# Characterization of new rubber compounds with incorporation of recycled rubber powders

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**Abstract.** The global growth of discarded end-of-life tires (ELT) is leading to a significant challenge of waste management. Many of these are incinerated or exported to countries with less stringent environmental regulations, exacerbating pollution and wasting resources.
Until now, the main destinations for recycled rubber were floorings, rubberized pavements, infill for synthetic turfs and moulded goods. The increasing necessity of the industry to adopt more sustainable products and processes by incorporating recycled materials, needs to be supported by innovative solutions.
The present paper introduces a recycled rubber material, characterized by different certified laboratory tests performed to evaluate the mechanical and physical performance of a virgin rubber compound combined with different content levels of rubber powders derived from ELT, and with different particle sizes and production methods.
There is extensive research on the ELT management systems and the various methods for recycling rubber from end-of-life tires, but limited studies on the impact of particle size and the influence of content of recycled rubber powders, added in new rubber compounds suited for performance-demanding applications.
Based on the laboratory test results and the case study presented in this paper, it was possible to conclude that this recycled material can be incorporated into new rubber compounds, without compromising the performance.

## Introduction

There is an extensive body of evidence and studies that focus on the different methods for recycling of rubber from end of life tires, addressing their progresses and limitations, as well as several studies focused on well documented processes such as reclaiming, devulcanization, surface activation and modification and also on specific applications that have been studied for decades, such as rubberized concrete or rubber modified asphalt. There is limited available information and published studies on the influence of particle size and content levels of recycled rubber powders that can be incorporated into new rubber compounds, for various applications such as new tires, conveyor belts, gaskets and profiles.

The increasing number of waste tires is serious threat to the natural environment and human health. [1]. In 2019, the total amount of end-of-life tires (ELT) has been of around 30.9 million tons worldwide, with only 59% of all end-of-life tires being correctly disposed, while 41% are still landfilled, stockpiled or lost

A discarded tire can be mechanically recycled without the use of chemicals, by processing it through several stages of grinding and separation into three fractions: rubber, steel, and textile. The rubber can be recycled for the production of new tires usually referred to as a closed loop, or it can be used for other applications such as the production of rubber-modified-asphalt, playground surfaces, artificial turfs, and insulating panels. [2]

In the statistical data presented by the European Tire and Rubber Manufacturers' Association (ETRMA). In 2021, over 3.9 million tons of end-of-life tires were generated in Europe alone. Despite the advances in recycling, 40% of the tires produced annually in Europe are still incinerated, releasing large amounts of CO2 and other pollutants. Additionally, this incineration wastes valuable materials such as rubber, textile fibers and steel, which could be recycled and reused, promoting a circular economy.

The most popular industrial scale waste tires grinding technologies are the ambient and cryogenic methods. In the first method, waste rubber is passed through shearing mills, working at room temperature, while the cryogenic grinding method is based on effective cooling of waste rubbers below their glass transition temperature using liquid nitrogen. This results in the conversion of an elastic rubber material into a frozen brittle form, which can be easily crushed by using hammer mills. [3]

The purpose of this study is to investigate the cure characteristics, mechanical properties and processing behaviour of recycled rubber powder-filled virgin rubber compounds. By evaluating the physical and rheometrical properties of the tested formulations, we are able to better understand the impact on performance of the different particle sizes and content levels on a specific mixture.

There is a widespread interest in recycling of scrap rubber, driven by legislations that provide a sustainable solution to preventing the processing of tire waste for energy and material recovery [4].

The increasingly demanding requirements of the industry emphasize the need to thoroughly characterize recycled rubber materials, to assure its suitability to these new specific processes and compounds.

In order to meet the current market demands and overcome limitations, this study explores the development of a sustainable and cost-effective rubber compound by incorporating recycled rubber powder derived from End-of-Life Tires (ELT) with different content levels and particle sizes but also different production methods (ambient and cryogenic).

Innovation driven by scientific research can promote the use of recycled rubber from end-of-life tires in new products and production processes. This effort is supported by compiling reliable data that demonstrates the technical benefits of these recycled products. Through collaboration, innovation, and a focus on circularity, the industry can leverage these advancements in recycling technology to more easily incorporate recycled rubber. This approach drives progress and resilience while maintaining a commitment to performance and sustainability.

**State of the art**

In the Annual report from European Tire & Rubber Manufacturers Association (ETRMA), President Christian Kotz described the rubber and tire industry one of the main drivers of economic growth, innovation, and productivity, which are all vital for Europe's industrial competitiveness. Tires are not just about mobility - they are about keeping the economy moving forward and leading Europe's transition to a more competitive and sustainable future.

The ETRMA report reveals that the industry's efforts are headed in one direction: protecting the environment, by reducing CO2 emissions, while improving tire safety and maintaining the industry's competitiveness.

