

Indoor Environment CONNECTIONS Online-Friday, June 28, 2013

BLACK CARBON, CARBON BLACK, SOOT, WILDFIRES....ARE THEY CONNECTED?

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If you ever wondered what is the black residue on your air filter when your gas or oil heating unit is not working properly, or what makes the ghosting effect on your walls when burning candles, or what makes the tires black, or what does the residue of a wild fire contain, then this article will give you the answers. There have been an increasing number of complaints in recent years related to the appearance of dark stains observed in homes and commercial buildings. These stains outline the shapes of wall decorations, may form on carpets, plastic objects and other materials, and have been particularly noted around ventilation systems or electrical outlets. There could be several culprits for this phenomenon, such as black carbon/soot, carbon black, microbiological components (mold, fungus, mildew), tobacco smoke, cooking fumes, or just plain dust. Investigators also identified the metal fins on electric baseboards heaters or deteriorating insulation as additional sources. James R. Millette & al. in “Microscopic Investigation of Outdoor “Sooty” Surface Problems” gives a comprehensive description of possible causes of darkening agents on surfaces. However, more often than not, it is caused by black carbon/soot or carbon black.

Definition of terms

Carbon black (CAS number 1333-86-40, also called acetylene black, channel black, furnace black, lampblack or thermal black) is the manufactured material produced from controlled combustion or thermal decomposition of hydrocarbons. It is usually present in forms of colloidal aggregates with grape-like structure (aciniform). It is intensively used in tires, rubber and plastic products, printing inks and diverse coatings and its quality is determined by the specific surface area, particle size and structure, its conductivity, and color. A TEM micrograph of carbon black is presented in Figure 1. (mirica1.jpg goes here)

Black carbon and soot are somewhat similar with carbon black in terms of morphology and size, but these terms are used in conjunction with “unwanted” products of incomplete combustion of hydrocarbons, as opposed to the “wanted” and controlled process that produces the industrial carbon black. Black carbon and soot are produced for example in the case of fuel-based heaters, cars exhaust, wood fires, candles, and even food cooking. The size and shape of these particles are dependent on the nature of the fuel and the combustion temperature.

There is no clear definition that differentiates between black carbon and soot. ASTM D6602 “Sampling and Testing of Possible Carbon Black Fugitive Emissions or Other Environmental Particulate, or Both” is the best standard that completely defines the terms and the proper testing procedures of carbon black present in a mixture of various environmental particles. ASTM D6602 defines soot, but does not mention black carbon. This term is vehiculated lately in regards to pollution and the effects on global warming and climate changes. However, the indoor air quality industry professionals use the black carbon and soot terms interchangeably and this will be the case in this article as well. A TEM micrograph of black carbon/soot produced by an oil heater is presented in Figure 2. (mirica2.jpg goes here)

There are some important differences between carbon black and black carbon/soot. Carbon black is more uniform in size than black carbon/soot and the particle and aggregate size vary depending on the grade. The size of the primary particles ranges between 10-100nm up to 200-500 nm. The size of black carbon/soot particles is mostly in the upper range mentioned above, and the morphology of the aggregates differ. Also, the “neck” connecting the particles is small when compared to the primary particle diameter in carbon black, whereas it can be as large as the actual particle in black carbon/soot.

Other important differences are: the amount of aciniform particles (>97% in carbon black and variable starting from 60% in black carbon/soot), the concentration of sulfur (<2% in carbon black and variable in black carbon/soot, but usually higher than in carbon black), and the amount of residual hydrocarbons (low in carbon black, varying up to 20% in black carbon/soot). However, there are isolated cases where the differentiation between black carbon/soot and carbon black is not possible, especially if the conditions that yielded black carbon/soot mimic the industrial process of carbon black production.

Sampling and Analysis

ASTM D6602 describes in details the procedures and protocols to follow in order to sample and analyze black carbon/soot and carbon black. These procedures can in fact be extended for sampling surfaces by the wiping method for analysis of dust in general. One of the most important decisions that needs to be taken at the beginning of the sampling procedure is the selection of the area to collect a representative sample. Particles with the submicron size of black carbon/soot easily penetrate furnace or air conditioning filters. Only high efficiency electronic air cleaners may have the ability to retain them. Black carbon/soot particles that build up in the air collide, agglomerate, and they will deposit on various surfaces under the effect of gravity. Due to their electrostatic properties, good surfaces to sample are TV’s and computer displays, plastic surfaces, windows, mirrors, and the internal surfaces of refrigerators. Painted surfaces are not recommended due to the transfer of the paint on the sampling media and hence, a loaded background that hinders the analysis.

