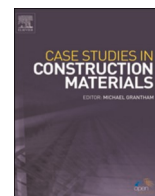


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Case Studies in Construction Materials

journal homepage: www.elsevier.com/locate/cscm

Case study

Composite materials made of waste tires and polyurethane resin: A case study of flexible tiles successfully applied in industry

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ARTICLE INFO

Keywords:

Waste tires
Rubber tiles
Polyurethane resin
Materials characterization
Leaching
Toxicity

ABSTRACT

This research presents a case study of producing flexible tiles from rubber powders obtained from automobile tire waste using a polyurethane resin as a binder matrix. The process was made in collaboration with a company located in Colombia, where the manufacturing of these materials has been optimized. The material is a green solution to an increasing worldwide problem, rubber car tires mostly put in landfills or burned to extract their reinforced steel wires instead of properly recycled. Several rubber contents and particle size distributions were investigated and tested. Tension, density, scanning electron microscopy, and thermo-gravimetric analysis characterization were used to evaluate the composites. Leaching analysis of contaminant are also included. Results shows that the amount of rubber used is quite large in comparison with the binder, maximizing the rubber in the formulations, and thus using more waste. The flexible characteristics of both resin and rubber enable these composites to be used in multiple applications. The tensile tests showed the composite can work very well for structural applications of low solicitations, such as wall covers, soft floors and barriers. The project is a successful example of a small-medium enterprise company that contributes to the circular economy of these highly pollutant materials.

1. Introduction

The type of economic model and its consequent increase in the demand of new cars has led in an incredible and uncontrollable amount of waste tires everywhere [1]. Over 1.4 million of new tires are generated every year [2], and there are more than 4 billion tires [3] in landfills worldwide. Besides the multiple solutions, which includes waste to energy [4], asphalts [5], concretes [6], and composites [7], among others, there is a need of more solutions that increase the circularity of materials and maximize its useful time. In addition, tire waste is not only a common source of mosquitos from stagnant water, which is a source of dangerous diseases [8,9]; but also is a common illegal practice, particularly in developing countries, the tire burning for the steel extraction that produces very hazardous pollution [10]. Therefore, the handling of this problem is still far from a total solution, and thus, very diverse and innovative solutions are required.

Although most of the tires and tire waste are produced in China (with about 60 % of the total world production [11]), USA and Europe, in developing countries, new problems appear, such as a poor waste management and old technology, weak environmental

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<https://doi.org/10.1016/j.cscm.2021.e00681>

Received 14 May 2021; Received in revised form 23 August 2021; Accepted 31 August 2021

Available online 1 September 2021

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policies, and social issues (such as illegal tire burning) [12,13]. In the case of Colombia, the average annual consumption of tires is between 4.5 and 5.5 million, from which their waste, 72 % are treated by incineration, 17 % is hoarded and 6% have a craft disposition.

Tire waste has been successfully mixed with other polymeric materials, such as polypropylene [14], and polyurethane foams [15]. Several investigations related to the study of rubber sheets, their properties, and applications were found. Ahmed, Nizami, and Riza 2014, developed natural rubber composites reinforced with marble sludge by compression molding [16]. Lertloypanyachai and Thongsang have investigated rock powders acting as filler in natural rubber aiming higher mechanical properties in rubber-based floor tiles [17]. Zaera et al. developed a polyurethane and rubber-modified epoxy resin as a potential adhesive layer to bind ceramic tiles with a metallic plate [18].

On the other hand, the application of these tire residues in the construction industry has been studied by Hejna, Aleksander, et al. They investigated the interfacial interaction between the rubber residues and the polymeric matrix to improve the performance properties of the polymer/rubber composites, proposing an in-situ reaction between the matrix and the filler during the formulation of construction and engineering materials [19]. They also studied thermoplastic elastomers in recovered rubber evaluating the role of peroxide initiators in the compatibility efficiency of mixtures, significantly improving the traction, temperature and morphology of the base materials [20]. LI, Yue, et al. They studied ground tire rubber thermo-mechanically devulcanized in the presence of waste engine oil, improving the storage stability and processability of rubber modified asphalt [21]. Sienkiewicz, Maciej, et al. evaluated the specific surface area and the morphology of the ground tire rubber recycled grains in the properties of the rubber-asphalt binder demonstrating that the addition of rubber to the asphalt binder changes the percentage of asphalt fractions and affects the molecular weight distribution of the rubberized asphalt binder [22].

From an exhaustive analysis of the potential applications of the processing of this type of waste, a small company committed to the environment arises focused on the use and transformation of recycled materials "EcoReproducts" has been created as result of this research. The company develops flexible floors from granulated rubber from tires and industrial surpluses. Designing efficient, innovative and ecological impact solutions for all types of environments: gyms, playgrounds, kindergartens, sports plaques, decorative spaces (indoor and outdoor), stables for horses, livestock, among others.

In this research, unlike the aforementioned, tiles from four different granulometries of rubber waste powders were made in combination with polyurethane resin, using a hot-press machine. The preparation of composite materials and the tests carried out are detailed, which include voltage, density, scanning electron microscopy, and thermo-gravimetric analysis. These materials contribute to the reduction of pollution generated by rubber residues, but also create new business opportunities for local people.



Fig. 1. Rubber grinding process. a) Car tire waste, b) grinding, c) 30 mesh ground rubber, and d) industrial linear vibrating sieve shaker.

