





Use of end-of-life tires for the production of a waterproofing agent as a strategy to promote the circular economy

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Abstract

This paper develops a formulation to produce a laboratory-scale waterproofing for exterior walls and roofs using recycled tires. It begins by identifying its chemical composition using Fourier transform infrared spectroscopy and selects a technology using the ELECTRE method. It uses a reticular simplex mixing design of experiments to adjust the resin proportions according to their mechanical properties. In addition, it evaluates the critical pigment volume content and parameters such as water absorption, permeability, total solids content and mechanical properties for quality assurance. Acrylic emulsions were found to be favorable in cost, application, safety, and efficiency; the flexible resin offers better elongation and the rigid one better tensile strength, with less than 10% tire reducing porosity. The results support the competitiveness of the product and confirm the effective use of recycled tires in waterproofing, offering a favorable and functional approach to construction

Keywords: waste tire; circular economy; waterproofing; waterborne coating

Aprovechamiento de neumáticos al final de su vida útil para la producción de impermeabilizante como estrategia de promoción de la economía circular

Resumen

Este trabajo desarrolla una formulación para producir un impermeabilizante a escala de laboratorio para paredes exteriores y cubiertas utilizando neumáticos reciclados. Inicia identificando la composición química mediante espectroscopia infrarroja por transformada de Fourier y selecciona una tecnología mediante el método ELECTRE. Utiliza un diseño de experimentos de mezcla simple reticular para ajustar las proporciones de resina en función de sus propiedades mecánicas. Además, evalúa el contenido crítico de volumen de pigmento y parámetros como la absorción de agua, permeabilidad, contenido total de sólidos y las propiedades mecánicas para el aseguramiento de la calidad. Las emulsiones acrílicas resultaron favorables en coste aplicación, seguridad y eficacia; la resina flexible ofrece mejor alargamiento y la rígida mejor resistencia a la tracción, con menos de un 10% de porosidad reductora del neumático. Los resultados apoyan la competitividad del producto y confirman el uso eficaz de neumáticos reciclados en la impermeabilización, ofreciendo un enfoque favorable y funcional para la construcción.

Palabras clave: neumáticos usados; economía circular; impermeabilización; recubrimientos base agua

1 Introduction

Building materials formulations and selection have evolved over the past few decades in response to greater energy efficiency goals and a greater understanding of building science [1]. Although conventional coatings derived

from fossil feedstocks and based on solvents exhibit superior physical and mechanical properties [2,3], their use increases environmental damage and extraction costs, especially in the face of dwindling supplies of resources such as oil and natural gas [4]. Consequently, the materials industry has undergone a significant transformation toward the integration

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of renewable raw materials and emerging technologies, with a particular focus on sustainability through the use of water as a liquid vehicle in formulations [5]. This move towards a circular economy reflects the sector's persistent efforts to innovate and reduce environmental impact, contributing to sustainable development goals [6,7]. Within this trend, the incorporation of recyclable materials as additives seeks not only to reduce costs but also to improve key mechanical properties, such as tensile strength and durability [8-13].

The production of efficient and low-cost waterproofing has become a crucial breakthrough for building protection [14] and whose formulations are based on polymeric compounds in a liquid vehicle, either solvent or emulsion in water, together with viscosity-reducing additives, fillers, and fibers [15] highlighting the use of a waste with high recoverability potential such as end-of-life tires as a promising solution [16]. Currently these wastes represent a major environmental problem [17]. It is estimated that the annual generation of tires amounts to 1.5 billion whole tires worldwide [18]. In Colombia, between 5,5 and 6,7 million units of tires are imported to meet the growing demand for mobility [19] identifying that in the capital around 4,000 tires are collected weekly by the Mayor's Office that are not destined for any method of reuse [20]. They are not considered a hazardous waste; however, they are highly bulky, and their composition makes their degradation almost impossible [21-23].

