150°C and the raw material samples are placed into the mold. The process is automated with preset temperature and time parameters.

Once the samples have undergone optimal vulcanization, which takes different times for samples A, B, and C (4.1, 4.9, and 4.6 minutes respectively), they are removed from the press and quickly extracted from the molds. The samples are left at room temperature for 24 hours before being cut into five tensile bars each using a punch, following ISO 1826 standards (shown in Figure 9). Hardness and tensile tests are subsequently performed on each bar.

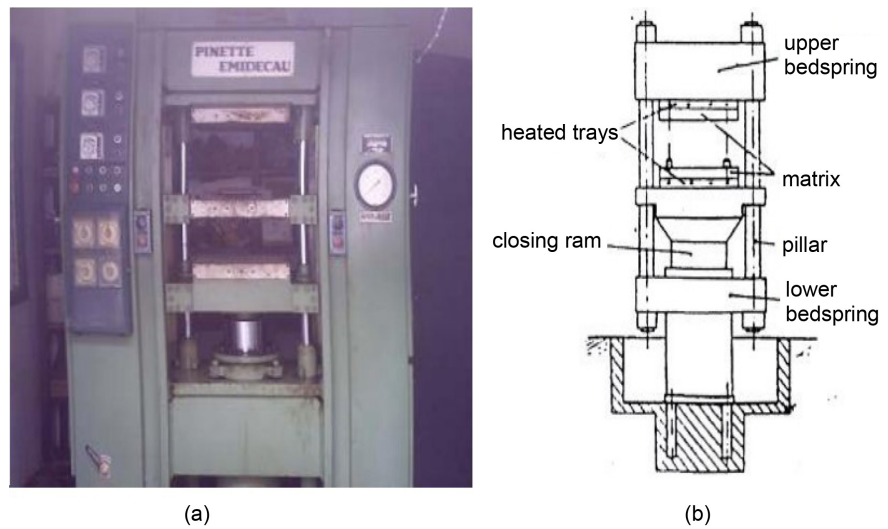
### 4.2.2. Hardness Tests

To test the hardness of the samples, the durometer method (Shore hardness) was used following the ISO 7619-1:2010 standard. There are two measurement

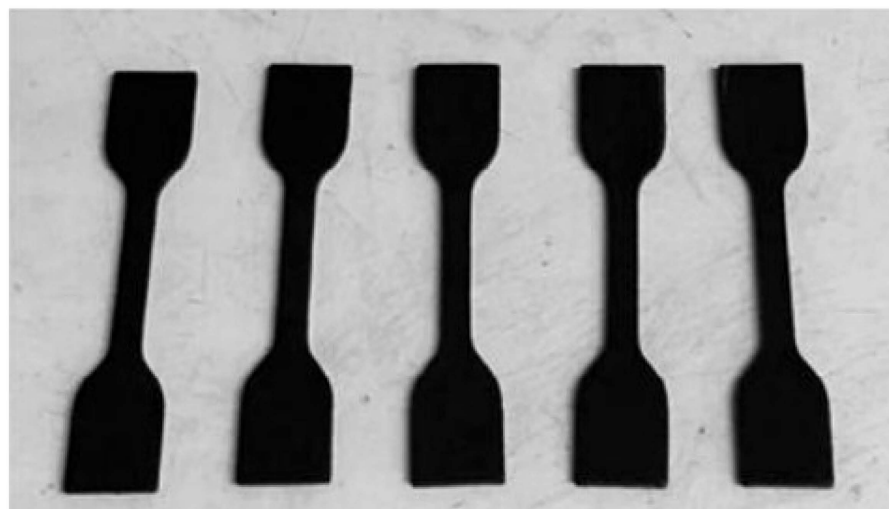
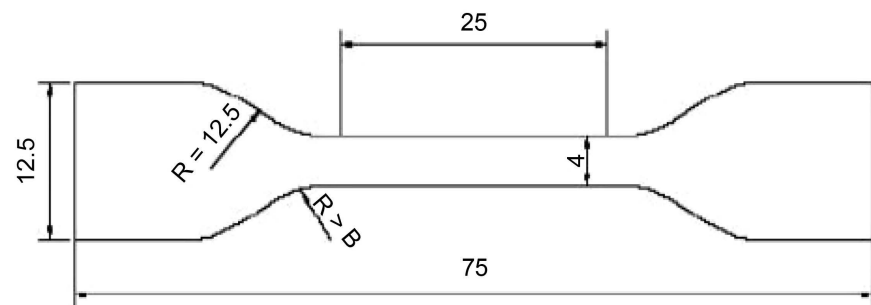
**Table 5.** Vulcanization parameters of the masterbatches.

