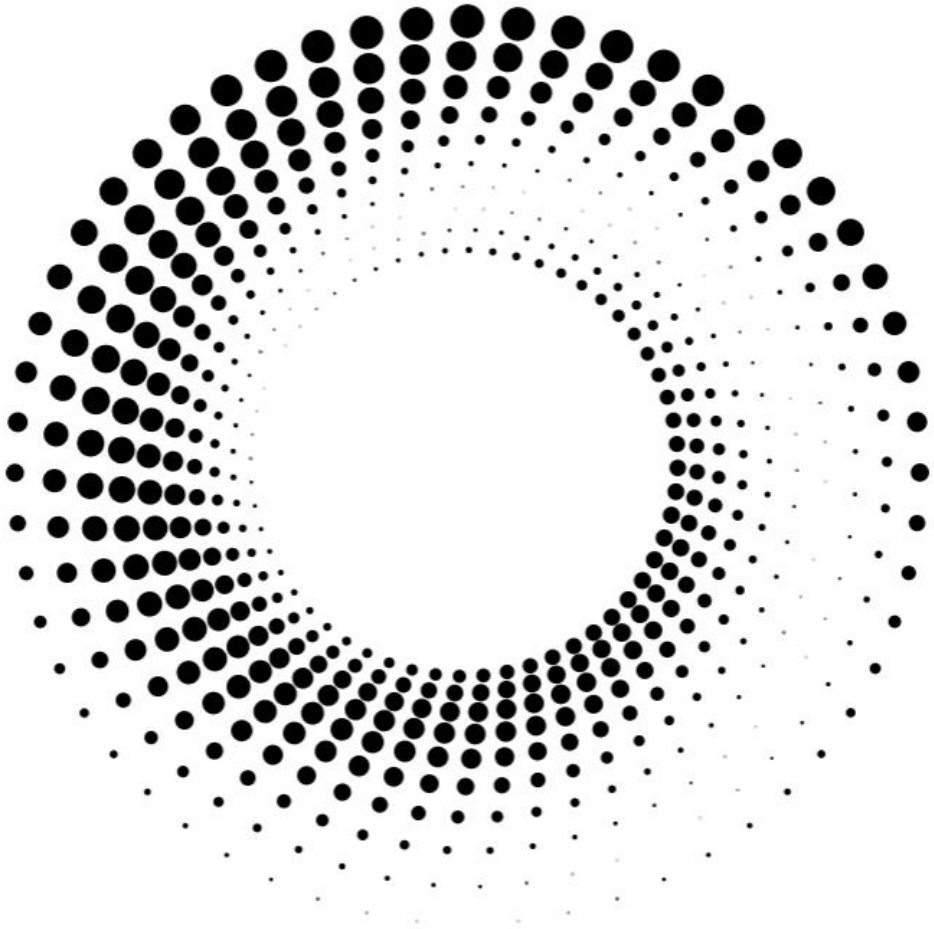

Bridgestone & Michelin

Recovered Carbon Black Guidelines

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Acronyms

AG: Agricultural Tire
ASTM: American Society for Testing and Materials
BET: Brunauer-Emmett-Teller (Specific Surface Area)
CB: Carbon Black
CEPA: Canadian Environmental Protection Act
CMR: Carcinogenic, Mutagenic, or Reprotoxic
COA: Certificate of Analysis
DSL: Domestic Substances List
EC: European Commission
ECHA: European Chemicals Agency
ELT: End-of-Life Tire
EoW: End-of-Waste
EPA: Environmental Protection Agency
HD: Highly Dispersible
HSE: Health, Safety, and Environment
LCA: Life Cycle Assessment
OAN: Oil Absorption Number
OTR: Off-the-Road Tire
PAH: Polycyclic Aromatic Hydrocarbons
PBT: Persistent, Bioaccumulative, and Toxic
PCR: Passenger Car Radial Tire
PMN: Pre-Manufacture Notice
PMT: Persistent, Mobile, Toxic
rCB: Recovered Carbon Black
REACH: Registration, Evaluation, Authorization and Restriction of Chemicals
SD: Standard Dispersible
SNUR: Significant New Use Rule
SSA: Specific Surface Area
STSA: Statistical Thickness Surface Area
TBD: To Be Determined
TBR: Truck and Bus Radial Tire
TGA: Thermo-Gravimetric Analysis
TSCA: Toxic Substances Control Act
vCB: Virgin Carbon Black
vPvB: Very Persistent and Very Bioaccumulative