The progress in waste tire recycling with a particular attention to incorporation of waste tire rubber into polymeric matrices has been reviewed [5]. This study addressed the methods of waste tire downsizing, the importance of particle characterization and current practices of surface modification. The influence of particle size, loading, modification, compatibilization and crosslinking on the rheological, mechanical and thermal properties of rubber, concluding that recycled rubber blends can show ease of processing and acceptable properties.

The effect of recycled rubber powder on cure characteristics, tensile properties and swelling behavior of natural rubber targeted the concentration range of 0 to 50 PHR was studied [6] and concluded that the viscosity of the compounds would increase with increasing recycled content whereas the scorch time exhibited a decreasing trend.

The study [7], addressed the different grinding technologies for the production of rubber powder from end of life tires in industrial scale. The processing capacity, energy consumption, particle size distribution, and morphology were evaluated. Additionally, the impact of the grinding process on particle characteristics and the properties of the resulting vulcanizates was also examined. The article concluded the ambient ground particles have more structured surfaces, but they are very inhomogeneous in this respect and that There are also large quantities of barely structured particles.

On the other hand [8] provides an overview of the technological development of different recycling options and their potential benefit to the circular economy. It was focused on devulcanization processes using a biotechnological approach with microorganisms and/or enzymes to reuse waste rubber for rubber compounds developed for high value applications. Most recently the activation of ground tire rubber was studied [9] but focused on the microwave method for modifying rubber powder for its application in off-road tire tread formulation.

The relevant literature describing the current status of end-of-life options worldwide, was compiled [10] and the European legislation regarding tire waste, their possible uses and the related environmental aspects was presented, while the study [11] presented the literature describing innovative organizational approaches in the management of used tires in the European Union member countries and the possible uses of waste tires as a source of raw materials or alternative to fossil fuels.

## Method

This study was based on laboratory tests that targeted two specific particle sizes, content levels and two different production methods. The incorporated rubber powder was introduced with the fillers, on a two-stage production process. Several tests were performed in an independent and certified laboratory. The test results were compared with the ones from the control mixture, with no addition of rubber powder.

The mechanical performance of the compounds was obtained through tensile strength, elongation at break and tear resistance tests, as well as compression set tests.

The physical tests were performed to evaluate the compounds' processing properties: hardness, viscosity and rheometry. These tests were useful to characterize the processing behavior and understand how rubber compounds respond to different forces and deformations during its processing and use.

Table 1 presents the sample codes, description and particle size ranges for each test compound, where S1 is the control compound, without any recycled rubber content added but with the same base recipe as all other four test formulations, so it can be clearly evaluated the influence of the incorporation of different particle sizes and content levels.

Table 1 -Samples description and particle sizes

| **Sample/Code** | **Description** | **Particle size range (mm)** |
|---|---|---|
| S1 | Control – Reference mix | N.A. |
| S2 | 10 PHR Micronized Rubber Powder | 0 – 0,180 |
| S3 | 20 PHR Micronized Rubber Powder | 0 – 0,180 |
| S4 | 10 PHR Ambient Fine Powder | 0,200 – 0,600 |
| S5 | 20 PHR Ambient Fine Powder | 0,200 – 0,600 |

The increase in particle size of the powder introduced impacted the structure of the mixtures when compared to the compound with no rubber added. The level of content introduced also had significant influence on the overall properties of the test formulations

The ambient fine powder (AFP) is produced with the traditional recycling method, that relies on mechanical grinding at room temperature, resulting in a large particle size distribution, while the Micronized Rubber Powder (MRP) is obtained with a cryogenic process technology, that uses liquid nitrogen to freeze the rubber to a temperature so low that milling results in a micronized rubber powder, with smoother surfaces and maximum particle sizes that can be even smaller than the ones produced by the traditional ambient method. The selected size for these tests was 80 MESH, 0-180 µm, as this product is optimized for improved mechanical properties and described as suitable for specialized applications with demanding performance requirements.

## Case Study and Results analysis

The performance characteristics for the different mechanical property were tested. Firstly, the selected specification is based on a standard requirement for this type of rubber compound and was already applied in internal quality controls of the S1 compound. In order to better understand the influence of particle size and content on the properties of the material, all results were compared with the specification and with the results obtained from the reference test compound. Table 2 presents the measurements obtained for each property and test sample.

