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Industry Update on ASTM International Committee D36 on Recovered Carbon Black (rCB)

Denise Kennedy, DK Enterprises

- Sub Committee Chair ASTM Committee D36.40 on Environment, Safety and Sustainability
- Task Chair ASTM D11.27.01 on Rubber Recycling

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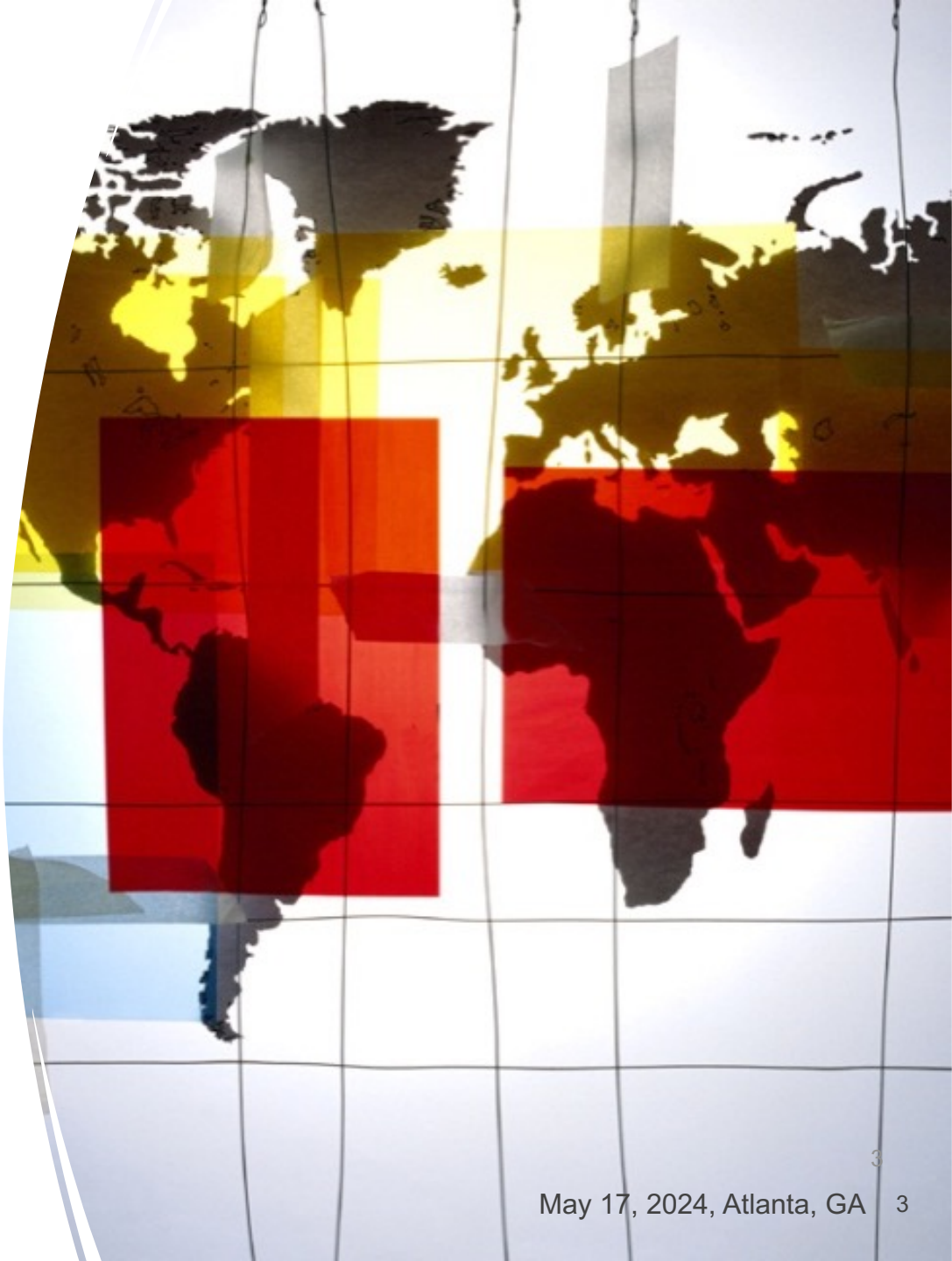
Overview

- ❑ What is ASTM International?
- ❑ ASTM D36 on Recovered Carbon Black
- ❑ Updates and Achievements
  - Two New Technical Standards
  - Pyrolysis Oil Sub Committee
  - rCB Shelf Life Balloted
  - Life Cycle Analysis Guideline Document
  - rCB Classification System

What is ASTM International?