Executive Summary

Bridgestone and Michelin collaborated with recovered carbon black (rCB) suppliers and other stakeholders in the value chain to define initial proposals for standards, including grades, specifications, and awareness of quality and performance requirements for the tire industry. The two companies have produced a set of definitions, proposed specifications, and guidelines for regulatory requirements, as well as a foundation for supporting the growth of the rCB industry. While the focus markets for this phase were Europe and North America, the principles of the work can be scaled globally. Bridgestone and Michelin plan to continue developing a point of view on factors impacting consistency, in-rubber properties, and environmental impact to inform standardization efforts in the rCB industry and will also continue to advance and scale sustainable carbon black technologies to achieve a shared vision of a sustainable supply chain in 2050. This will include rCB and other pathways, such as carbon black produced from renewable oils or tire pyrolysis oil. Bridgestone and Michelin acknowledge the feedback and input from many stakeholders who contributed to the development of this white paper.

I. Introduction

- **Bridgestone and Michelin delivered a joint presentation to share the results of their work surrounding recovered carbon black in the tire industry at the Smithers Recovered Carbon Black Conference on November 16, 2022.**
- **The presentation follows the position paper focused on their joint initiative to increase the use of recovered carbon black that was released in November 2022 in Berlin.**
  Michelin and Bridgestone have worked with different stakeholders around the world and across the tire and rubber value chain in the journey toward material circularity.

Both Bridgestone and Michelin have publicly shared their respective ambitions to make their products more sustainable and ultimately manufacture their products using 100% sustainable materials by 2050. This journey has already begun and both companies have identified various challenges to overcome. One of the shared observations is focused on pyrolysis as a solution to improve both the recycling markets for End-of-Life Tires (ELTs) and the supply of Sustainable Materials.

A core product of interest for the rubber industry that is obtained through the pyrolysis of ELT or other rubber goods containing carbon black, is recovered Carbon Black (rCB). Both companies have acknowledged a highly fragmented ELT pyrolysis marketplace, with a continuous inflow of new actors with new ELT pyrolysis technologies.

These technologies are often proven at prototype scale but remain unproven at industrial scale. Moreover, most new pyrolysis actors are relatively small start-up companies that often have limited experience in supplying large industrial companies like Bridgestone and Michelin. When discussing these challenges, both companies realize that the tire industry has not clearly articulated to rubber industry actors the capacity, demand, and technical requirements of recovered materials. This, together with limited guidelines that define and classify recovered materials, may be a key reason as to why there is no established rCB marketplace today. For large industrial companies the key condition that enables the adoption of a new raw material is the existence of sufficient market actors to supply materials in large quantities and ensure quality is consistent and up to industry standards. Active involvement of large consumers was a more effective way to accelerate the rCB industry's growth and development.

Therefore, the two companies decided to make a joint effort that was entitled, “A Call to Action.” It was proposed to collaborate actively with the recycling ecosystem, aiming to address the various challenges and find solutions that will boost the potential usage of recycled and recovered materials from ELTs in technical rubber products, including tires.

During this first year, the focus of the efforts has been on collaborating with key stakeholders of the ecosystem and proposed a first set rCB grade definitions and specifications. These proposals were presented in November 2022 at the Berlin Smithers conference.

The objective of this White Paper is to formalize the approach and share the first set of rCB grades and specifications but also to outline the remaining work and challenges for the coming years. It will be explained why the quality approach for rCB cannot be directly extrapolated from the historical quality approach for virgin Carbon Black (vCB).

II. Technical Information

A. Description of rCB

As illustrated by Martin Wolfersdorff below, rCB is a mixture of different products, some of them being a function of the ELT feedstock such as:

- The mixture of CB originally part of the ELT (soft grades (N7xx, N6XX, N5xx series) and tread or hard grades (N3xx, N2xx, N1xx series)
- The content of the inorganic ash, mainly Zn chemical compounds and silica.

The ratio of those different products is not only function of the original tire design, but also of the level of wear of the ELT. For example, pyrolysis of a batch of ELT consisting mainly of relatively new passenger car tires could result in an rCB with a high level of silica.

In addition to impact of the feedstock on the rCB composition, the process itself can trigger some differences, reflecting mainly the level of completion of the pyrolysis process (volatile content) and its impact on the “carbonaceous residues”.