Vulcanization temperature (°C)	Masterbatch	90% ( $M_H - M_L$ ) (torque units) <sup>a</sup>	$t_{52}$ (min)	$t_{\alpha(90)}$ (min)	$CRI$	$K_{1.90\%}$	$E_a$ (150 - 180) ( $\text{KJ}\cdot\text{mol}^{-1}$ )
150	A	31.1	1.8	4.1	43.5	0.56	6.94
	B	37.8	1.8	4.9	32.3	0.47	15.85
	C	42.3	1.8	4.6	35.7	0.5	13.29
160	A	32.4	1.4	3	62.5	0.77	-
	B	37.8	1.5	3.4	52.6	0.68	-
	C	39.2	1.0	2.8	55.6	0.82	-
180	A	23.4	0.9	1.6	142.9	1.44	-
	B	29.7	1.2	1.9	133.3	1.21	-
	C	31.1	0.9	1.8	111.1	1.28	-

a. 1 torque unit = 0.05 N m.



**Figure 8.** (a) Lab compression mold press; (b) Descriptive scheme.



**Figure 9.** Tensile bars for tests. Here represented the 5 specimens of a series.

scales, Shore A hardness for normal to hard rubbers and Shore D hardness for high-hardness rubbers. Each of the A, B, and C samples were tested at five evenly spaced points on the specimen. **Table 6** displays the results, including statistical quantities such as means, standard deviations, and coefficients of variation, which were used for analysis.



**Table 6.** Shore hardness measurements.

Hardness	Masterbatch	Mean value	STD <sup>a</sup>	CoV <sup>b</sup>
Shore A	A	58.4	1.5	2.6
	B	60.4	0.5	0.9
	C	61.4	1.1	1.9
Shore D	A	14.6	1.5	10.4
	B	16.4	0.9	5.5
	C	15.8	1.1	6.9

a. Standard deviation. b. Coefficient of variation.

The coefficient of variation measures the repeatability of measurements, and a lower coefficient of variation indicates better homogeneity. Formulation B has the lowest coefficient of variation at 0.9%, while A and C have coefficients of variation of 2.6% and 1.9%, respectively. Measurements for Shore A hardness are more repeatable than those for Shore D, indicating that the masterbatches are of normal-hardness range rubber material. The difference in hardness values between the three samples in each Shore scale is negligible, as the proportion of the strengthening fillers N330 is the same for all three formulations. Specifically, formulation A has  $58 \pm 1.5$ , formulation B has  $60.4 \pm 0.5$ , and formulation C has  $61.4 \pm 1.1$  as shore A hardness.

#### 4.2.3. Tensile Tests

Tensile tests are performed on masterbatches A, B, and C using the ISO 37:2017 standard. Five specimens of each masterbatch are tested by applying continuous loading to a tensile bar mounted on a dynamometer (Figure 10) at a speed of 0.25 mm/min until the material broke. This speed is chosen because elastomers are viscoelastic materials and in practice stresses occur relatively slowly. During the test, a digital display indicated the force and elongation, and the tensile curves are recorded for each specimen (Figure 11 shows curves of specimens B). The strength at 100% elongation, tensile strength at break, and ultimate length are recorded. The elongation at break, stress at break, modulus at 100% elongation, and modulus at break are then calculated and summarized in Table 7.