2. Experimental methods

Prismacaucho S.A.S from Colombia supplied the waste tire rubber in four different granulometries, passing 4 mm (mesh #5), 2 mm (mesh #9), 0.841 mm (mesh #20) and 0.595 mm (mesh #30). The rubber was ground in a large-scale blade milling, since only steel and metallic free tires were used in the process. This milling square rubber blocks of approximately 20 cm. Then, these blocks were introduced into the first grinding apparatus which delivers amorphous tire particles, which were later introduced to a second grinding process, which transformed the pieces into fine grains and particles. The primary and secondary grinding is made in roll crushers, which are used to optimize different granulometries. The products obtained from the secondary crushing are passed through an industrial linear vibrating sieve shaker to separate those particles whose size is already fine enough. This sieve separates the rubber particles into four different grain sizes 4 mm (mesh # 5), 2 mm (mesh # 9), 0.841 mm (mesh # 20) and 0.595 mm (mesh # 30). Fig. 1 shows part of the grinding process.

The polyurethane resin was obtained from Sumiglas S.A. from Colombia, which is a prepolymer that does not need a catalyst to accelerate the process of curing and bonding the rubber particles. This resin can be cured under ambient conditions, giving a density of 1.21 g/cm³. The manufacturer reports that the cured material is rigid, strong, with good dimensional stability, and high insulating properties against electricity and humidity.

The mixtures preparation was conducted at room temperature conditions, with resin mixtures kept in a low proportion concerning the composite costs due to the amount of polyurethane involved in the process. Eight mixture formulations (R1, R2, R3, R4, R5, R6, R7, R8) were developed as shown in Table 1. These rubber-resin mixtures were made in a mechanical mixer working at 60 rpm during 5 min, which guaranteed an appropriate resin-particle impregnation. Once mixtures were ready, each of the eight formulations was deposited on a heating plate, considering a uniform distribution of the whole mixture on the surface.

For mixtures of smaller granulometry such as 20 and 30 mesh, more time for heating and compaction was used in the process (approximately 40 s more), because the particles were very small and therefore the area to be wetted by resin is increased, as seen in Fig. 2a–c. The process optimization in the company found these results, which were implemented in this research. The rubber sheet manufacturing process was carried out under rigorous temperature control during pressing, the upper and lower plates of the press-maintained temperatures of 150 °C and 130 °C respectively, while the applied pressure was 1500 psi during 1 min. Since the lower plate has the mold on it, it has to be set at higher temperature to guarantee the same 130 °C in the top plate surface due to the heat loss by the mold thickness. Thus, the material at the end is between two surfaces at the same 130 °C, although the machine parameter control seems different.

After obtaining the rubber-resin tiles in the hot-press, these parts were cut with a die-cutter for tensile tests following the ASTM D638 standard. A total of 40 samples were die-cut distributed in five groups, consolidating 8 different formulations (see Fig. 2d).

From the flexible tiles designed, for density tests, samples were cut in squares of 3 cm side, following the ASTM D792 [23] standard. These tests were conducted based on the weight, pieces dimensions, and buoyancy (apparent density). A high precision Mettler Toledo balance that followed Archimedes' principle was used (true density) [24]. In these calculations, a set of 5 samples was tested for each formulation, taking into account that the density of isopropanol is 0.786 g/cm³. Both the true density and the apparent density were used because at the industrial level both methods are used frequently and although they give different results, they can be used as a density measurement contrast mechanism.

A Shimadzu AG250KN universal testing machine was utilized for tensile tests, running at a speed of 50 mm/min. During this procedure, 5 samples were analyzed for each formulation, taking at each step images alluding to their fracture surface, as shown in Fig. 7. The hardness tests were elaborated employing a shore scleroscope which measures hardness in terms of material elasticity. A diamond-tipped hammer (size of 40 grains; 2.59 g; 0.0914 oz) is used to strike the testing surface from a known height (10 in.; 254 mm), in which the rebound of the hammer was measured. The harder the material, the higher the rebound. A rule of mixtures was used to calculate the composite density, taking 1.10 and 1.21 g/cm³ densities for the raw rubber and polyurethane resins, respectively.

Table 1
Sample formulation fabricated using different waste contents and particle sizes.

Sample by particle size	R1	R2	R3	R4	R5	R6	R7	R8
Rubber (g)	1200	1200	1200	1200	1200	1200	1200	1200
Polyurethane Resin (g)	120	60	120	60	120	60	120	60
Particle size (mm)	4	4	2	2	0.841	0.841	0.595	0.595
Mesh	5	5	9	9	20	20	30	30
Total (g)	1320	1260	1320	1260	1320	1260	1320	1260
Polyurethane Resin (wt%)	9.1	4.8	9.1	4.8	9.1	4.8	9.1	4.8
Rubber (wt%)	90.9	95.2	90.9	95.2	90.9	95.2	90.9	95.2
Total (wt%)	100	100	100	100	100	100	100	100
C/R Ratio*	10	20	10	20	10	20	10	20
Rubber (cm ³)	1090.9	1090.9	1090.9	1090.9	1090.9	1090.9	1090.9	1090.9
Polyurethane Resin (cm ³)	99.2	49.6	99.2	49.6	99.2	49.6	99.2	49.6
Total (cm ³)	1190.08	1140.5	1190.08	1140.5	1190.08	1140.5	1190.08	1140.5
Rubber (V%)	91.7	95.7	91.7	95.7	91.7	95.7	91.7	95.7
Polyurethane Resin (V%)	8.3	4.3	8.3	4.3	8.3	4.3	8.3	4.3
Total (V%)	100	100	100	100	100	100	100	100

* C/R Ratio refers to the ratio between rubber/resin.