However, if painted surfaces are absolutely necessary to be sampled, excessive rubbing should not be employed. Instead, light pressure applied to the surface should be sufficient. As sampling media, one square inch alcohol pads (found in drug stores), polyester/cotton balls, or glass fiber

pads can be used. Sticky tapes (tape lifts) are not recommended for this analysis. There are laboratories that approach the analysis of black carbon/soot and carbon black using only light microscopy. Therefore, it is important to highlight in this article the appropriate methods for the analysis of these analytes. The selection of these methods is dictated primarily by the size of the particles. It was mentioned before that black carbon/soot and carbon black particles are mostly submicron size.

Therefore, in order to resolve the submicron size and the aciniform morphology of particles of black carbon /soot and carbon black, the resolution power of Transmission Electron Microscopy (TEM) is needed. In fact, ASTM D6602 designates TEM as the mandatory evaluation technique. Examination of the samples using light microscopy should be used only as a screening/presumptive method. Even the best light microscopes can only achieve 1,000x magnification. At this magnification, observation is limited to particles no smaller than 300nm. When optical microscopy is used, the particles in the sample are examined for traits such as color, size, and dispersion properties in the mounting medium. However, the results derived by this method should not be used as a conclusive determination regarding the presence of black carbon/soot or carbon black. The same ASTM D6602 method mentions using Scanning Electron Microscopy (SEM) as ancillary method for black carbon/soot and carbon black analysis. But similar to light microscopy, the method should be used only for screening purposes or for supporting the TEM data.

The initial step in the analysis of the samples in the laboratory involves the extraction of the particles from the sampling media. For this extraction, the media is sonicated in iso-propanol or chloroform. The resulting suspension is used for analysis. For the TEM analysis, droplets of 5-10 μ l are drop-mounted on formvar-coated copper grids, the solvent is left to evaporate, and the grid is examined using 80-100 keV at magnifications up to 100,000x. The particles present on the grid are examined for size, shape, and the aspect of the agglomerations. Elemental analysis using an Energy Dispersive X-ray detector (EDX, usually an attachment to the TEM instrument) is very useful for verifying the composition of the particles.

A large variety of nanosized particles, such as iron oxides, titanium dioxide or aluminum oxide have acinoform morphology similar to black carbon/soot and carbon black. For example, titanium dioxide (TiO₂) is a common interference due to its spherical morphology and its expansive use in food and other diverse commercial and pharmaceutical products. Hence, the elemental composition determined by EDX is quite necessary to verify if the material in the sample is actually black carbon/soot or carbon black.

The concentration of black carbon/soot and carbon black in a sample can be derived by the Visual Estimation Technique (VAE), widely used in the asbestos industry. The technique estimates the relative projected area of a certain type of particulate in a mixture by comparison with data derived from analysis of calibration materials having similar texture and particulate content. For best results, a compilation between the analysis derived by optical microscopy and TEM should be used in this case.

NIOSH 5000 is the method that addresses the analysis for carbon black in air samples. This method is gravimetric (it measures the total dust that was collected during a certain period of time), non-specific, and therefore, prone to interferences with any other components of the dust present in the air at the time of collection. This method can be used as initial screening or for OSHA compliancy. Unless additional methods employing electron microscopy are used to characterize the particles in terms of morphology and assess if they really are consistent with carbon black, this method does not give any additional information than the regular methods used to measure the exposure to total nuisance dust (such as NIOSH 0500/0600).

Identifying the origin of black carbon/soot formation can help to eliminate the potential sources. Source identification relies upon the analysis of the chemical fingerprint of the residual hydrocarbons that are still present in the residue. This can be accomplished using Fourier Transform Infrared Spectroscopy (FTIR) and Gas Chromatography (GC). These methods analyze for the presence of selected functional groups that distinguish sources such as paraffin residue from candles or fuel oil from oil heaters.

For example, in the case of combustion of liquid fuels and diesel, the residue usually contains a large variety of residual hydrocarbons and inorganic components (such as iron-, chromium-, and nickel-containing dust from deteriorating engines) besides black carbon/soot. Combustion of candles leaves residues that contain alkanes, alkenes, wax esters, and polycyclic aromatic hydrocarbons (PAH). Therefore, the combined analysis by FTIR, GC, and the elemental composition derived by EDX mentioned above offer a comprehensive picture that enables the source identification.

What about wildfires?