The reuse of these tires is therefore a practical and economical solution supported by studies suggesting that tire rubber powder is suitable for the industrial production of waterproofing coatings [17,24].

This article focuses on designing a formulation to produce a waterproofing agent that, through the correct selection and dosage of compounds together with the physicochemical transformation of the tires, reincorporates this waste into the production chain as a secondary raw material. The main objective of the project is to offer an alternative to the current problem of managing these wastes, contributing to sustainability and the extended life cycle of the materials. This approach not only helps preserve natural resources and mitigate negative environmental impacts, but also supports three sustainable development goals: SDG 9 (innovation and infrastructure), improving the properties of construction materials; SDG 11 (sustainable cities and communities), properly managing waste and improving infrastructure; and SDG 12 (responsible production and consumption), reducing the environmental impact of waste and improving the production of waterproof construction materials.

2 Materials and methods

2.1 Characterization of the waste Tire

Tire dust particle size registered by the manufacturer of ≤0,71mm was used to analyze the possible chemical composition of the residue by Fourier transform infrared spectroscopy (FTIR) using the Spectrum Two equipmentTM and comparing the spectra with the library available in the Spectrum softwareTM 10.

2.2 Technology selection

The ELECTRE (Elimination Et Choix Traduisant la Realitè) multi-criteria decision-making methodology was applied to evaluate and select the most favorable technology. First, low cost, trouble-free application, waterproofing efficiency and low chemical risk were defined as the criteria for evaluating the different available technologies. Second, a weight was assigned to each criterion by means of a survey of 37 people, who indicated their order of preference when purchasing a waterproofing product. Finally, each alternative was rated according to the information available in the literature and provided by the manufacturers, normalizing the values on a scale of 0 to 1 with correspondence intervals of 0,25. This allowed obtaining the initial decision matrix used to run the ELECTRE method and thus identify the hierarchy of technologies.

The ELECTRE method considers the relationship between the alternatives considering both the absolute values of performance and the significant differences between them and thus determine the dominance between technologies. Therefore, once this matrix was obtained, we proceeded to create the concordance and discordance matrix whose indexes were calculated with eq. (1), (2) respectively.

$$C_{ik} = \sum_{j:c_j(A_i) > c_j(A_k)} w_j + \frac{1}{2} \sum_{j:c_j(A_i) = c_j(A_k)} w_j$$
 (1)

$$D_{ik} = \frac{\max |c_{j}(A_{i}) - c_{j}(A_{k})|_{j:c_{j}(A_{i}) < c_{j}(A_{k})}}{\max |c_{j}(A_{i}) - c_{j}(A_{k})|_{j:c_{i}(A_{i}) > (A_{k})}}$$
(2)

Subsequently, the concordance and discordance thresholds, c and d, were calculated from a simple average with the values of their respective matrices, which allowed the creation of the concordant and discordant dominance matrices, such that if $C_{ik} > c = 1$ if, if $C_{ik} < c = 0$ and that if $D_{ik} > d = 0$ and if $C_{ik} < d = 1$. Finally, the two dominance matrices were multiplied to obtain the aggregate dominance matrix and thus obtain the ELECTRE graph that allowed the identification of the most favorable technology.

2.3 Sample preparation

Sample preparation was conducted in three stages. The first consists of the preparation, in a paddle stirrer the water was mixed with the wetting agent, the dispersant and the thickener for 2 minutes before adjusting the pH to 8 and continued mixing until a gel was formed, then a third part of the defoamer was added. The second stage is the dispersion stage, in which the pigment (titanium dioxide) and tire powder are added to the previous mixture. This stage is the most important, so it was left to mix for 15 minutes. Finally, the second part of the defoamer is added. The final phase is the adjustment phase; in this stage the polymeric dispersions were added together with the coalescent agent, the rheology modifier, the acrylic thickener, the biocide and finally what was left of the defoamer.