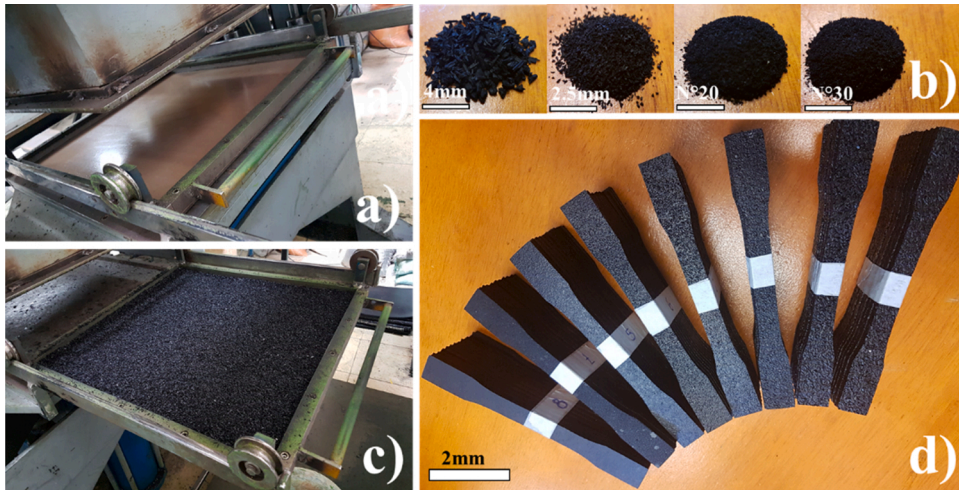


Fig. 2. Flexible tile fabrication, a) hot-press, b) rubber particles size, c) binder on the heating plate, and d) samples ready for the tensile tests.

Using scanning electron microscopy (SEM) and dispersed energy spectroscopy (EDS), raw materials and some rubber formulations were analyzed. All samples after the drying process were mounted on a carbon ribbon, and gold was sprayed with a Hummer 6.2 system, under 15 mA AC conditions for 30 s, process followed to form a thin film. The equipment used was a JEOL JSM 6700R in a high vacuum mode.

The particle size distribution of the images obtained by SEM was analyzed using the ImageJ software, for this purpose, 100 measurements of the particle diameters of the SEM images were taken for each mesh and, subsequently, the particle distribution was analyzed with the Statgraphics software using a statistical analysis of a variable under the normality model established by Shapiro Wilk.

Thermogravimetric analysis (TGA) was conducted using a PerkinElmer TGA. Approximately 10 mg of sample was heated at a rate of 20 °C/min from room temperature until 650 °C under Argon atmosphere.

For the Fourier Transform Infrared Spectroscopy (FTIR) tests, samples of rubber powder, polyurethane resin, and formulations R4 (9 mesh) and R6 (30 mesh) were analyzed with the aid of a Shimadzu IR Tracer-100 apparatus. For this test, spectra were studied between ranges of 400 and 4000 cm⁻¹.

An atomic absorption spectrophotometry test was conducted on 0.25 g of sample for the resin and rubber sample, which were homogenized and weighed. Then, samples were exposed to an acid digestion process with 65 % HNO₃, using an Ethos One Microwave analytical digester equipment. Later, samples were analyzed in the ICE- Atomic Absorption Spectrophotometer 3000, using an air-acetylene flame for the determination of Zn and graphite furnace for the determination of Pb, Cd, Cr, Ni. Tests were conducted at room temperature.

3. Results and discussions

Using multiple SEM images from the rubber powders and Image-J software, particle size distribution curves were built for each mesh, which appears summarized in Fig. 3. It represents a normal distribution as a function of the particle length. From the statistical analysis, it is observed that all the rubber powders have different kurtosis, due to the different heights of the distribution curves. Results for rubber powders like mesh 5 and 9 show curves almost symmetrical, while the mean value of 355.35 μm for rubber powder of mesh 20 shows their particles are dispersed and are not concentrated in a single point, which means their distribution is not homogeneous. This mesh size allows formulations will be less stable, since a smaller particle size requires more temperature to degrade

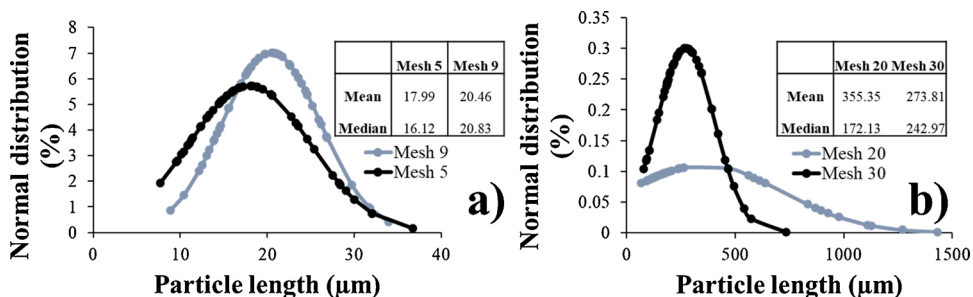


Fig. 3. Particle size distribution for the rubber powders. a) Mesh 5 and 9, and b) Mesh 20 and 30.

the material because there is more exposed area. This is evidenced in the TGA test. The distribution range of rubber powders of mesh 5, 9 and 30 do not deviate to a great extent from the mean and median.

Fig. 4a shows the summary of densities, with all values very close to 1 g/cm^3 (the mean values between 0.95 and 1.15 g/cm^3) but plotted from 0.6 g/cm^3 to zoom out the small differences. The rule of mixture was higher because it does not include the small voids visible in the SEM images. True and apparent densities showed high variation, particularly for formulations R6 and R8, see Table 1, which correspond to smaller particle size and lower resin content. The differences in the true and apparent densities of formulations R1 and R2 are mainly influenced by the particle size: when working with a greater granulometry it is more likely to find empty spaces and larger cracks between the particles, generating porosity in the material, and facilitating the incrustation of unwanted elements that can change the results of the density. The higher standard deviation can be associated to a higher voids and agglomeration defects, typically more associated to the smaller particle sizes. While Fig. 4b shows the values of the volumetric fractions of rubber and polyurethane resin in stacked columns, obtained from the mixing law for density. A higher percentage of rubber can be evidenced in all formulations exceeding the 90 vol%.