- Established in 1898
- 150 Committees & 12,700+ Standards
- Over 30,000 members
- 8,000+ International Members from 149 countries
- 7,100 ASTM standards used in 80 countries
- Accreditation: American National Standards Institute (ANSI)



ASTM Industry Driven

Process complies with World Trade Organization (WTO) principles. Annex 4 of WTO/Trade Barriers to Trade (TBT) Agreement

Market Relevant Globally

Recovered Carbon Black Committee D36

Represented by
- Associations
- Academia
- Laboratories
- Producers
- Users

- Committee formed in 2017; 75 members (10 European Countries represented)
- Scope:
  - The promotion of knowledge of the properties of recovered carbon black (rCB), including stimulation of research and the development of test methods, specifications, classifications, and nomenclature.
  - This scope includes, but is not limited to, materials recovered by a decomposition process of end-of-life tires and other end of life rubber components.

ASTM D36 Recovered Carbon Black (rCB) Subcommittees

- D36.10 Recovered Carbon Black
- D36.20 Tire Pyrolysis Oils and Other Rubber Pyrolysis Co-Products
- D36.30 Nomenclature
- D36.40 Environmental Safety and Sustainability
- D36.70 rCB in Rubber Testing

ASTM D36 Roadmap Toward New Standards

Correlating test methods and product understanding, provides producer guarantee product consistency during the production process

- **Meaningful test methods and standards**
- **Validated test methods**
- **Categorization of rCB grades**

What is rCB?

Recovered Carbon Black (rCB) is not a grade of carbon black!

Recovered Carbon Black is more than the recovery of a carbon black grade.

rCB =

Spectrum of rCB Grades

Mix of all grades used in passenger tires

Inorganic Contaminations

70 – 90% C Content

- Silica
- Zinc (S/O)
- Sulfur
- Trace elements

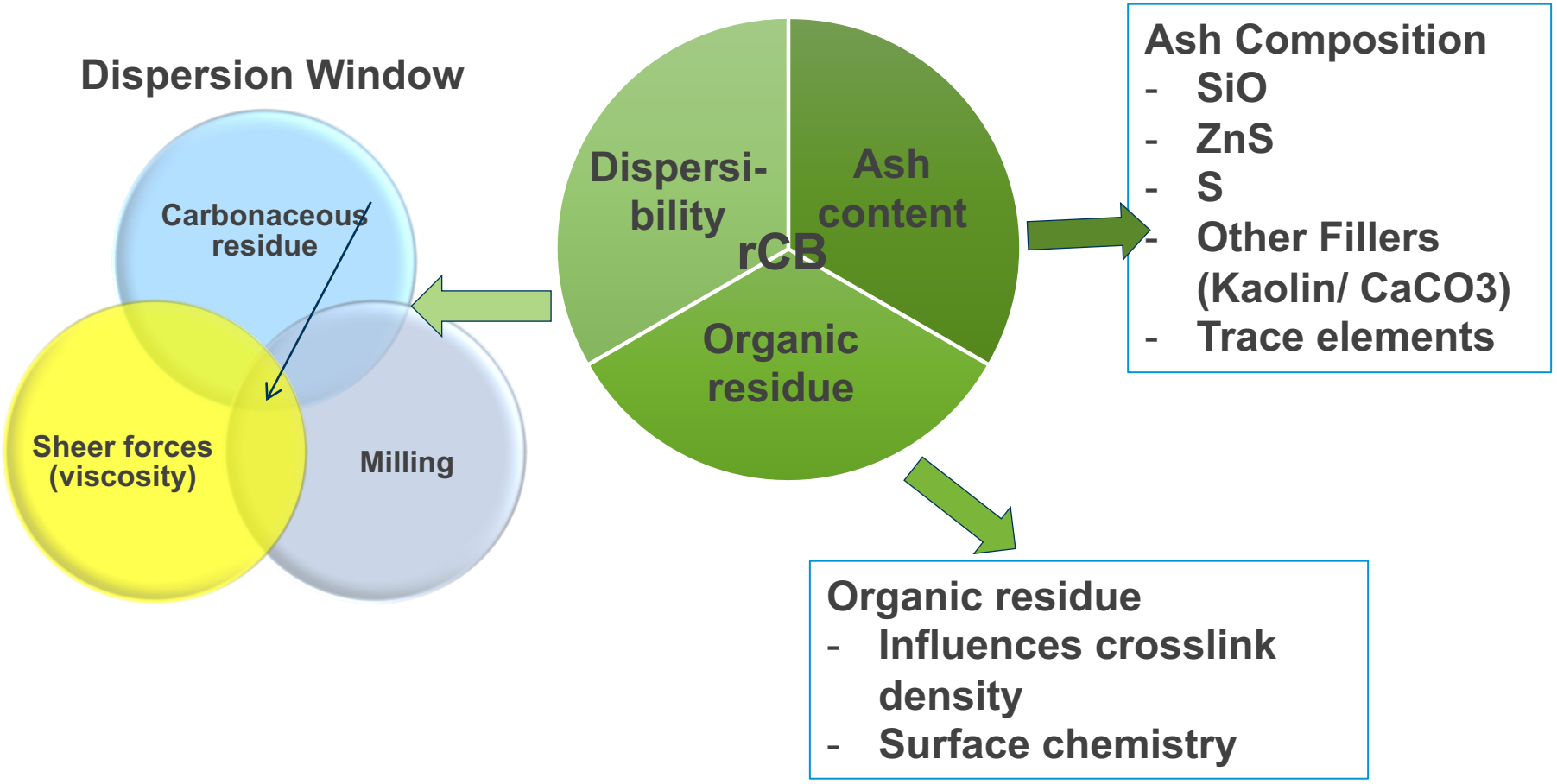
Chemical Effects (Thermal/Coating)

