Detection of aerosols using Laser-Induced Breakdown Spectroscopy (LIBS)

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Aerosols released in the course of industrial processes may represent a threat for workers and the environment. Possible emission of micrometric / submicrometric particles such as CNT (carbon nanotube) powders at production sites or heavy metals at exhaust stacks of foundries and incinerators are two telling examples. The above considerations emphasize the need to develop an instrument allowing in-situ and real-time elemental identification and mass concentration determination of airborne particles. A LIBS-based (Laser Induced Breakdown Spectroscopy) system may be the appropriate tool to deal with such delicate issue.

LIBS consists in focusing a powerful laser pulse on a material (solid, liquid, gas, aerosol, nanoparticle flow) whose elemental composition is to be determined. The strong heating of the sample at the focusing spot leads to the ignition of a hot and luminous transient ionized gas called plasma. Plasma light contains the signature of all the chemical elements the interrogated material is made of. This signature is read by sending the emitted light through a spectrometer equipped with a detector. The LIBS signal presents itself as an optical emission spectroscopy spectrum displaying lines corresponding to the detected elements.

LIBS displays advantages of great interest for industrial applications. Samples do not need preliminary preparation making LIBS eligible for on-line monitoring. It is a multielemental analysis technique with simultaneous detection of all the elements contained in the sample. Being all optical, it is not intrusive requiring only optical access to the sample and thus allowing in-situ measurements. LIBS is therefore a promising analytical chemistry method intended to be operated at industrial sites. Current elemental analysis techniques require time consuming procedures involving several steps. Skilled personnel are sent to the sites where samples are to be picked up. Back to the laboratory, these are prepared prior to the analysis itself. The LIBS systems developed at INERIS are intended to be operated on-site, in automatic mode, without manual intervention except for maintenance.

Experiments carried out by INERIS (Dutouquet *et al*, 2014) have demonstrated the potentialities of the LIBS technique for in-situ and real time quantitative detection of heavy metals in particulate form. A transportable LIBS system was first calibrated in laboratory using aerosol generators. Particle size distribution of the generated aerosol was obtained from an APS (Aerosol Particle Sizer) whereas the mass concentration of the targeted elements was inferred from TEOM (Tappered Element Oscillating Microbalance) measurements. The LIBS analyzer was then put to the test in a French research and development center specialized in metal casting (CTIF). Mass concentrations of copper particles released through an evacuation duct when melting copper bars in an induction furnace were monitored in real time.

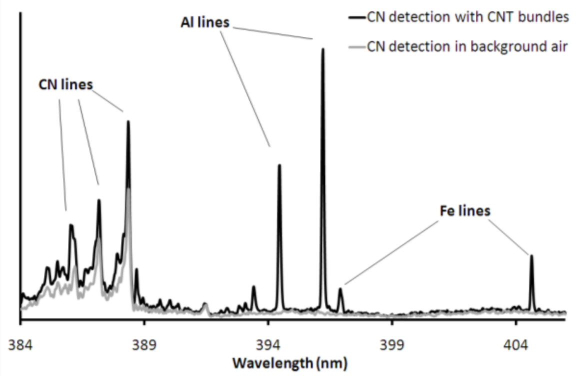

Figure 1. Simultaneous detection of CN, Al and Fe lines, spectral signature of a CNT ball

Other experiments aiming at detecting possible particle emission when handling CNT powders were performed in a high safety cell at ARKEMA research center (Dutouquet, 2015). A LIBS system was implemented along with TEM (Transmission Electron Microscopy) samplers. TEM sampling allowed differed analysis of morphologies, sizes and elemental composition of the emitted particles. Real time particle elemental composition monitoring was assured by LIBS. Though the obtained results are qualitative, LIBS spectra crosschecked with TEM observations of the collected particles demonstrated LIBS potentialities to identify carbone nanotubes intertwined in bundles. A spectral signature based on simultaneous detection of carbon, iron and aluminum (the latter two being catalyst and catalyst support respectively) was found to act as a fingerprint allowing CNT ball identification.

A prototype mounted on wheels and encased in a 19” rack is now operational and available at INERIS for field measurements.

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