2.4 Tensile test

To prepare the specimens, a mold was used with the dimensions defined by the ASTM D638 standard for a type IV specimen, which is used when a comparison between soft polymers and other more rigid polymers is sought. The specimens were molded with acrylic sheets and kept for 4 days under standard laboratory conditions, then placed in a drying oven at 40°C for 2 days before the tensile test.

2.5 Experimental design

An experimental design of simple cross-linked mix was conducted, considering as response variables the maximum stress (MPa), elongation at break (%) and maximum force (N) obtained from tensile tests performed in a SHIMADZU universal testing machine, with a maximum capacity of 50 kN and 10 kg, following the ASTM D2370 standard. Additionally, a design of experiment with simplex reticular (2,4) mixtures was used for two components, rigid resin and flexible resin, with two levels 70/30 and 90/10, obtaining two replicates for each response variable. The results were analyzed by analysis of variance using Minitab with quadratic adjustment to determine the impact of resin proportions on the mechanical properties of the final product.

2.6 Quality test

Viscosity, density, volume of solids and weight of solids tests were conducted according to standards D2196, D1475, D2697 and D1644, respectively, for the liquid material. Additionally, the capacity of the already applied and dry waterproofing was determined in an extreme situation of water presence, allowing it to pass through it without altering its internal structure, that is, without suffering deterioration, by means of the permeability test calculated from the measurement of the amount of water passing through the dry waterproofing film for one week. On the other hand, water absorption was evaluated to determine how well the product performs under extreme humidity conditions, immersing the test specimens in distilled water for seven days. At the end of this period, the percentage of water absorption of each specimen was calculated by weight difference. Finally, the elongation at break and tensile strength were measured. All tests were compared to the range required by ASTM D6083/D6083M - 21 "Standard Specification for Liquid-Applied Acrylic Coating Used in Roofing".

3 Results and discussion

3.1 Chemical composition of the waste

Tires are complex materials containing various components to function in various environments, their end-of-life properties are determined by the particle size according to the grinding technology and composition, which influences their mechanical behavior and longevity.

All tires contain four groups of fundamental materials: natural (polvisopropene) and synthetic rubbers such as butadiene (BR) hydrogenated nitrile-butadiene (HNBR), styrene-butadiene (SBR) and ethylene-propylene-diene (EPDM), carbon blacks and silicas, reinforcing materials such as metals and/or textiles, and chemical additives [24] as vulcanizing agents whose objective is to facilitate the irreversible cross-linking of rubber macromolecules [25]. They are all selected and dosed to improve properties such as abrasion resistance in the tread, flexural strength in the sidewall and impermeability of the inner liner [26]. In addition to increasing resistance to biodegradation, photochemical decomposition, chemical reagents and thermal degradation [27]. Fourier transform infrared spectroscopy was used to identify various compounds present in the recycled material sample.

Fig. 1 shows the % transmission of infrared light as a function of wavelength in cm⁻¹, which is presented in such a way that the higher frequencies are on the left and the lower frequencies on the right. In the stretching region comprising frequencies between 1400 and 4000 cm⁻¹, two types of bands are identified, a strong one between 2800 and 2950 cm⁻¹ corresponding to stretching vibrations between C-H bonds with sp3 hybridization carbons, suggesting the presence of the methyl group and aliphatic structures. Another moderate band between 1500 and 1550 cm⁻¹ is found within the double bond region which may suggest the presence of 1 carbonyl group. Fig. 1 also contrasts the spectrum of the recycled tire with the known spectrum of zinc stearate which presented the highest coincidence with 66.1%, this means that the chemical characteristics of zinc stearate can coincide with several other substances present in the tire dust, generating a coincidence in the spectral bands and/or that the transmittance bands of zinc stearate can be very intense, dominating the spectrum even if it is present in a minor proportion within the sample.