Fig. 5 shows the results of the tensile stress test, where the stress-strain curves can be observed. The composite with the highest stress (1.70 MPa) corresponds to the R2 formulation, followed by formulations R4 and R3 (1.49 MPa and 0.70 MPa respectively). The grain size in these formations is much greater, therefore promoting the development of a higher tensile strength. On the other hand, as the grain size decreases, the stress decreases, a situation is best observed for the formulations R1, R5, R6, R7, and R8. Additionally, it can be observed that all the formulations have an elastic behavior, and when they reach the yielding stress, the test samples break. According to Fig. 5, these formulations have a considerable elastic zone, although the materials have a low strength to rupture.

The maximum values for the tensile stress and the maximum elongation results were summarized in Fig. 6. The results show that the R5 formulation is of great importance because corresponds to the highest elongation range among the other formulations (161.8 mm). Its rubber composition is moderate and its small grain size (20 mesh), which may contribute to improving the bonding between the rubber and the polyurethane resin. On the other hand, the formulation R2 showed the highest value of tensile stress, a formulation that is characterized by having the least amount of resin and the largest particle size (4 mm). The high variation in standard deviation for the R7 formulation could be due to a complex combination of particle parameters and matrix, producing a combination with higher elongation properties. Further modeling research could give support to this explanation. R2, R3, and R4 formulations are in general stiffer than others because these have greater granulometries, and regardless of the resin content, the presence of larger tire rubber grains, a material stiffer than the polyurethane matrix, strengthen the composite.

Fig. 7 shows samples after the tensile tests. From these images is observed that formulations R7 and R8 look smoother than others in the surface finishing, being some of the more difficult to hold by the grips. Furthermore, these formulations have low resin content, and perhaps the rubber particles do not easily adhere to each other, being the reason why these samples were more fragile.

In general, SEM images shows the rubber powders after the grinding process with an amorphous shape, see Fig. 8. On the other hand, no macro-level defects were observed in general for all the composites fabricated (just some small pores and small impurities from the car tire additives in some of them), which validates the followed manufacturing process, see Fig. 9. The excellent adhesion between the polyurethane resin and the rubber is reflected in the homogeneity of the surface.

Table 2 shows SEM-EDS results with mostly carbon and silicon from the polymeric compounds in the rubber, and silicon and zinc from the additives some of them as oxides in the tire for mechanical and other property enhancement objectives.

Fig. 10 shows the results of differential scanning calorimetry (DSC) and thermogravimetric analysis (TGA), made by R7 and R8 formulations. R7 and R8 formulations were selected for these analyses because the aim was to understand how these results were affected by the variation of the resin content, which was made only at 120 g and 60 g . Results showed that both samples in Fig. 10a and b have the same trend.

DSC results are shown in Fig. 10a. In both curves, lines start in 4 W/g and $0 \text{ }^\circ\text{C}$ and finish at approximately -1 W/g and $700 \text{ }^\circ\text{C}$. As a large part of the curve is above zero on the axis "y", it is an exothermic process, where the sample that is tested releases heat, representing the beginning of the degradation of the material. The peaks that form the curve are of great extension indicating the presence of an amorphous polymer, rubber. Additionally, the concavity of the downward curve demonstrates a process of melting at about

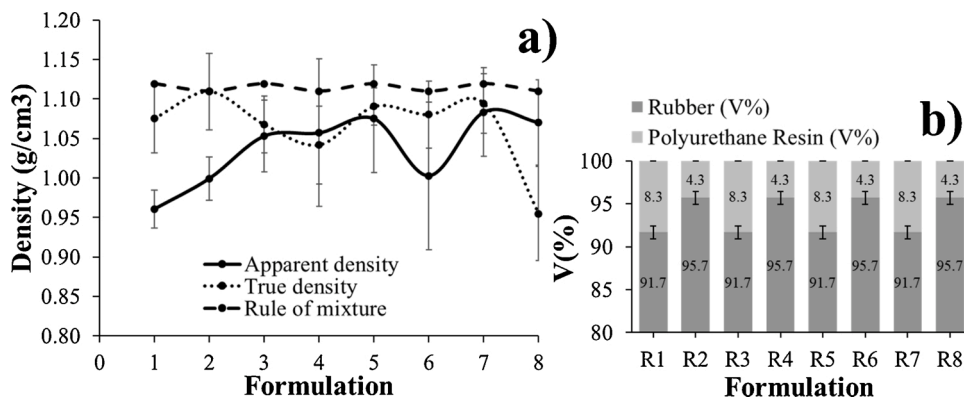


Fig. 4. Density results. a) For the different formulations and b) volumetric fractions from the law of mixtures.

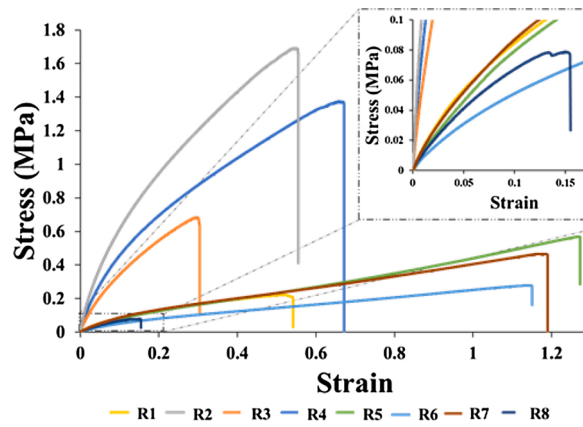


Fig. 5. Stress-strain curve.

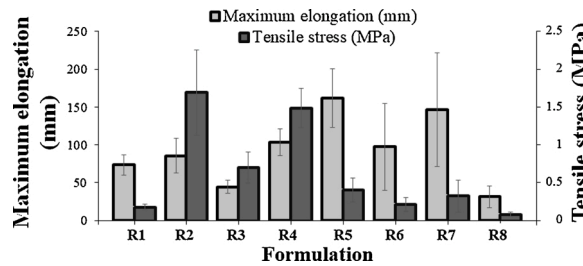


Fig. 6. Mechanical test results.