Table 2 – Performance results for the different mechanical property tests

| **Property** | **Spec** | **S1 (Control)** | **S2** | **S3** | **S4** | **S5** |
|---|---|---|---|---|---|---|
| Tensile Strength [MPa] | > 6 | 7,9 | 7,7 | 7,5 | 6,8 | 5,5 |
| Elongation [%] | > 350 | 450 | 455 | 440 | 380 | 375 |
| Hardness [Shore A] | 74 ± 5 | 74 | 73 | 73 | 73 | 72 |
| Tear Resistance [N/mm] | > 11 | 12,8 | 13,1 | 13,9 | 12,9 | 13,1 |
| Compression Set [%] | < 50 | 42 | 40 | 35 | 42 | 36 |

Despite the study [6] concluded that the viscosity of the compounds would increase with the increasing recycled content whereas the scorch time exhibited a decreasing trend, our findings showed that, for cryogenic powder, there was indeed an increase in Mooney viscosity and the scorch time decreased, but with the inclusion of the ambient rubber powder with bigger particle size, results showed the opposite trend: there was a viscosity reduction and an increase in scorch time.

Particle size plays a crucial role in the mechanical and physical properties of the rubber formulations. The table 3 addresses the five mechanical properties' tests by commenting on the obtained results for each and the main takeaways from the observed.

Table 3 – Effect of particle size on the different mechanical properties

| Property | Control (S1) | MRP (S2 & S3) | AFP (S4 & S5) | Comments |
|---|---|---|---|---|
| Tensile Strength (MPa) | 7,9 | 7.7 / 7.5 | 6.8 / 5.5 | Smaller particles (MRP) reinforce better, resulting in higher tensile strength. AFP worsened results, likely due to poorer dispersion and lower interactions with the rubber matrix. |
| Elongation (%) | 450 | 455 / 440 | 380 / 375 | Smaller particles allow greater elasticity, finer particle sizes improve flexibility due to increased interaction with the polymer network. |
| Hardness (Shore A) | 74 | 73 / 73 | 73 / 72 | Both MRP & AFP had no significant influence on the hardness results |
| Tear Resistance (N/mm) | 12,8 | 13.1 / 13.9 | 12.9 / 13.1 | Smaller particles can improve performance, while larger particles (AFP) maintained tear resistance levels |
| Compression Set (%) | 42 | 40 / 35 | 42 / 36 | MRP provides slightly better elasticity retention, with comp. set values indicating less permanent deformation. |

### Understanding the influence of particle size

Using MRP particles, which are smaller in size, leads to better reinforcement of the rubber compounds, in terms of tensile strength, elongation, and improved tear resistance. These properties make MRP particularly suitable for structural reinforcement applications, where mechanical strength and durability are critical.

On the other hand, incorporating AFP, consisting of larger ambient particles, maintained hardness, tear resistance, and compression set levels. These results showed that this type of powders should be appropriate for flexible applications, where maintaining form and resilience without compromising flexibility is essential.

However, at higher content levels (20 PHR), the use of larger particles (AFP), can weaken the compound's performance. The results showed a reduction in tensile strength and an increased permanent deformation, limiting the effectiveness of larger particles in rubber compounds for applications that require high mechanical performance under stress. Even at higher levels of recycled content, MRP showed improvements on the compressibility and tear resistance test results, while presenting a minimal impact on tensile properties, hardness and elongation.

### Influence of content level (10 vs. 20 PHR) on properties

Two content levels were evaluated, 10 PHR (S2, S4) and 20 PHR (S3, S5) and compared with the results obtained from the control compound, which had no recycled rubber powder added, revealing how increasing the recycled rubber content affects each property, depending on the particle.

Table 4 – Effect of content level on the different mechanical properties

| Property | Control (S1) | 10 PHR (S2 & S4) | 20 PHR (S3 & S5) | Comments |
|---|---|---|---|---|
| Tensile Strength (MPa) | 7,9 | 7.7 / 6.8 | 7.5 / 5.5 | For larger particles (AFP), higher content significantly weakens tensile strength's results, while with MRP the decrease is minor. |

| Property | Control (S1) | 10 PHR (S2 & S4) | 20 PHR (S3 & S5) | Comments |
|---|---|---|---|---|
| Elongation (%) | 450 | 455 / 380 | 440 / 375 | Higher filler content slightly decreases elongation, making the material more rigid. |
| Hardness (Shore A) | 74 | 73 / 73 | 73 / 72 | No impact from filler content on hardness. |
| Tear Resistance (N/mm) | 12,8 | 13.1 / 12.9 | 13.9 / 13.1 | Higher filler content slightly improves tear resistance for MRP but has minimal effect on SFP. |
| Compression Set (%) | 42 | 40 / 42 | 35 / 36 | At 20 PHR, compression set is lower (better elasticity retention) for MRP, but AFP sees little improvement. |

At lower recycled content, specifically at 10 PHR, the compounds exhibit higher tensile strength, improved elongation and resistance to tear.