Between 550 and 1400 cm⁻¹ is the fingerprint region, it means, characteristic vibrations of each molecule, so reference spectra of different polymers were used to identify the identity of the sample. The weak bands 1, 2, 3 and 4 of the bending regions in Fig. 1 have a possible identification at frequencies 1461 cm⁻¹, 960 cm⁻¹, 722 cm⁻¹ and 699 cm⁻¹ of the functional groups' propylene, butadiene, ethylene and styrene [23,28-30] respectively, present in EDPM, SBR and NR rubber polymers.

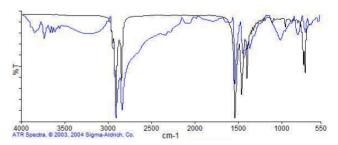


Figure 1. Comparison of tire FTIR spectrum (blue line) with zinc stearate FTIR spectrum (black lines) from the library available in SpectrumTM 10 software. Source: Own elaboration

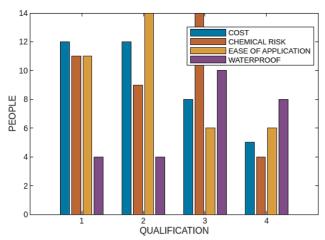


Figure 2. Consumer preference results Source: Own elaboration

3.2 Selection of technology

Waterproof coatings are complex materials that contain binders. These are especially important in heterogeneoussystems, such as emulsions and dispersions, where their main function is to maintain a firm and stable structure [31]. The wide variety of available waterproofing systems allows the selection of a wide range of materials and techniques that provide and ensure product performance and durability. Some of the most commonly used water-based waterproofing systems in construction are silicone, polyurethane, acrylic, and asphalt emulsions.

Fig. 2 shows the survey results, determining that the most important criterion when acquiring a waterproofing product is its efficiency in waterproofing, while cost has the least weight.

The weights assigned to each criterion are detailed in Table 1 shows the normalized ratings for each alternative based on the information available in the literature. Additionally, Table 1 presents the standardized ratings of various waterproofing technologies according to the literature. Considering that siliconized resins are hydrophobic and resistant, but costly [32]; polyurethane membranes are durable and adhesive, but expensive and toxic [33]; acrylic technologies are inexpensive and UV-resistant [34]; and asphaltic emulsions, derived from petroleum, are consistent, adhesive and durable, in addition to being low-cost [35].

Once the alternatives were compared with each other, the Electre method was run and the hierarchy of preference in the alternatives studied was obtained together with the identification of their net flows. Fig. 3 illustrates the outflows in which one alternative is preferred over others, and the inflows in which other alternatives are preferred over these.

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Table 1. Normalized decision matrix

-	Criteria			
Alternatives	Cost	Ease of application	Chemical risk	Water resistance
Silicone	0,20	0,75	0,50	0,55
Polyurethane	0,10	0,50	0,35	1,00
Acrylic	0,60	0,70	0,60	0,60
Asphalt	0,70	0,50	0,20	0,70
Criteria weight	0,1	0,2	0,3	0,4

Source: Compiled by the author

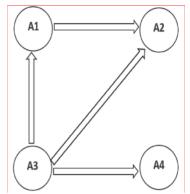


Figure 3. ELECTRE network, net flows of each alternative Source: Own elaboration

In the case of polyurethane emulsions (A2), it is a technology that does not have outflows, that is, it is not considered preferable with respect to the others, in spite of having excellent waterproofing efficiency, other criteria such as cost and its components that reduce its chemical safety make other technologies stand out, as is the case of acrylic (A3), which was positioned in first place as the most preferable, balancing each of the criteria. Therefore, it was chosen as the base technology on which the tire-based waterproofing was formulated.