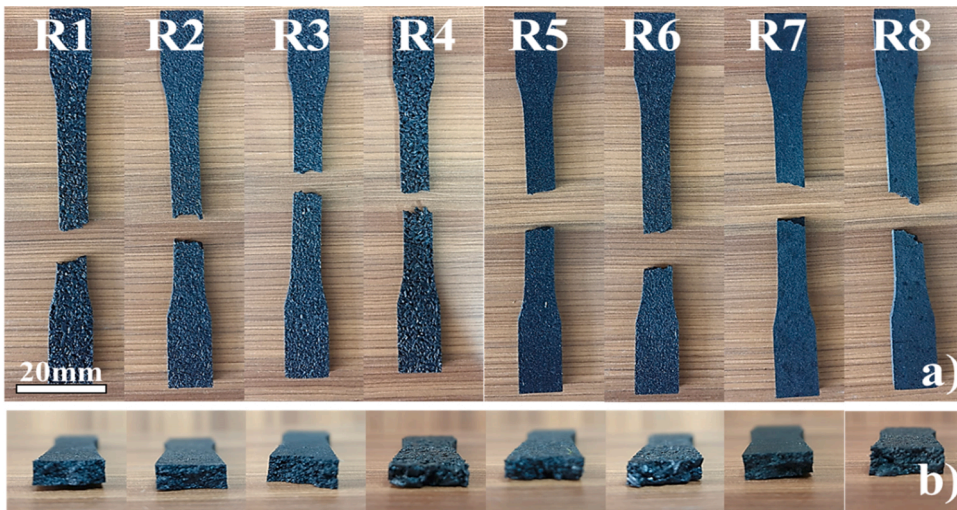


Fig. 7. Fracture after tensile stress. a) Failed samples and b) cross-section of the samples.

400 °C and 4 W/g, representing the maximum fusion speed of the material. From this point, a small concave zone continues upward indicating a gummy state, where the polymer chains of the material have free movement, absorbing energy to increase its temperature and mobility, which is converted into heat capacity. Finally, the area where the curve decreases indicates a low heat flow.

TGA results showed in Fig. 10b starts in 100 wt (%) and 0 °C and finishes at approximately 30 wt% and 40 wt% at around 650 °C for the solid and dashed curved lines respectively. The region between 100 and 300 °C represents the presence of a controlled diffusion-reaction, where the transport of reactive material is limited, and the reaction proceeds at an almost constant speed (almost constant slope in the curve). It is highlighted the region between 200 and 300 °C where the rubber melts and the presence of a possible hollin combustion during the gas change. Between 300 and 400 °C the curves indicate a release of moisture during the fusion process.

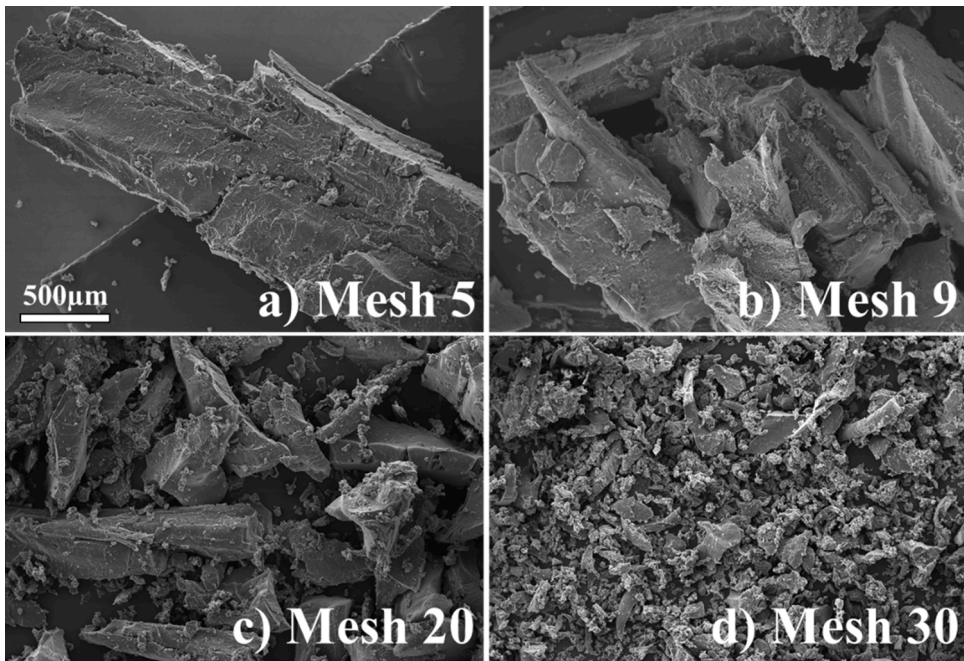


Fig. 8. SEM images for rubber powders. a) Mesh 5, b) mesh 9, c) mesh 20, and d) mesh 30.