Carbonaceous Residue (Coke):

- Formation of amorphous coke layer
- Formation of organic layer from depolymerized (bound) rubber

Key Product Parameters

D36.10 Development



ASTM D8474 Compositional Analysis by Thermogravimetry (TGA)

D36.10 Recovered Carbon Black

- Bulk composition fingerprint
- Carbon content
- Organic residue
- Ash Content

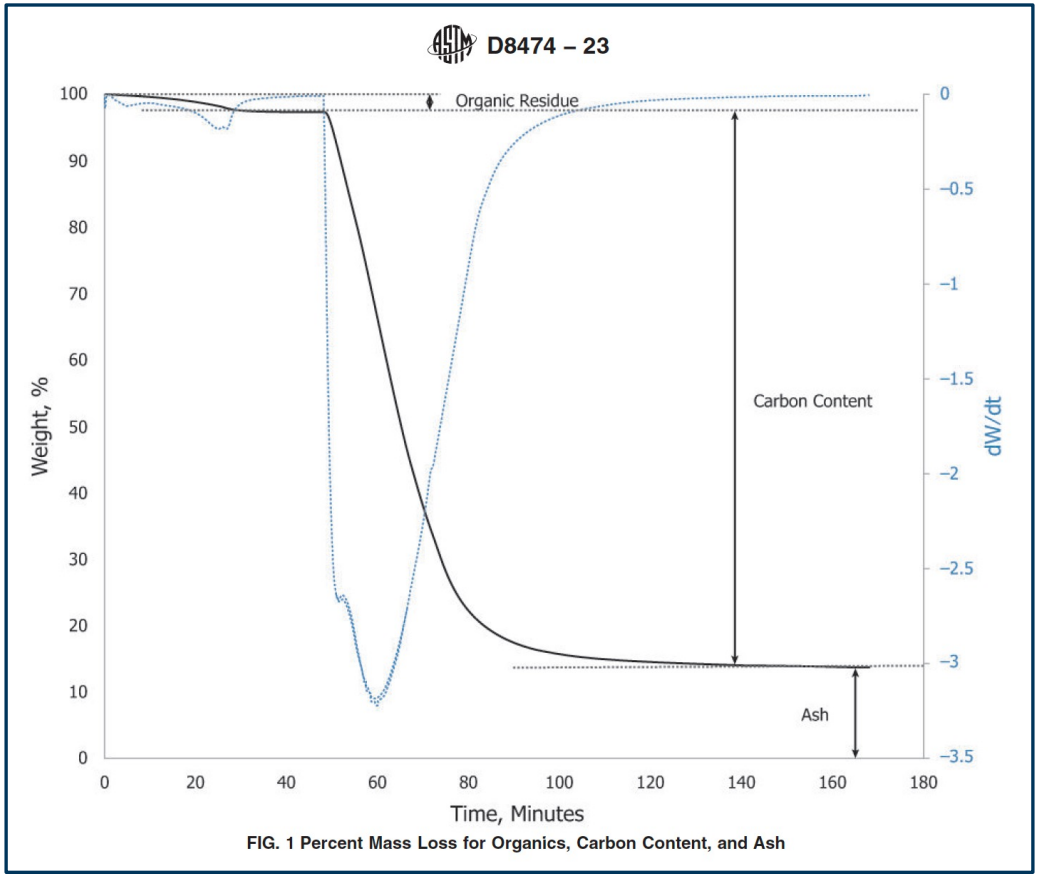


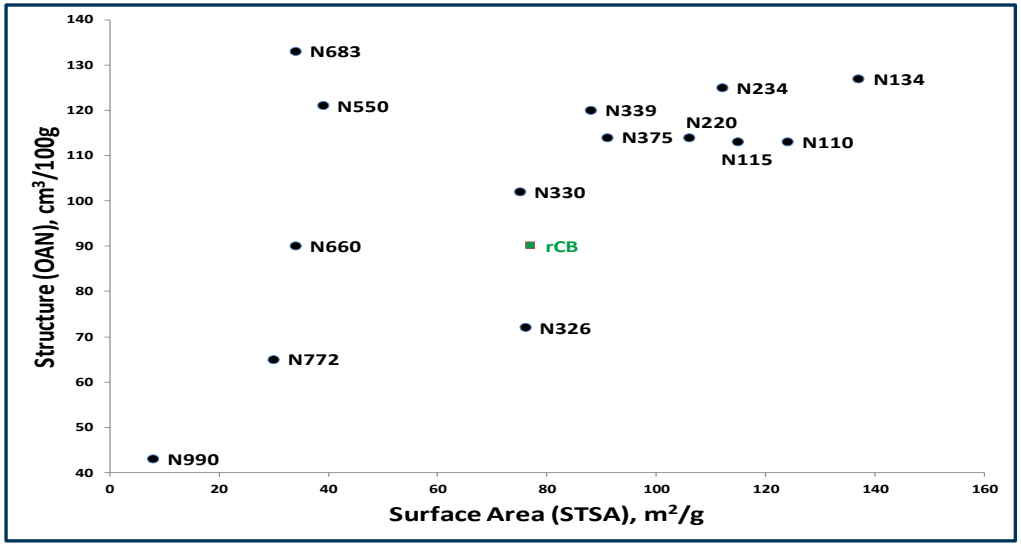
FIG. 1 Percent Mass Loss for Organics, Carbon Content, and Ash

ASTM D8466 Carbon Black Test Methods for Testing rCB

D36.10 Recovered Carbon Black

Total 23 Carbon Black D24 Standards Adopted

- Properties (Ash, Pellet, etc) 14
- Sampling and Preparation 2
- Reference materials and statistics 4
- Reference compounds 2
- Terminology 1



3.1 Committee D36 has concluded that typical carbon black characterization methods based on **structure level** and **surface area** measurements **do not** seem to **correlate to in-rubber performance** in an equal manner for Recovered Carbon Black products.