The initial useful length of the specimens is  $L_0 = 25$  mm, and the rectangular section  $A_0$  of the specimens is

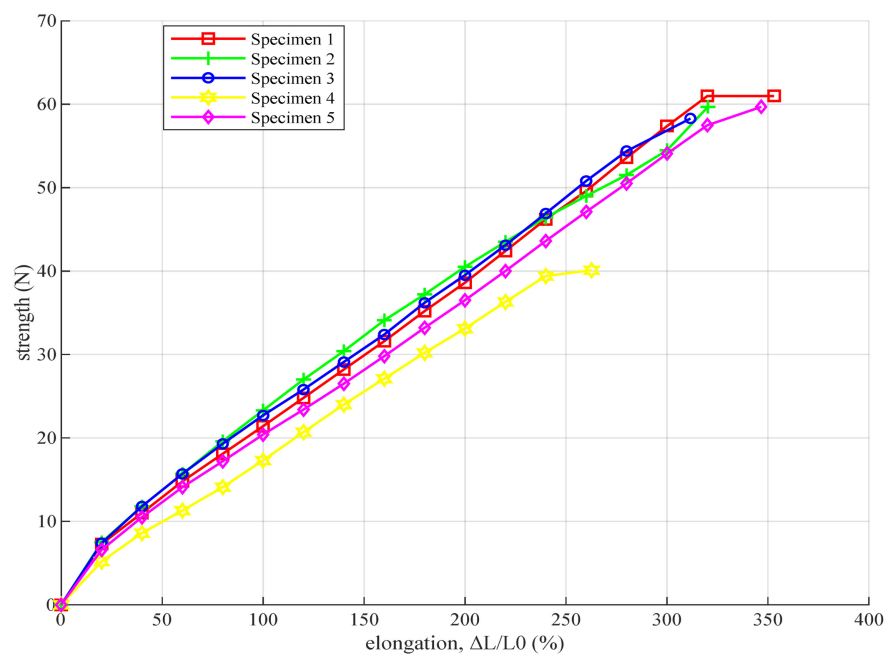
$$A_0 = 4 \text{ mm} \times 3.7 \text{ mm} = 14.8 \text{ mm}^2 \quad (5)$$

All specimens break at elongation levels beyond 300% and exhibit almost identical tensile behavior, except for specimen 4, which breaks slightly earlier. This demonstrates the masterbatch's quasi-homogeneity.

Tensile tests show that formulation B is of higher quality than the other two. Masterbatch B can withstand a greater stress before breaking compared to masterbatch A and C, as shown in Table 7, with a value of  $(3.82 \pm 0.64)$  MPa. Similarly, it can elongate more than the other two before breaking, with an elongation



**Figure 10.** Lab dynamometer for tensile testing of rubbers.



**Figure 11.** Tensile curves of masterbatch B.

at break of  $(319\% \pm 36\%)$ . In comparison, masterbatch A has an elongation at break of  $(245\% \pm 37\%)$ , and masterbatch C has an elongation at break of  $(262\% \pm 27\%)$ .

#### 4.2.4. Masterbatch Ageing Assessment

The thermo-oxidative ageing technique is used to assess the ageing of the newly developed material through accelerated ageing tests. This method, which follows the ISO 188:2011 standard, evaluates the relative resistance of vulcanizates to deterioration over time. In this technique, vulcanized specimens are placed in a cabinet oven with forced air circulation (Figure 12) for 24 hours at a temperature of  $100^\circ\text{C}$ . The ventilation rate is ranged from 3 to 10 changes per hour. After

**Table 7.** Tensile tests results.

Measure	Masterbatch	Strength 100 <sup>a</sup> (N)	Ultimate length (mm)	Elongation at break (%)	Strength at break (N)	Stress at break (MPa)	Modulus at break (MPa)	Modulus 100 <sup>b</sup> (MPa)
Mean value	A	17.6	61.2	245	36.1	2.44	0.98	1.19
	B	21.0	79.7	319	56.5	3.82	1.19	1.42
	C	22.5	65.4	262	44.6	3.02	1.16	1.52
STD	A	3.5	9.2	37	10.4	0.70	0.17	0.23
	B	2.4	9.0	36	9.5	0.64	0.10	0.16
	C	3.6	6.7	27	4.8	0.33	0.19	0.24
CoV (%)	A	19.9	15.0	15.1	28.8	28.7	17.3	19.3
	B	11.4	11.3	11.3	16.8	16.8	8.4	11.3
	C	16	10.2	10.3	10.8	10.9	16.4	15.8

a. Strength at 100% elongation of specimens. b. The modulus at 100% elongation of the specimens.

