CB4REACH Consortium

Carbon Black Information Letter 6

Characterisation of nanoforms of carbon black

As per the ECHA Guidance document, carbon black grades are characterized through the following methods of identification:

1.1 Composition

- Main chemical elements C, S, O, N and H
  Carbon (as per test standard ASTM D7633)
  Sulfur (as per test standard ASTM D1619)
  Oxygen (complementing CHNS to 100%)
  Nitrogen (through CHN analyser)
  Hydrogen (through CHN analyser)

- Heavy Metals Sb, As, Ba, Cd, Cr, Cu, Pb, Hg, Ni, Se, Sn and Zn
  No standard specific to carbon black exists; however, various internationally recognized test standards are available for the determination of various individual heavy metals in materials like carbon black.
  One consists in combusting the carbon black in a muffle furnace at 550 °C as per ASTM D1506 prior to digesting the remaining ash with hot concentrated acids. The individual heavy metals are then quantified in the digested ash solution using an appropriate analytical technique (e.g. ICP-AES, ICP-OES, ICP-MS). A suitable example is ISO 17294 Part 1 and 2 (ICP-MS): Application of inductively coupled plasma mass spectrometry. Part 1: General guidelines (ISO 17294-1:2004); Part 2: Determination of 62 elements (ISO 17294-2:2003).
  Another consists in digesting entirely the carbon black with nitric acid in a closed quartz reaction vessel under a pressure of approximately 100 bar at 250 °C. Such technique minimizes the potential loss of some volatile metal oxides for Hg or As. The individual heavy metals are then quantified in the digested solution using an appropriate analytical technique (ICP-AES, ICP-OES, ICP-MS, etc.).

- Polycyclic Aromatic Hydrocarbons
  Sum of the EU-8 PAHs (as per test standard ASTM D8143).

1.2 Particle size distribution and number fraction of constituent particles

The morphology of carbon black can be characterized using Transmission Electron Microscopy (TEM) as per the ASTM test standard D3849.

Aggregate Size Distribution can also be determined by Disc Centrifugation Photosedimentometer (DCP) through the ISO 15825 Standard by measuring the time a particle travels from the injection point to the light scattering detector in a known fluid. The particle sedimentation behaviour follows Stokes law. With known instrumental parameters (e.g. fluid density, fluid viscosity, disc rotational speed, etc.) the particle size can be determined by the travel time of the particle to the detector.

The constituent particle and aggregate size distribution can be quantified and D10/D50/D90 values determined via image analysis of a sufficient number of aggregates (2,000 at least).

1.3 Morphological characterisation

1.3.1 Shape including aspect ratio

The shape and aspect ratio of the various carbon black grades can be visualized through Transmission Electron Microscopy (TEM). Samples are prepared in accordance with ASTM D3849.

1.3.2 Crystallinity

The crystallinity of carbon black can be investigated using X-ray diffractometry (XRD). The main feature of the X-ray diffraction pattern of carbon black, using CuKα radiation with λ = 0.1542 nm, is a broad peak present within a range from 24.5 degrees to 26 degrees 2-theta. The graphite unit cell is used as a reference for carbon materials, and the major peak in the range from 24.5 degrees to 26 degrees 2-theta corresponds to the (002) plane of the graphite unit cell.

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1.4 Surface functionalisation or treatment of carbon black - oxidation

The main post-production treatment of carbon black is by means of oxidation. Volatile content is a surrogate measure for the degree of oxidative treatment of the carbon black surface. The measurement assesses the decomposition of functional groups and adsorbed substances present on the surface of carbon black. It is usually quantified using the ISO 21870 test standard.

1.5 Specific Surface Area

The ASTM D6556 test method is used to determine the specific surface area of carbon black through nitrogen adsorption.