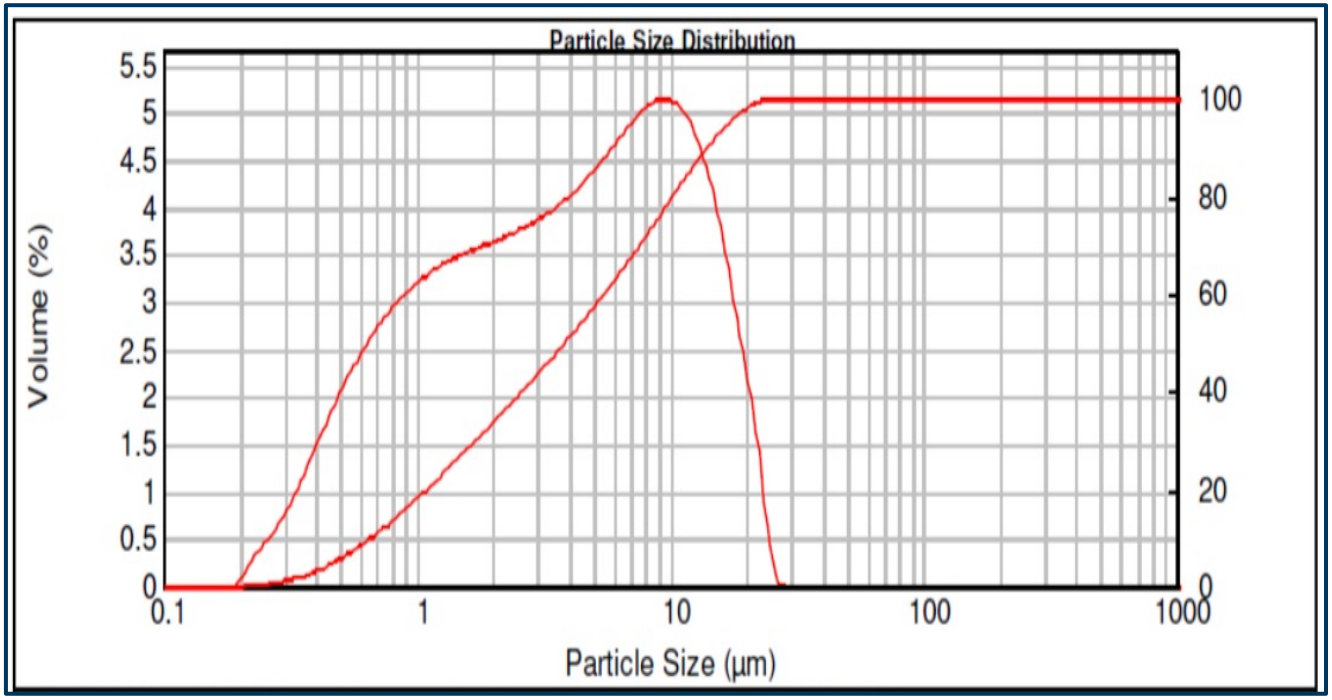
Therefore, the committee does not recommend such test methods for predicting the applications performance of recovered carbon black products. The committee is open to new research that can contradict above statement.

Standard Test Method for Dry Powder rCB Particle Size Analysis via Laser Diffraction WK87480

D36.10 Recovered Carbon Black

- Dry laser diffraction
- Proof of concept stage with multiple dry laser diffraction manufacturers with 3 reference samples.

https://www.dksh.com/global-en/products/ins/malvernpanalytical-mastersizer-range



Other activities

D36.10 Recovered Carbon Black

- Ash Composition method (XRF?)
  - Discussions and interest to erect a task group dedicated to developing a rCB specific standard to analyse Ash compostion. This might become a XRF or comparable based test method.

- Ash content method rCB
  - Due to inherent differences in Ash content, and origin between rCB and CB, a new rCB specific standard is currently being developed. One of the main objectives is to reduce the analysis duration

- TGA method refinement WK85957
  - Task group to further develop the TGA D8474 Standard.`

D36.20 Tire Pyrolysis Oils and Other Rubber Pyrolysis Co-Products

ASTM D02 on Petroleum Products, Liquid Fuels, and Lubricants, method validation for Tire Pyrolysis Oil (TPO)

- Task group working on initial validation of set of D02 test methods, Flash Point, Total Acid Number (TAN), etc
- Liason between D36.20 and the D02 committee

https://www.zimmermann.chemie.uni-rostock.de/en/research/fuels-materials/crude-oil/

D36.30 Nomenclature

char, *n*—solid carbonaceous residue formed during carbonization of organic compounds.

raw rCB, *n*—solid material resulting from thermal decomposition of rubber goods which contain carbon black; exhibiting poor dispersion so requiring milling to become rCB.