**Figure 12.** Lab air-oven for thermo-oxidative ageing of rubbers.

the ageing process, the specimens undergo hardness and tensile tests, similar to the unaged ones and the resulting measurements (shown in **Table 8** and **Table 9**) are compared with the corresponding measurements obtained from the unaged vulcanizates.

In a final phase of performance evaluation, the test results are compared to the previous measurements obtained from the unaged specimens. The change in properties after ageing is determined by the ratio of the value obtained after ageing to the value obtained before ageing, as shown in **Table 10** and **Table 11**.

After ageing, masterbatch B maintains the main mechanical characteristic for O-ring's materials that is its hardness ( $99\% \pm 3$  in **Table 10**), but its elasticity (modulus, 85%) and resistance (stress at break, 61%) decrease (**Table 11**). This

**Table 8.** Hardness tests results after ageing.

Hardness	Masterbatch	Mean value	STD <sup>a</sup>	CoV <sup>b</sup>
Shore A	A	59.4	1.3	2.2
	B	59.6	1.5	2.5
	C	64.0	2.2	3.4
Shore D	A	14.8	0.8	5.4
	B	14.8	0.8	5.4
	C	16.6	0.5	3.0

a. Standard deviation. b. Coefficient of variation.

**Table 9.** Tensile tests results after ageing.

Measure	Masterbatch	Strength 100 <sup>a</sup> (N)	Ultimate length (mm)	Elongation at break (%)	Strength at break (N)	Stress at break (MPa)	Modulus at break (MPa)	Modulus 100 <sup>b</sup> (MPa)
Mean value	A	19.2	74.9	299	51.6	3.49	1.16	1.30
	B	17.5	56.8	227	33.3	2.25	1.00	1.18
	C	25.2	61.2	245	48.3	3.27	1.36	1.70
STD <sup>c</sup>	A	1.0	10.8	43	9.6	0.65	0.09	0.07
	B	2.2	9.1	36	3.8	0.26	0.08	0.15
	C	2.9	13.0	52	7.9	0.54	0.16	0.20
CoV <sup>d</sup> (%)	A	5.2	14.4	14.4	18.6	18.6	5.63	5.38
	B	12.6	16.0	15.9	11.4	11.6	8.00	12.7
	C	11.5	21.2	21.2	16.4	16.5	11.76	11.76

a. Strength at 100% elongation of specimens. b. The modulus at 100% elongation of the specimens. c. Standard deviation. d. Coefficient of variation.

**Table 10.** Evolution of hardness after ageing.

Hardness	Masterbatch	Mean value (%)	STD <sup>a</sup>
Shore A	A	102	4
	B	99	3
	C	104	4
Shore D	A	102	9
	B	91	8
	C	105	8

a. Standard deviation.

poor retention of mechanical properties is also observed for the other masterbatches, although the trend is upward for them (mean values above 100% in **Table 10** and **Table 11**). This result was previously expected as the chosen vulcanization system is known to sacrifice ageing resistance for good mechanical

properties and fatigue resistance. To improve ageing resistance, a semi-efficient vulcanization system (SEV) that offers a good compromise between mechanical properties, resistance to fatigue and ageing should be recommended.

## 5. Summary of the Optimum Obtained Experimental Results

**Table 12** summarizes the main results of the methodology developed in this work to make a new material from used tires. As above presented, the process includes formulation, processing, and mechanical characterization. The obtained

**Table 11.** Evolution of tensile characteristics after aging.

Measure	Masterbatch	Strength 100 <sup>a</sup>	Ultimate length	Elongation at break	Strength at break	Stress at break	Modulus at break	Modulus 100 <sup>b</sup>
Mean value (%)	A	114	125	125	156	156	122	114
	B	85	72	72	61	61	85	85
	C	115	95	95	110	110	119	115
STD	A	32	28	28	64	64	30	32
	B	21	14	14	16	16	13	21
	C	28	26	26	27	27	24	28

a. Strength at 100% elongation of specimens. b. The modulus at 100% elongation of the specimens.