In contrast, increasing the rubber powder content to 20 PHR brings both benefits and drawbacks. On the positive side, higher recycled content improves tear resistance and compression set. This is emphasized for smaller particle sizes, where the micronized rubber powder particles act as a reinforcement, enhancing these specific properties. However, the downside of higher levels of content is a reduction in tensile strength and elongation, although the impact on elongation at break is relatively minor.

Granulometry and content level not only deeply influence the mechanical and physical properties of the rubber formulations, but also on the rheometrical properties. These are characterized by curing curves, that track the evolution of flow and deformation behaviour of the mixture. Understanding the influence of adding recycled rubber powders into new compounds on the viscoelastic properties is essential for optimizing the vulcanization process and ensuring consistency on the final processes and products.

Particle size also plays a crucial role in performance at higher loadings. In this study, cryogenically micronized particles performed better with increased content compared to ambient fine powders which experienced a significant drop in mechanical performance at 20 PHR, which can be attributed to its less homogeneous and bigger particle size, making it more difficult to disperse evenly into the compounds and to bond effectively with the elastomeric matrix. Moreover, AFP's fluctuating particle morphology, characterized by a wider distribution of particle structures, further contributes to its inconsistent performance under higher recycled rubber content.

Table 5 – Processing performance test results.

| Property | S1 | S2 | S3 | S4 | S5 |
|---|---|---|---|---|---|
| Mooney Viscosity (MU) | 65,3 | 63,7 | 66,7 | 61,5 | 59,3 |
| Scorch Time T'5 (min) | 6,95 | 7,15 | 6,85 | 6,65 | 7,05 |
| T'10 (s) (3 min, 180 ºC) | 31,2 | 32,7 | 34,1 | 35,5 | 36,47 |
| T'90 (s) (3 min, 180 ºC) | 99,7 | 104,4 | 108,1 | 111,2 | 113,7 |

### Impact on processing performance

Evaluating the viscoelastic and rheometric profile is necessary for ensuring the final compound's stability and performance. MRP (S2 & 3) delivered optimal results, showing only a slight decrease in viscosity at 10 PHR and a small increase at 20 PHR of rubber powder added, compared to the control measurements, and well within the acceptable standard deviations. In contrast, using higher recycled rubber content with AFP can significantly reduce viscosity, which may compromise the material's uniformity and weaken its structural integrity.

For applications that prioritize fast curing and productivity, MRP at 10 PHR provides the best balance. It results in a minimal drop in viscosity, slightly extends scorch safety, and maintains a reasonable cure time, optimizing both processing efficiency and final material performance.

For maintaining the compound's viscosity and faster curing requirements, MRP at 10 and 20 PHR provided the best results. It offers a strong combination of optimal flow behavior and efficient processing times. However, higher recycled rubber content levels with larger particle sizes, such as AFP, should be avoided in applications where quick curing is essential, as they significantly delay both the processing and curing stages, reducing overall efficiency.

When it comes to extending the curing window, ambient fine rubber powder presents the longest scorch and cure times. This makes it particularly suitable for applications that require more time before crosslinking begins, such as the production of larger molded goods. The extended scorch time allows for greater flexibility in shaping and processing the material before it begins to set. For most applications these results present a disadvantage in terms of productivity and process control, but for some they are crucial to assure process stability and final product quality.

## Conclusions

There is a substantial body of research on recycling rubber from end-of-life tires, covering various methods, their progress, and limitations. Well-documented processes like reclaiming, devulcanization, and surface modification have been studied for applications such as rubberized concrete and rubber-modified asphalt. However, there is limited information on the impact of particle size and content levels of recycled rubber powders in new rubber compounds for applications like tires, conveyor belts and for various other industrial sectors.

By focusing on the impact on performance of reducing particle size, we were able to clarify that the cryogenic particles are more suitable for incorporating into new rubber compounds because they are more homogeneous and due to its particles morphology they will be more easily blended into the polymer matrix, thus reducing the impact on performance and even improving some specific properties of the compounds.

The study of different content levels on the overall properties allowed to conclude that for ambient fine powders with particles between 0,2 and 0,6mm there is a limit of incorporation that will start impacting significantly critical properties. Regarding cryogenically micronized rubber powders, with particles sizes below 0,180mm, increasing content led to an improvement in tear resistance and compression set performance results, while only slighting impacting the tensile and elongation properties of the final test compounds.

Incorporating recycled rubber powders derived from end-of-life tires (ELT) into new rubber compounds is a crucial step towards reducing the consumption of virgin raw materials, many of which are sourced from fossil fuels. This approach not only maintains performance standards but also opens up opportunities for various demanding applications. As industry requirements become increasingly stringent, the scientific characterization of the performance impact of recycled rubber powders from used tires is essential to promote their widespread use across different sectors.

Transforming tires into valuable new resources is key to closing the tire life cycle and achieving sustainable development.

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