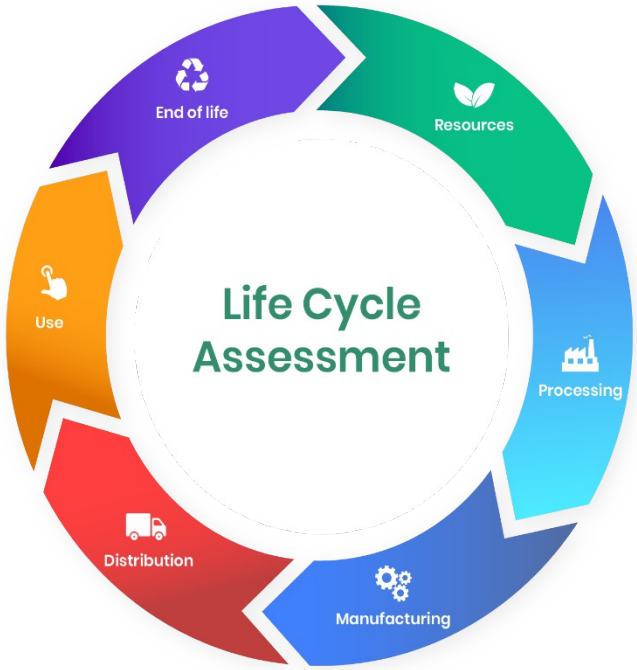
recovered carbon black (rCB), *n*—solid product recovered via thermal decomposition from rubber goods which contain carbon black, which is free of wire and fabric, and when milled typically gives semi-reinforcing properties in rubber.

Discussion—A type of filler derived from post-consumer rubber goods (feedstock) via a variety of thermal decomposition processes. This semi-reinforcing filler predominantly consisting of carbon, also containing inorganic compounding ingredients originating from the feedstock but is free of wire and fabric. This material, typically pelletized currently marketed under the name recovered carbon black (rCB), should not be confused with "Raw rCB" which exhibits poor dispersion and minimal reinforcing properties when used in rubber compounding.

Developing Standards

D36.40 Environment, Safety and Sustainability

- rCB Shelf Life Balloted
- Life Cycle Analysis Guidelines (LCA)
  - ISO has some key definitions and we do not want to duplicate.
  - Addressing first, Product Category Rules (PCR) to ensure consistency in the preparation of the LCA guidelines.
- Defining standard methods for characterizing the properties of pyrolysis oil
  - Chemical composition, physical properties, stability, and impurities
  - Standardized characterization techniques to enable effective quality control and facilitate comparisons across different sources and production processes.
- Peer review process



https://www.oneclicklca.com/life-cycle-assessment-explained/

ASTM D8491 Rheological Non-Linearity of a Rubber Compound by Fourier Transform Rheology

D36.70 rCB Testing in Rubber

- First rCB specific in-rubber test
- This test method provides a measure of rheological non-linearity of a rubber compound filled with rCB to assess its reinforcement capabilities.
- This test method requires the use of a sealed cavity rotorless oscillating shear rheometer for the measurement of the torque with increasing sinusoidal strain applied to an uncured rubber compound containing significant amounts of colloidal fillers, such as recovered carbon black, alone or as blend with virgin carbon black

https://www.alpha-technologies.com/instruments/premier-rpa-rubber-process-analyzer/

Proposal for rCB Classifications

D36.70 rCB Testing in Rubber

Particle Size, D97 [µm]	<7.5	>7.5	>12.5	>17.5	>22.5
WK71958	**1**	**2**	**3**	**4**	**5**
Toluene Transmittance [%]	<60	>60	>80	>95	
D1618	**1**	**2**	**3**	**4**	
Ash Content [%]	<12	12 to 17	17 to 25	>25	
D8474 or D1506	**1**	**2**	**3**	**4**	
In-Rubber Surface Activity [-]	< N660	N660 to N550	>N550		
D8491	**7**	**6**	**5**		

One example: **P21T82A22-660** → **P4T3A3S6** → **R4336**

The Last Word in Standards

- Four D36 rCB specific Standards in place
- Dry laser diffraction particle size distribution method under development
- rCB Classification under development
- rCB Shelf Life balloted
- Life Cycle Analysis (LCA) Guidance under development

Thank you

Denise Kennedy, President
DK Enterprises

- **Sub Committee Chair ASTM D36.40 on Environment, Safety & Sustainability**
- **Task Chair ASTM D11.27.01 on Rubber Recycling**

denise@dkenterprises.us
(916) 804-0931

Pieter ter Haar
Chairman ASTM Committee D36 Recovered Carbon Black
Director Sustainable Carbonaceous Materials, CIRCTEC
p.terhaar@circtec.com