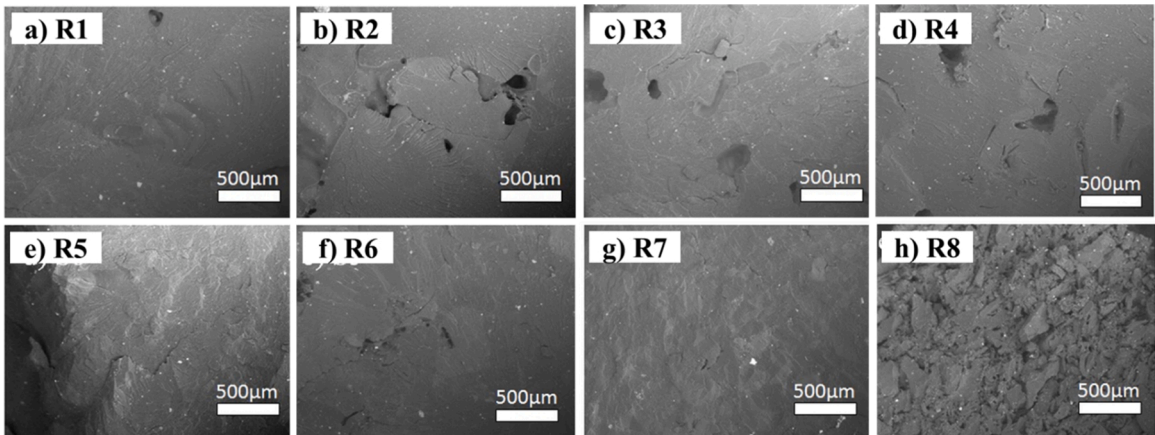


Fig. 9. SEM images for all rubber-resin formulations.

Table 2
SEM-EDS results for rubber powder.

Element	Weight (%)	Atomic (%)
C	80.56	88.12
O	10.46	8.59
Si	5.57	2.61
Zn	3.40	0.68
Totals	100.00	100.00

In this region, the constituents of the sample decompose very quickly, and the TGA signal suffers backward distortion. Finally, the region of the curve after reaching 500 °C represents a zone of constant weight. For the TGA analysis, at temperatures below 150 °C, the loss of components such as water, low molecular weight organic solvents, or gas desorption appeared. At temperatures between 150 °C and 250 °C there is a loss of low molecular weight components such as additives, bounded water, plasticization, or first decomposition products. At temperatures between 250 and 500 °C, the process of thermal degradation (inert gas) or thermo-oxidative

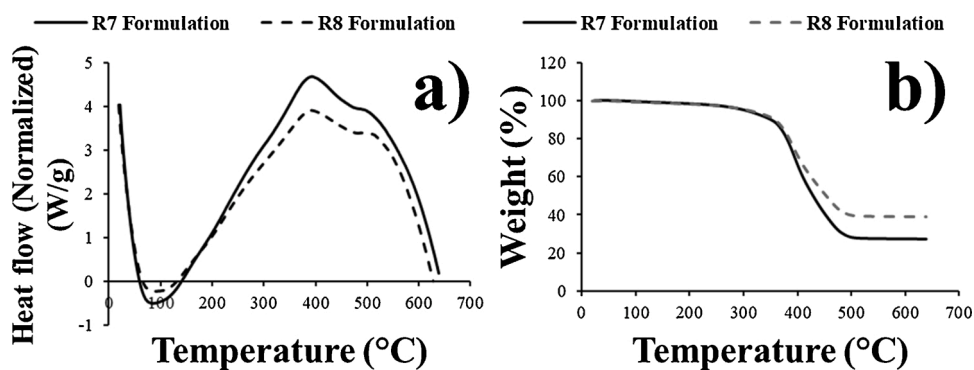


Fig. 10. Results. a) DSC analysis and b) TGA analysis.

decomposition (O2) begins. At temperatures above 500 °C, the carbonization of hydrocarbon compounds occurs whose pyrolysis does not lead to the formation of volatiles and there is the presence of ash of metal oxides or non-oxidizable salts.

Fig. 11 shows the results of the hardness tests under two scales, shore scleroscope and Rockwell type A (Diamond cone 60 Kg). According to ASTM D2240 [25], hardness tests on polymers are commonly measured using a durometer or Rockwell method, where for elastomers such as rubber, type A is mainly used on the Rockwell scale. It is possible to show that the results are similar for the two methods, the peaks of the formulations 2 and 5 are highlighted, which contain a higher hardness. Sample R1 has the lower hardness perhaps due to the use of a lower C/R ratio (10) and larger grain size (4 mm), which corresponds to resin in excess, which reduces the property of the composite. The particle size does not have a great influence on the hardness, only the resin content affects this property where the hardness decreases proportionally with the decrease in the resin content except for the formulations R1 and R2. It is important to note that in the hardness results, the high error ranges presented by the hardness curves can be affected by several factors, such as the smoothness of the test surface at the time of the bounce of the sphere, the mass of the sphere, the depth and the contour of the sample.

Fig. 12 shows FTIR spectra for R4 and R6 Formulation, rubber powder mesh 30 and, polyurethane resin. Fig. 12c shows peaks of the $-\text{CH}_2-\text{CH}_2-$ vibrations at about $400-500\text{ cm}^{-1}$. A signal appears at about 1000 cm^{-1} which represents a $=\text{C}-\text{H}$ bond. In the range at $1372-1447$ appears two peaks which belong to CH_2 vibration and, the peaks at 1505 and 1538 cm^{-1} were assigned to the $\text{N}-\text{H}$ stretching and $\text{C}=\text{S}$ bond respectively. Peaks of $\text{C}-\text{H}$ stretching vibration were showed are $2800-3000\text{ cm}^{-1}$, and a little signal at $2900-3000\text{ cm}^{-1}$ represents the $\text{C}=\text{C}-\text{H}$ bond. For Fig. 12d, it was possible to observe several peaks of the $\text{C}-\text{H}$ vibration ($1200-1600\text{ cm}^{-1}$) and the $\text{C}-\text{H}$ stretching vibration ($2800-3000\text{ cm}^{-1}$). The peak at 800 cm^{-1} was assigned to the $\text{C}-\text{N}$ bond, while the peak at 1100 cm^{-1} was assigned to the $\text{C}=\text{O}$ bond, which probably comes from an aromatic ketone or an ether. Peaks at $1300-1400\text{ cm}^{-1}$ and, 1500 cm^{-1} , represent $\text{O}-\text{H}$ and $\text{N}-\text{H}$ bonds respectively. The cyanide group is represented in the range $2200-2300\text{ cm}^{-1}$. R1 and R8 formulations represented in Fig. 12a and b showed several peaks as a combination between peaks of the base materials represented in Fig. 12c and d. In general terms, the main peaks represented in the spectra show the main bonds which compound the structure of polyurethane resin and rubber as a base material evidencing a chemical interaction between these materials.