3.3. Additive study

The incorporation of additives improves the miscibility of polymer blends by lowering their interfacial tension, which is called compatibilization. The wetting agent forms an envelope around the pigment particles and the dispersant improves the incorporation of pigments and fillers into a coating, ensuring its stability [36]. The pH regulator neutralizes the coating to prevent pigment shock, the rheology modifier controls viscosity and provides a suitable texture for application [37,38]. The defoamer reduces foaming during emulsion manufacture and application [39]. The biocide prevents the growth of microorganisms in the emulsion and prolongs its shelf life. The thickener optimizes the flow behavior by adjusting the viscosity for storage, processing and application and the coalescing agent acts as a film former [40]. Table 2 shows the additives that were selected in order to achieve good stability and excellent coating properties.

Table 2.
Base formulation and function of additives

Additive	Type	%
Moisturizer	Indole, nonionic wetting agent	0,1
Dispersant	Sodium polyacrylate	1,2
pH Regulator	Sodium silicate	0,1
Rheology modifier	Monoethylene glycol	0,4
Defoamer	Polyacrylate defoamer	1,2
Biocide	Stabilized aqueous solution of isothiazolinones with bromine derivatives.	0,2
Thickener	Hydroxy-propoxymethylcellulose	0,4
Coalescing agent	Haltane isobutyrate	1,2

Source: Compiled by the author

3.4. Formulation adjustment

3.4.1 Percentage change (%) of tire

As pigment volume content (PVC) increases and approaches critical volume content (CPVC), properties such as gloss and blistering decrease, while permeability, susceptibility to oxidation, and viscosity increase [37]. This abrupt change is observed experimentally, so the percentage of tires was varied first. Fig. 4 shows that at 0% tire, the porosity is low and the structure is compact. At 5%, porosity increases slightly, improving shock absorption without compromising strength. At 10%, a significant increase in porosity is observed, with more prominent particles and pigments beginning to agglomerate, indicating proximity to the critical pigment volume content, and at 23%, the lack of binder increases pores, resulting in low quality. Therefore, formulations with PVC below CPVC, such as 5% tire, are preferred. And although incorporating as little as 10% by weight of tire in polymeric matrices would mean a large consumption of scrap tires [41] even a smaller percentage, such as 5%, can represent a significant impact. Incorporating 5% by weight of recycled tire is still beneficial because, on an industrial scale, it translates into large volumes of recycled material. This approach not only helps reduce the volume of tire waste that ends up in landfills but also allows it to flow continuously into production cycles, as well as maintaining the desired properties of the final material.

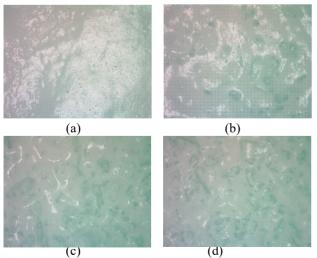


Figure 4. Porosity of different tires % a) 0%, b) 5%, c) 10% and d) 23% Source: Own elaboration Table 3.

Results of mechanical tests for different proportions of rigid and flexible

Rigid resin	Flexible resin	Maximum force (N)	Maximum stress (MPa)	Elongation at break (%)
70	30	10,8322	0,7952	450,0765
	30	11,4759	0,7332	402,3961
30	70	5,0068	0,3599	294,5056
	70	4,1564	0,2832	198,8111
00	90 10	9,7116	0,9502	295,5902
90		9,7672	0,9429	267,4216
10	90	3,8068	0,2973	191,8667
10	90	3,1710	0,2343	183,8111

Source: Own elaboration

Once it is determined that 5% of the total weight of the formulation corresponds to recycled tire powder and 35% is made up of additives, solvent (water), and pigment, the remaining 60% is attributed to the acrylic resin, which is further divided into rigid and flexible resin.

Selecting an optimal resin ratio that satisfies 60% of the formulation results in improved mechanical properties, impermeability, and resistance to environmental conditions of the coating, ensuring efficient protection for terraces and exterior walls. It is expected that increasing the proportion of flexible resin will improve the elasticity and adaptability of the material, while increasing the proportion of rigid resin can improve tensile strength and therefore durability.