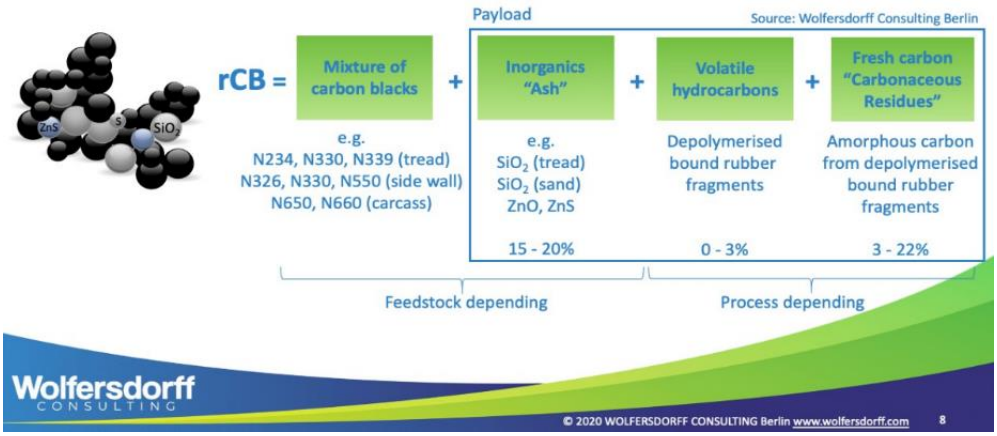


Figure 1. Description of rCB prepared by Wolfersdorff Consulting

In addition to the chemical composition of rCB, it is important to note that the solid product from the pyrolysis of ELT results in particles as large as several millimeters in size, some of which are not readily breakable. This results in the need for a grinding/milling post-treatment of the pyrolysis product to achieve particle sizes which facilitate good in-rubber dispersion. Other post-treatment processes of rCB, including leaching of ash, surface activation, and others, are currently under development. Due to the developmental stage of these post-treatments, they will not be further described in this document.

It is clear that rCB is not identical to virgin carbon black but is a different class of filler with some unique chemical characteristics and physical properties.

The key 'definition parameters' may be summarized as feedstock composition and stability, quality and consistency of the pyrolysis and post-treatment processes, including particle size reduction levels and/or potential post-treatment of the product

Recovered Carbon Black needs to be classified in types. ASTM committee D36 has been working on this topic, and a proposal titled WK84831 is in draft form and is under development within this ASTM Committee. This proposal will be based on test methods available in D36 or D24 to facilitate specifications between producers and customers.

The following rCB grades can be proposed as exemplified below. Potential differentiation is shown for grade A, which could be applied for other types of grades, including B, C, and others. The final classification will come from the ASTM. The letter grades are not intended as a ranking of expected quality or performance, but simply a naming system to differentiate the grades.

Table 1. Definitions of rCB grades

Definition		Grade A1.1	Grade A1.2	Grade A1.3	Grade B	Grade C	Grade D
Stable ELT feedstock		ELT from PCR, TBR, AG, OTR …			100% TBR	100% OTR	tbd
In rubber rCB dispersion	Laser diffraction (under development by ASTM / WK7 1858).	HD 1.8< D50 < 2.6 µm 7 <D99 < 10 µm	SD 2.5 <D50 <3.5 µm D99 : 20 +/- 2.5 µm	HD 1.8< D50 < 2.6 µm 7 <D99 < 10 µm	HD 1.8< D50 < 2.6 µm 7 <D99 < 10 µm	HD 1.8< D50 < 2.6 µm 7 <D99 < 10 µm	tbd
Post-treatment (e.g. activation, ash leaching …) &/or other special technology		No	No	Yes	No	No	tbd
Ash content	ASTM D1506	>15% / <20% *	>15% / <20% *	<20% **	< 17%	< 17%	tbd

* : Ash content target only if a REACH compliance is achieved through a REACH exemption (see REACH chapter)