The recent influx of insurance claims prompted by the wildfires in California and Florida revitalized the analysis of black carbon/soot in this context. However, considering the materials that are subjected to combustion, the analysis of wildfire residues should cover more than black carbon/soot. In fact, the concentration of black carbon/soot in these residues is generally low compared with carbonized (carbonaceous) material. The generic term of carbonized material applies to char, ash, and coke/coal. Carbonization, as a chemical term, is defined as a chemical process of transformation of an organic substance by means of pyrolysis in a residue with carbon as the main elemental component.

Char is defined in ASTM D 6602 as being composed of particles that are larger than 1 μ m and may preserve the original cellular morphology of the material that was burnt. These particles can range up to millimeter in size. Char is mostly elemental carbon, but it may also contain trace concentrations of mineral components and ash. The main difference between ash and char is that ash may not preserve any of the original morphology of the precursor and it may have a higher concentration of inorganic components due to the complete consumption of some of the organic matrix.

The carbonized material from a wildfire is not significantly different than the components of a residential fire where wood was the primary component that was combusted. However, the

residential fire debris usually contain a higher concentration of black carbon/soot and also, char from plastics and fabrics. A Polarized Light Microscopy (PLM) micrograph of residue from wildfire in California is presented in Figure 3. The black particles in the image are carbonized material (char) (mirica3.jpg goes here)

The analysis of wild or residential fire is similar to black/carbon black analysis. The same sampling and sample preparation procedures can be used. Due to the large difference in particle size between the carbonized material and the black carbon/soot, the TEM analysis needs to be accompanied in this case by optical microscopy. SEM with EDX analysis could also be employed as ancillary method. The TEM part of the analysis addresses the black carbon/soot component and it is run as previously described. The optical microscopy addresses the carbonized material component. For this part of the analysis, aliquot of the suspension prepared as described before is filtered onto a filter membrane (such as 0.1 μm pore size polycarbonate filter). The particles retained on the filter are analyzed using optical microscopy (epi-reflected and polarized light microscopy). The samples are analyzed for traits such as color, size, morphology, evidence of cellular morphology. If SEM with EDX analysis is used, the elemental composition of the carbonized fragments is useful to assess if the particles are really carbon-based. The concentrations of the various components of the debris are best derived using the VAE technique.

In conclusion, the analysis for black carbon/soot, carbon black, and carbonized material in general is a complex process that requires substantial training and expertise on the part of the analyst. It involves analysis of primary particles and agglomerations for size and morphology using the sophistication and the resolution level of the TEM instrumentation. The EDX elemental composition of the grape-like particles detected in a sample is useful to confirm if the particles are indeed carbon-based, and therefore consistent with black carbon/soot or carbon black. Light and SEM microscopy could be added as additional methods for supporting the evidence of the presence or absence of these analytes in a sample; however, the results derived by these techniques alone should not be used as a conclusive determination. If detected, the source identification using FTIR and GC analysis helps to eliminate the culprits for black carbon/soot and carbon black formation.

Eugenia Mirica, Ph.D., is the Project/Client Services Laboratory Manager of the Materials Science Division and Senior Materials Scientist at EMSL Analytical, Inc., Westmont, NJ. She earned her Ph.D. in Materials Science from Stevens Institute of Technology. She joined EMSL Analytical in 2002 where she has been working on analyses for materials identification, morphological and chemical characterization of materials, product comparison, contamination control, and forensic analysis. She can be reached by email at emirica@emsl.com or by phone at 1-800-220-3675 ext. 1247.

Mirica, Eugenia <EMirica@emsl.com>

to 'walter, Dana

Walter,

The EPA method below mentions that the limit of detection of the method used to determine the analytes is 1%. This is the only formal method that mentions a limit for analysis in bulk material and it is for asbestos, a known carcinogenic. Since carbon black is also known as carcinogenic, this is most likely the connection.

However, there is no formal guidance that relates the two.

D'Ulisse, Dana

to 'walter, Eugenia

Walter,

Attached is a link to the EPA method mentioned

http://www.epa.gov/region1/info/testmethods/pdfs/EPA_600R93116_bulk_asbestos_part1.pdf

It is the method for determination of asbestos in bulk building materials. We are using similar visual area estimation techniques when determining the concentrations of combustion by products.

Thank you,

Dana D'Ulisse | *Laboratory Analyst*

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to me

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Walter,

As per our phone conversation here is a link to an article written by Eugenia Mirica about Carbon Black testing.

http://www.ieconnections.com/archive/aug_08/#article7

You may also refer to ASTM D6602 for further information. As mentioned, there is no formal guidance for what is considered contaminated. The 1% threshold commonly used by insurance companies is likely borrowed from EPA 600.

Thank you,

Dana D'Ulisse | Laboratory Analyst

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