From the Atomic absorption spectroscopy results [26], see Table 3, metals such as Pb, Cd, Cr, Ni, and Zn can be leached from the rubber and resin. The zinc content for both rubber (13.958 mg/kg) and resin (11905 mg/kg) is higher than the other heavy metals content. Since tires rubber have about 1 to 2 wt% of Zn, this leaching can also contribute to the environmental pollution [27,28].

Table 4 provides information on the maximum concentration of metal contaminants with toxicity potential. The Atomic absorption spectroscopy results when compared with the data reported by EPA-40 CFR Part 261 and some Colombian decrees and resolutions, such as Decree 4741 of 2005 and Resolution 2115 of 2007, show that there is an excess of Pb (7.326 mg), Ni (2.820 mg) and Zn (11905 mg) in the leachate content of the rubber samples, which exceeds the limit allowed by the American standard (5 mg for Pb, 1 mg for Ni, and 70 mg for Zn). For Colombian standards the content of all metals exceeds the allowed limit. However, Cd and Cr levels remained below the maximum contamination limit for both rubber and resin by American standards. The release of metals at these concentration levels depends not only on the composition of the tire, but also on the tire used before being discarded. It is important to know that these results do not limit completely the use of this waste, on the contrary, give guidelines to use them in the proper applications and avoid at all just landfills or other worst disposition. These results confirm that tire rubber and roads can be a source of hazardous materials, which can include lead and arsenic [29].

In Colombia tire industry has a high impact in the economy, and beyond the manufacturing place its production requires diverse raw materials such as water, energy, hydrocarbons, textiles, steel, sulfur, pigments among others, which necessarily implies an impact on the environment [33]. Scrap tires, upon completing their life cycle, become a solid waste that is managed as waste even in processes that involve recycling or remanufacturing. This is a burden that adds significant value reflected in costs associated with waste disposal and in many cases becomes an obstacle to improve resource efficiency [3]. Therefore, the treatment, reuse, and recycling of solid waste have become an opportunity to get different materials to be reincorporated into production processes, increasing their useful life, and reducing the negative environmental impacts generated by the different products and materials. Colombia can process 10 million tires per year, but it only recycles about 20 % because many of the products obtained do not meet marketing standards and also, there is no culture to develop a specific market for the treatment of used tires to make the rubber market more attractive, taking advantage of its

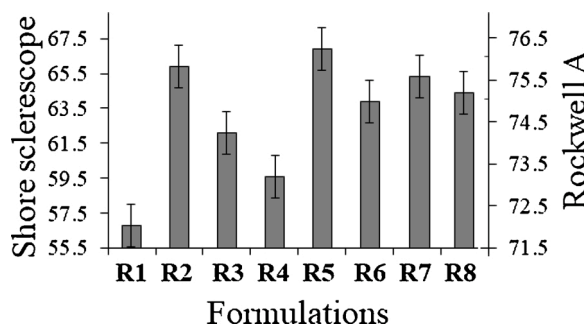


Fig. 11. Scales for hardness test.

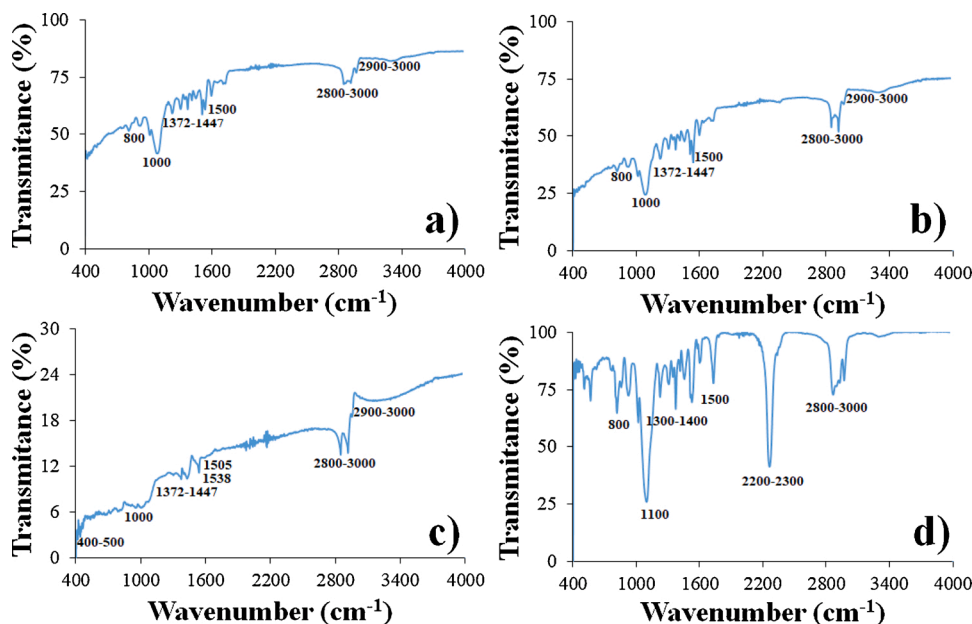


Fig. 12. FTIR test. a) R4 formulation (rubber mesh 9), b) R6 Formulation (rubber mesh 20), c) rubber powder mesh 30 and, d) polyurethane resin.

Table 3
Atomic absorption spectroscopy test results.

Element	Method	Sample	
		Resin (mg/kg)	Rubber waste (mg/kg)
Pb	Internal method PS-O-005 LAE	0.245	7.326
Cd		0.020	0.276
Cr		0.617	2.090
Ni	Internal method PS-O-001 LAE	1.292	2.820
Zn		13,858	11905

properties and prices in various sectors of the industry.