** : Ash content target will depend on the post treatment

B. rCB Specifications

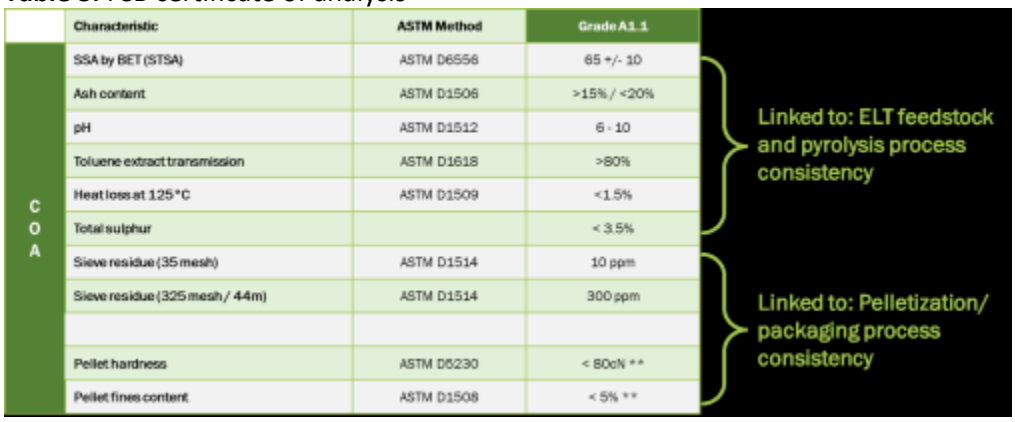
Significant amount of work has been done and is still underway within the ASTM Committee D36. At this stage it is clear that the needed characterization methods for rCB are not yet available. Taking this under consideration and trying to fulfill the need to assess the quality of the rCB at reception, table 2 below summarizes a proposal for the specification.

Table 2. rCB specifications

Definition		Grade A1.1	Grade A1.2	Grade A1.3	Grade B	Grade C	Grade D
ELT		ELT from PCR, TBR, AG, OTR …			100% TBR	100% OTR	?
SSA by BET (STSA)	ASTM D6556	65 +/- 10	65 +/- 10	to be defined based on the nature and impact of the physicochemical treatment.	tbd	tbd	tbd
Ash content	ASTM D1506	>15% / <20%	>15% / <20%		< 17%	< 17%	tbd
pH	ASTM D1512	6 - 10	6 - 10		6 - 10	6 - 10	tbd
Toluene extract transmission	ASTM D1618	>80%	>80%		>80%	>80%	tbd
Heat loss at 125°C	ASTM D1509	<1.5%	<1.5%		<1.5%	<1.5%	tbd
Total sulphur		< 3.5%	< 3.5%		< 3.5% (tbc)	< 3.5% (tbc)	tbd
Sieve residue (35 mesh)	ASTM D1514	10 ppm	10 ppm	10 ppm	10 ppm	10 ppm	tbd
Sieve residue (325 mesh / 44µ)	ASTM D1514	300 ppm	700 ppm	300 ppm	300 ppm	300 ppm	tbd
Pellet hardness	ASTM D5230	< 80cN **	< 80cN **	< 80cN **	< 80cN **	< 80cN **	tbd
Pellet fines content	ASTM D1508	< 5% **	< 5% **	< 5% **	< 5% **	< 5% **	tbd

The objective of the specification proposal is to ensure the consistency of the rCB produced by the suppliers to facilitate industrial performance at the customers' factories, thereby resulting in uniform in-rubber properties. Additionally, the characteristics identified in the Certificate of Analysis (CoA) will enable the detection of deviation of the material properties by the receiving customers.

Table 3. rCB certificate of analysis



**: Pellet hardness and fines content are very specific of the final user equipment and processes.

Some evolutions or needs for new characterization methods have been already identified or initiated. They are summarized below:

C. Stability of rCB

+ **Ash%** evolution of the method: ASTM TGA new method approved by ASTM D36 but not yet fully deployed, so specification needs to be defined in the future based on the deployment and usage of this method by the different parties.
+ **Organic Volatile content:** ASTM TGA Method approved by the ASTM D36 committee. The deployment of this new TGA method will allow the definition of specifications and how to use them.
+ **Toluene discoloration specifications**: evolution of the minimum tolerance based on future studies?
+ **In Rubber properties:**
    - **Particle size distribution both before and after** pelletization (especially for HD grades): Method under development by ASTM subcommittee.
    - **Modified OAN:** ASTM confirmation of the interest, definition of specifications.
    - **Evaluation of the polymer-filler interactions:** Standard Test Method for Recovered Carbon Black—Rheological Non-Linearity of a Rubber Compound by Fourier Transform Rheology ASTM D8491.