**Table 12.** Results summary.

Ingredient	Masterbatch formulation	
	Concentration (pphr)	Quantity (g)
NR, 50 <sup>a</sup>	1/4	100
Inner tube crumb	3/4	300
Zinc oxide	4.0	16
Stearic acid	1.25	5
Sulphur	2.5	10
TMTD	0.75	3
N330	50	200
Vulkacit H/30	3	12
Vulcanization temperature	Processing parameters	150°C
Optimum cure time	Mechanical properties	4.9 minutes
	Developed material	Manufactured products
Hardness (Shore A)	60.4 ± 0.5	50 to 90
Elongation at break (%)	319 ± 36	300 to 1000
Stress at break (MPa)	3.82 ± 0.64	3 to 20
Modulus 100 <sup>b</sup> (MPa)	1.42 ± 0.16	1 to 4
Modulus at break (MPa)	1.19 ± 0.10	-

a. Technically specified NR of grade 50. b. The modulus at 100% elongation.

results point out that it is possible to manufacture functional O-rings from rubber waste.

## 6. Conclusions

Used tires can be recycled through mechanical transformation into semi-finished products, such as crumbs, which can be used to make molded parts. This is a great opportunity to provide raw materials for industries all over the world. The successful production of wheelbarrow tires by a Cameroonian company is a good testimony. Thus, due to the simple implemented processes, this product can be also developed in all non-industrialized countries as those located in sub-Saharan countries.

The process developed in this study integrates the phases of preparation of masterbatches from used tire crumbs, the vulcanization aptitude and thermo-oxidative ageing tests, as well as the mechanical characterization of the new material thus developed. The main results obtained show that the final product has interesting characteristics in terms of elasticity, hardness, and ageing. In particular, it was obtained with masterbatch B, for an optimal vulcanization temperature of 150°C, a modulus of elasticity of  $(1.42 \pm 0.16)$  MPa, an elongation at break of  $(319\% \pm 36\%)$ , and shore hardness of  $(60.4 \pm 0.5)$ , some values largely included in the range of manufactured products on the market, and keep well over time, with a retention rate of around 100% almost after thermo-oxidative ageing.

This study shows a successful recovery of waste from used tires with the aim of circular economy and sustainable development. The development at state-of-the-art of O-rings material indicates the feasibility of mass-producing products made from recycled tires in an industrial or artisanal way in all countries around the world and therefore, in developing countries such as many sub-Saharan countries in Africa. Moreover, the production cost of an O-ring made from used tires is 3 orders of magnitude lower than the cost of commercially available O-rings. This makes the use of recycled tire materials a promising and cost-effective alternative for the manufacturing of O-rings and other similar products.

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## Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.



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## Appendix: Nomenclature of Elastomers

The following nomenclature gathers the big families of elastomers.

(<http://www.scti.fr/114-elastomer-properties>)

Symbol	Common name	Formula
NR	Natural rubber	Natural polyisoprene
IR	Isoprene	Synthetic polyisoprene
BR	Butadiene	Polybutadiene
SBR	Butadiene-styrene	Styrene and butadiene copolymer
NBR	Nitriles	Acrylic nitrile and butadiene copolymers
IIR	Butyl	Isoprene and isobutylene copolymers
EPDM	-	Terpolymère of ethylene, propylene and a diene
CR	Chloroprene	Polychloroprene
CSM	-	Chlorosulfoné polyethylene
FPM	Fluorocarbonaceous	Rubber with alkylfluorés or alkoxyfluorés side groups
PTR	Polysulphide	Polysulphide
PU	Polyurethane	Polyesteruréthane or polyetheruréthane
VMQ	Silicone	Polysiloxane with side groups methyl and vinyl
FVMQ	Fluorinated silicone	-
ACM	Polyacrylic	Ethyl acrylate copolymer
CO	Epichlorhydrine	Polychlorométhoxyrane
ECO	Epichlorhydrine copolymer	Oxide copolymer of ethylene or oxyrane and chlorométhoxyrane