The rubber granules from the mechanical crushing process of used tires is the main raw material for various applications such as the development of modified asphalts, offering higher quality conditions in the construction of roads [34]; in concrete where vibration damping is needed; for filling ditches and piping beds, where resistance to impact or blasting is required; as in railway dampers; jersey barriers and bunkers [35]; and for the elaboration of decorative floors in sports and children’s environments [36]. Unfortunately, the tire recycling market has not been exploited yet and part of the material derived from the tire for the aforementioned applications has been imported from countries such as Spain and Trinidad and Tobago, reducing the price competitiveness of the national product [37].

Many different rubbers have been formulated elsewhere based on properties such as tensile strength, elongation at break, permanent deformation at the break, hardness, temperature of brittleness among others. On the other hand, a rubber formulation has a

Table 4
Maximum Concentration of Contaminants for the Toxicity Characteristic.

Contaminant	CAS No. ^a	Regulatory Level (mg/L)		
		Regulation CFR EPA 40 part-261 Government of the United States [30]	Decreto 4741 de 2005 Government of Colombia [31]	Resolución 2115 de 2007 Government of Colombia [32]
Cadmium	7440-43-9	1.0	1.0	0.003
Chromium	7440-47-3	5.0	5.0	0.05
Lead	7439-92-1	5.0	5.0	0.01
Nickel	7440-02-0	1.0	N/A. ^b	0.02
Zinc	7779-88-6	70	N/A. ^b	3

^a Chemical abstracts service number.

^b Information not available.

wide variety of additives of the process, including accelerators, activators, antidegradants, bonding additives, fillers, resins, among others [38,39]. In order to further improve the tension properties of the samples formulated in this research, it is recommended the addition of silica and other additives [40,41]. The use of polyurethane resin as a wetting additive for rubber particles in the lamination process favors fragmentation damage in the tiles for future applications [18].

Finally, a successful small business enterprise company for the rubber waste processing was born from this project, Ecoreproducts, which is now commercializing a wide variety of products made of rubber powders with polyurethane binder, see Fig. 13. Up to day, about 100 tons of waste is used, which is a great impact for the city, and if the project is multiplied, the impact in the country will be large.

This research is quite significant from the environmental point of view because the produced formulations used about 90 % of recycled material. This is a real upcycling solution for this waste type, and it is also an economic alternative as shown in the developed company.

If this process is scaled at super large scale, at least in many countries the rubber tire waste will be fully used in this application.

4. Conclusion

This research showed a successful example of university-industry work in a small business enterprise, giving as result a large-scale

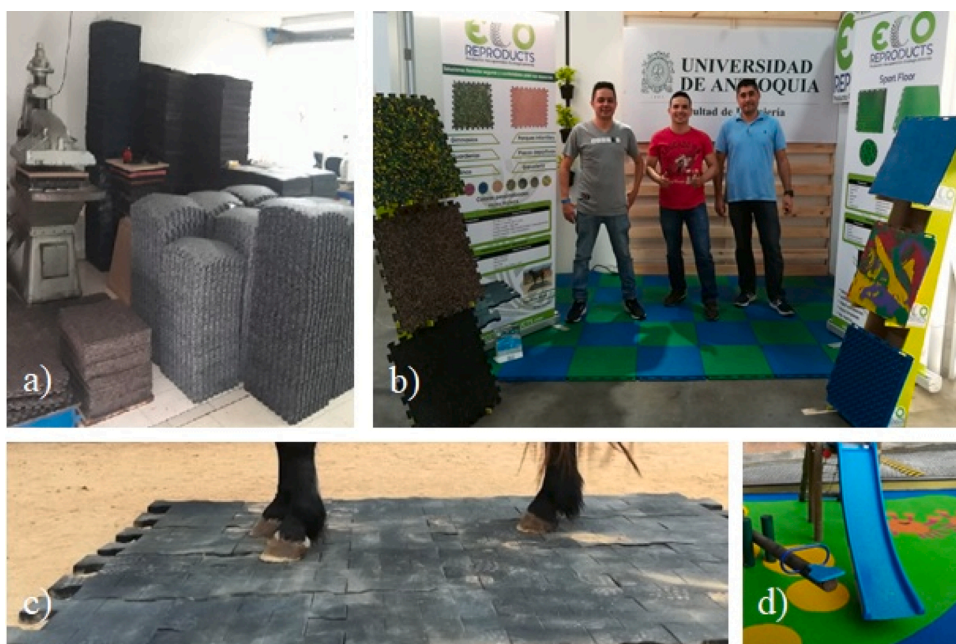


Fig. 13. Entrepreneurship Company of recycled rubber tires: a) product storage, b) sells people in a large city business fair, c) heavy duty horse floor product, d) kids playground product.

process that can be multiplied in the city, and also copied everywhere, giving not only a sustainable solution for people as a business, but also for reducing pollution of rubber tire waste. The resultant manufactured composite material can be used in several applications due to its flexibility and other properties, especially in applications where impact resistance is one of the main requirements. Moreover, upon the use of different additives, colorants, and reinforcements, multiple composites with fibers [42] [43], particles [44], nanocomposites [45,46], and diverse resin binders [47,44], can open up new business and research projects for other applications. It has been shown in this research that rubber tires leach many components, and some of them can be in harmful doses for the environment. Therefore, a smart use of this solid waste has to be done in order to increase the positive impact related to its use in a product that increase the material circularity. A new generation of circular economy derived materials [48] with smart building and architecture [49,50] as part of smart and sustainable cities is urgently needed.

Funding

This research has been funded by the Universidad de Antioquia.

Declaration of Competing Interest

The authors report no declarations of interest.

Acknowledgments

The authors wish to thank the engineer Juan C. Salazar from Prismacaucho S.A.S. for his support and useful suggestions in this investigation.

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