D. LCA of rCB Compared with Virgin Carbon Black (vCB)

Various industry players have conducted LCAs on rCB production. Bridgestone and Michelin have not conducted a detailed analysis of each study to make a robust comparison of the underlying assumptions, but it is encouraging that the available results indicate a significantly better cradle-to-gate CO_2 footprint compared to vCB.

III. Intellectual Property Protection and Licensing

The content of this white paper is for information purposes only and as a proposal, is subject to change as information evolves.

It should be understood that the specific composition of the proposed grades of rCB and/or methods of production may be patented. To ensure a consistent supply of quality rCB products, it may be useful for suppliers to be able to secure any licenses needed from the patent holders.

IV. Regulations

All businesses must adhere to certain laws and regulations as part of its operations. Agencies involved in regulating materials such as rCB may include, but are not limited to, the Environmental Protection agency (EPA) with the Toxic Substances Control Act (TSCA), the European Chemicals Agency (ECHA) with the Registration, Evaluation and Authorization of Chemicals regulation (REACH), the European End of Waste regulation for recycled materials, and other relevant regulations, globally.

It is the rubber industry's aim to ensure that rCB complies to all current environmental and human health regulations, globally. To this end, and given the scope of this white paper, a particular focus is put on TSCA, REACH and waste regulations.

A. TSCA

The Toxic Substances Control Act of 1976 (TSCA) provides the U.S. Environmental Protection Agency (EPA) with authority to regulate chemicals processed, distributed, used, produced, and/or imported into the United States. TSCA generally provides EPA with authority to limit production of, limit the uses of, or completely outlaw any chemical substance that is deemed to be unsafe.

With some limited exceptions, chemical substances must be listed on the TSCA Inventory in order to be manufactured in or imported into the U.S. As a result, before processing, distributing, using, producing, and/or importing any chemical substance in the U.S., companies should first determine whether the chemical substance is listed on and/or covered by the TSCA Inventory. If a chemical substance is being newly introduced into U.S. commerce, it is not included in the TSCA Inventory and is not otherwise exempt from TSCA Inventory listing, then EPA must be notified, through a pre-manufacture notice (PMN), 90 days before the manufacture or import of the chemical substance occurs. Furthermore, EPA, at its discretion, may require that additional testing and studies be performed on new chemical substances.

As it relates to rCB, companies should review the current listings with care and may wish to submit an inquiry to EPA—to confirm that the form of rCB they intend to process, manufacture, import, and/or use, is included on the TSCA Inventory or may be considered as a mixture under the definition of "mixture" given in 40 CFR Part 710. Additionally, 40 CFR Part 710.4(d)(1) states that impurities are excluded from the TSCA Inventory. To the extent a company intends to manufacture, use, import, or process a different form of rCB, the company may be required to submit a PMN before doing so.

TSCA imposes reporting requirements, record-keeping requirements, and restrictions, on those who process, distribute, use, produce, manufacture, and/or import a chemical substance, including those chemical substances in mixtures and articles. Generally, manufacturers and importers are subject to more stringent obligations under TSCA than other entities that merely use or process chemicals that are manufactured or imported by others. As a result, it is important to carefully evaluate how a company's activities (such as activities involved in recovering rCB) fit within the relevant definitions under TSCA in order to understand which requirements apply.
Companies should also verify whether any significant new use rules (SNURs) govern the import, manufacture, processing, and/or use of their chemical substances. SNURs may impose additional notification requirements or other obligations on manufacturers, importers, and processors of the chemical. See Congressional Research Service, Title I of the Toxic Substances Control Act (TSCA): A Summary of the Statute, at pg. 8 (July 20, 2021), available at https://sgp.fas.org/crs/misc/R45149.pdf. For example, a SNUR has been issued related to the TSCA Inventory listing for "carbon black derived from the pyrolysis of rubber tire shreds (generic)," discussed above. As such, understanding whether companies' activities fall within the SNUR associated with this form of rCB will be critical to TSCA compliance.

Note, the information contained herein is not an exhaustive discussion of obligations and requirements under TSCA. In addition to the general requirements outlined above, TSCA imposes a variety of additional requirements on processors, importers, and exporters dealing with chemical substances that are subject to TSCA. A summary of TSCA, the current TSCA Inventory, TSCA exclusions and exemptions, the PMN process, and contact information for EPA can be found at https://www.epa.gov/tsca-inventory.