The results of varying the resin ratio are shown in Table 3, where it can be observed that as the proportion of rigid resin increases, the maximum tension tends to increase in the same way. However, the behavior for the other response variables is not similar. Regarding elongation at break, it would be expected that a higher percentage of flexible resin would result in a higher percentage, however, the highest value is for 70R/30F. This behavior can be visualized in Fig. 5, corresponding to the contour plots for the response variables of maximum strength and elongation at break. It can be observed that for higher force values, the proportion of rigid resin is between 60 and 100, and for higher elongation at break percentages, the optimal proportion of rigid resin is between 60 and 80.

Before selecting the proportion to use, a statistical analysis of the data was performed. First, the goodness of fit was identified to determine how well the statistical model fits the data. For maximum stress, maximum force, and elongation at break, the R2 values are 99,0%, 98,24%, and 90,64% respectively, indicating that the model explains the variability of the data better for maximum stress compared to the other response variables. On the other hand, to determine the level of confidence in the obtained results, it is analyzed whether the results are statistically significant, which indicates an insignificant risk of being due to chance.

First, the "p" values shown in Fig. 6 were evaluated, corresponding to the probability against the null hypothesis for lack of fit, in order to determine if the model required more terms or data transformation. For the three response variables, the "p" values are greater than 0,05 indicating that no lack of fit was detected and that the model explains the relationship between the predictors and the response.

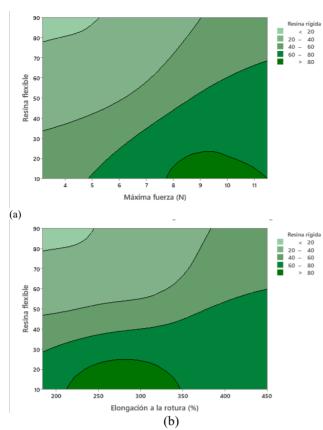
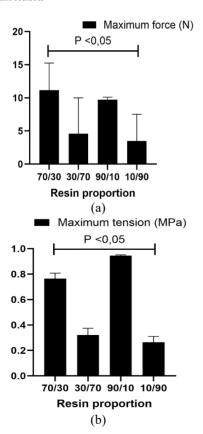


Figure 5. Contour charts for (a) maximum strength and (b) elongation at break. Source: Own elaboration



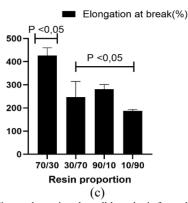


Figure 6. Probability results against the null hypothesis for each of the studied mechanical properties evaluated with alpha 0,05. (a) Maximum force, (b) Maximum tension and (c) Elongation at break. Source: Own elaboration

Additionally, the "p" values for the interaction between resins were examined, which were found to be less than 0,05, indicating a statistically significant difference when working with a significance level of 95%. This indicates that at least one of the terms in the group has an effect on the response, leading to the rejection of the null hypothesis

Finally, the regression coefficients for each response variable presented in Table 4 were analyzed. The coefficients explain that for maximum stress, the rigid resin has a significantly higher coefficient (0,9412) compared to the flexible resin (0,3716), and the interaction has a negative coefficient (-0,0551), indicating that an excess of flexible resin can reduce maximum stress. For maximum force, the interactions between the rigid and flexible resins are statistically significant and balanced proportions can be managed. The rigid resin has a higher coefficient (6,052) compared to the flexible resin (4,877), indicating a greater contribution of the rigid resin to maximum force. Lastly, in terms of elongation at break, the flexible resin has a much higher coefficient (310,8) than the rigid resin (21,6) and the interaction is positive (779), suggesting that a higher proportion of flexible resin increases elongation at break.

Since the model shows that maximum stress is more closely related to the rigid resin, a higher proportion of this in the mixture is preferable. Therefore, a 95/5 ratio of rigid resin to flexible resin would be a suitable choice to maximize maximum stress.