B. DSL

Canada has a Domestic Substances List (DSL), administered through the Canadian Environmental Protection Act, 1999 (CEPA), which is a listing of over 28,000 substances which are manufactured or imported into Canada on a commercial scale. Additional investigation by rCB producers is required to determine if rCB may be regarded as a substance or a mixture in regard to CEPA requirements.

C. REACH

EC REACH (Regulation (EC) No 1907/2006 on Registration, Evaluation, Authorization, and restriction of usage of Chemicals) is in force since June 2007 in Europe. Among other provisions REACH requires that all chemical substances on their own or in mixtures manufactured or imported into the EC at 1 ton or more per year are registered. It applies to EC manufacturers and importers of chemical from non-EC manufacturers and EC Only Representatives of non-EC manufacturers.

The compliance of rCB with Reach registration requirements can be achieved by a full registration process. Producers such as CIRCTEC have initiated this process. Under this CIRCTEC's registration process, ECHA has designated rCB as "**Amorphous carbon and silicon dioxide recovered from processing of spent tires**". As shared by Robert Harper, CIRCTEC's CEO during the Smither's rCB conference in November 2022, other companies are welcome to join this registration, or they can initiate their own registration.

For recovery operations realized in EC, an exemption of REACH registration can be invoked as provided by article 2.7(d) of REACH. This exemption is applicable only under the conditions that

- The substance resulting from the recovery process is the same as an already registered substance,
- The safety information relating to the substance that has been registered is available to the establishment undertaking the recovery

The article 2.7(d) exemption is interesting in the sense that the demonstration of sameness with pre-registered substances for the rCB either as a mono-constituent substance or as a mixture would allow savings of both tests, including possibly on animals and costs. The only challenge under this approach is that the chemical analysis study to the demonstration of the sameness could be demanding.
The main advantage of a full registration approach as initiated by CIRCTEC is that it provides the opportunity for non-EC manufacturers to join the registration process, of course there is also the possibility to start a new registration process.
The possible registration strategies available to both EC manufacturers and non-EC Manufacturers are summarized in the table below.

Table 4. Summary of possible registration strategies for EC and non-EC manufacturers

Manufacturing site	Article 2.7(d) Registration exemption	Full registration
EC Countries	**Yes**	**Yes**
Non-EC Countries	**No**	**Yes**

V. Environmental, Health, and Safety

As the recovering of Carbon Black from end-of-life tires is a significant opportunity for the development of circular economy within the tire industry, the knowledge and management of the potential hazards for health, safety and environment of recycled Carbon Black are key for safe and sustainable use in the tire industry.

Recovery operators, as manufacturers of rCBs, and tire manufacturers, as downstream users of rCBs, should ensure that the risks to Health, Safety, or the Environment (HSE) are assessed as required by applicable laws. To the extent required by applicable laws, exposure assessments of humans and the environment to any harmful substances should take into account all steps of the life cycle of the rCB, from manufacturing, through the use in tires and the tires usage, up to the waste and possible recycling.

rCB from end-of-life tire pyrolysis may contain up to approximately 20% ash. This ash is mostly composed of amorphous synthetic silica and zinc sulphide. The composition of this ash should be identified and closely monitored by the rCB producers to enable safe use by the tire manufacturers.

Manufacturers of rCB should collect analytical data to demonstrate that they have sufficiently identified and characterized their rCB in order to ensure compliance with relevant regulations, provide an appropriate Safety Data Sheet, and provide all information needed to enable tire manufacturers to employ appropriate risk management measures. Particular attention should be paid to the possible presence of substances that are potentially hazardous to human health or the environment, including:

- Polycyclic aromatic hydrocarbons (PAHs)
- Heavy metals
- Substances known to be carcinogenic, mutagenic or toxic for reproduction (CMR)
- Substances known to be endocrine disruptors
- Substances known to be toxic for aquatic organisms (PBT, vPvB, PMT)
- Substances known to have an acute or chronic toxicity

Manufacturers of rCB should strive to eliminate these potential substances from the composition of the rCB and in any case should maintain them below the applicable regulatory thresholds.

rCB manufacturers should also engage in research developments and advancements in control measures for the elimination of the substances of concern, for example via quality control of the feedstock or via the process settings or evolutions.

VI. Waste-to-Product status

Within the EC regulatory framework materials resulting from recycling processes realized in EC are deemed to be “waste” until it can be demonstrated that they meet pre-defined End of Waste (EoW) criteria. In this regard it is important to understand that compliance to REACH or to any “product” regulation alone does not affect the “waste” legal status of a recycled material. EoW status is a pre-requisite to bring the material in the scope of REACH or any “product” regulation.

EC-harmonized EoW criteria are in place only for a limited number of waste streams (iron, steel, aluminum and coper scraps and glass cullet), which do not include ELT derived materials. For the other waste streams the definition of EoW criteria lies on national decisions with possible divergence between member states and the corresponding uncertainties as there is no regulatory obligation of mutual recognition between member states on national EoW decisions.
Given both the administrative and financial burdens related to waste management, EC harmonized **End of Waste criteria for End of Life Tire derived materials are key for rCB market development in Europe.**

Such regulation would enhance the reuse and recycling of material derived from ELT into new tires and in general rubber industry, and promote new alternative key raw materials for tire and rubber production. End of Waste criteria are crucial for the confidence in the quality and safety of recycled materials and their uptake on the market[1].

Therefore, it is a strong contribution to the overarching objectives set in The European Green Deal to speed up the transition towards a circular economy and achieve climate neutrality by 2050.

In addition, recognizing chemical recycling or advanced recycling as a clear recycling operation based on quality output material would ensure certainty for investments in research and development in recycling technologies and business cases. Pyrolysis is a recycling operation by which waste materials (ELT) are reprocessed into chemical products (rCB, tire derived pyrolysis oil). It is important that pyrolysis processes, that can meet the criteria for the output materials flows and deemed recycling, are categorized as a recycling operation.

In Europe, pyrolysis plants are generally defined as “incineration or combustion units”, whereas in the US, the US EPA (Environmental Protection Agency) proposes technical clarification on pyrolysis units and states that pyrolysis is not a combustion process, and thus pyrolysis units should not be regulated under the waste incineration rules.[2] In the US, 14 states already set important precedents enacting laws that legally define advanced recycling as a manufacturing process. (same source)

VII. Conclusion

At the outset, Bridgestone and Michelin sought to:

- Define an initial proposal for rCB standards, including definition of grades and associated specifications, in collaboration with rCB suppliers and other stakeholders in the ecosystem
- Raise awareness and give guidance to emerging rCB suppliers on the quality and performance requirements for the tire industry
- Create a common language and understanding between producers and consumers of rCB

After a year of collaboration and many feedback loops with stakeholders, the companies have produced:

- A set of definitions of rCB product grades
- A proposed set of specifications

[1] From Weibold / Lamer presentation at the rCB conference – Ref to: CEN-CLC BTWG 11, 2018, cited in ECHA 2021
[2] From Weibold / Lamer presentation at the rCB conference

- A list of test methods which still need to be developed
- A set of guidelines for awareness of the regulatory requirements in Europe and North America
- A summary of the importance of achieving “End of Waste” criteria in Europe

This work has laid a foundation to help support the growth of the rCB industry, but there is still work to be done to set a course toward a shared vision of 100% sustainable materials in 2050. By 2030, with continued improvement in quality and performance, Bridgestone and Michelin expect that conditions will exist for the rCB market to reach up to 1 M tons (based on market assumptions and 3rd party expert input). To support this growth, in 2023 and 2024, the companies encourage the industry to develop a point of view on factors impacting:

- Consistency
    - Ash % (evolution of TGA method defined by ASTM and specification)
    - Toluene Discoloration (specification)
    - Organic Volatile Content (based on ASTM-confirmed definition and specifications)
- In-Rubber Properties
    - Dispersion (Particle size distribution, and specification of pelletization methods)
    - Modified OAN (based on ASTM-confirmed definition and specifications)
- Environmental Impact
    - LCA compared with traditional carbon black, based on a clear set of assumptions and in accordance with relevant standards

This aligned point of view will be a starting point toward standardization in the rCB industry, informing and complimenting the ongoing work within ASTM.

Bridgestone and Michelin will continue work to advance and scale sustainable carbon black technologies to meet the future needs of the industry and remove hurdles for their development. This includes further advancement of rCB, but also other pathways such as carbon black produced from renewable oils, or from tire pyrolysis oil. A combination of these technologies are likely to be applied together in order to achieve the common vision of a sustainable supply chain in 2050.

It is only the beginning of the journey, and Bridgestone and Michelin continue to explore ways to upgrade and expand the proposal in the coming months.

VIII. Acknowledgements

Bridgestone and Michelin would like to recognize the feedback and input from many stakeholders who have contributed to the process of developing this white paper.