Table 4.
Results of estimated regression coefficients for each response variable

Term	Maximum stress Coefficient	Maximum force Coefficient	Elongation at break
Rigid resin	0,9412	6,052	21,6
Flexible resin	0,3716	4,877	310,8
Rigid resin* Flexible resin	-0,551	12,28	779

Source: Own elaboration

3.5. Quality test

Quality tests are performed to obtain the technical specifications of the finished product and their values are recorded in Table 5. The waterproofing agent is white with slight black particles, which is beneficial for exterior walls and roofs as it reduces heat accumulation by reflecting light. The viscosity of the product at 12500 cps is suitable for trouble-free application. On the other hand, the solids volume exceeds 50% and the solids weight is greater than 60%, ensuring that a larger portion of the applied product remains on the surface. Thanks to the water-based formulation, the curing time is longer, however, a low content of volatile organic compounds is guaranteed.

A pH around 8,5 ensures optimal dispersion efficiency [37]. In terms of the mechanical properties of the material, they exhibit an elongation at break percentage greater than 100%, indicating high flexibility and adaptability to movements and deformations of the building, along with sufficient tensile strength to support pedestrian traffic in the case of being used on terraces, ensuring greater durability of the material and better resistance to external loads and environmental stresses, resulting in more effective protection of the building against aging and weathering.

Finally, the efficiency of the waterproofing is reflected in the properties of permeability and water absorption. The material's permeability is 12 Perms, which is a convenient value and reflects proper formulation and selection of tire percentage by avoiding particle agglomeration and pore formation. However, the water absorption of 20% indicates that the material can absorb a moderate amount of water, so it is recommended to apply the material where there is not a high and prolonged accumulation of water, or a good drainage system is suggested.

The obtained formulation shows good physical and mechanical properties, as well as high efficiency in waterproofing. This not only ensures that the product is competitive in terms of performance but also demonstrates that tires can undergo an extended life cycle by transforming into valuable materials that enhance the properties of products used in the construction industry.

Table 5.

Quality test results

Property	ASTM	Units	Value
Color	=	-	White
Viscosity	D2196	cps	12500
Density	D1475	g/mL	1,39
Volume of solids	D2697	%	>50
Weight of solids	D1644	%	>60
pН	-	-	8,9
Curing time	-	hours	96
Elongation at break	D2370	%	295,59
Tensile strength	D2370	MPa	1,37
Permeability	D1653	Perms	12
Water absorption	D471	%	20

Source: Own elaboration

4 Conclusions

An appropriate process for collecting and processing tires at the end of their useful life is crucial to identify the compounds present in the waste and effectively select additives that ensure their compatibility and stability. Simultaneously, the application of a multicriteria analysis method like ELECTRE has allowed determining the favorability of available alternatives. This study has highlighted acrylic emulsions as the most convenient option, balancing criteria of cost, ease of application, efficiency, and chemical safety.

The statistical analysis revealed that the ratio between rigid and flexible resins directly impacts the mechanical properties, and to improve weather resistance, a formulation with a 90/10 ratio in favor of rigid resin was chosen. Additionally, controlling the volume content of critical pigment has been crucial to avoid particle agglomeration and pores that could compromise the quality of the final product; a content below 10% has shown improvements in physical properties, as evidenced in the final product quality tests. These tests guarantee good stability, with a pH that facilitates particle dispersion, a color that reduces heat accumulation, and a solids content that ensures optimal performance by retaining over 60% of the product on the surface once applied.

In conclusion, transforming tires at the end of their useful life into loads for waterproofing emulsions not only effectively extends their lifespan but also significantly improves the mechanical properties of the coating. After their useful life as a waterproofing agent, these materials can be recovered and reused in new products, thus completing their life cycle, minimizing waste without a destination, and maximizing the value of the resources used. This strategy not only addresses the environmental problem of tire disposal but also actively promotes the circular economy, standing out as an innovative and sustainable solution for the construction materials industry.

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