Multi-scale assessment of soot using electron microscopy: applications on soot from bench-scale fire of polymers

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Multi-scale assessment of soot using electron microscopy: applications on soot from bench-scale fire of polymers

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Soot is a degradation product of material burning, having the multi-scale fingerprints of the conditions in which it is formed. Micron sized aggregate morphology is dependent on residence times and burning rates, whereas particle size and nanoscale order reflects the burning source and temperature rates. In this work, we will present comparison of techniques for ex-situ analysis of soot probed in-flame from flaming combustion of natural and synthetic polymers, by means of SEM/STEM (Scanning electron microscope in transmission mode) and TEM.

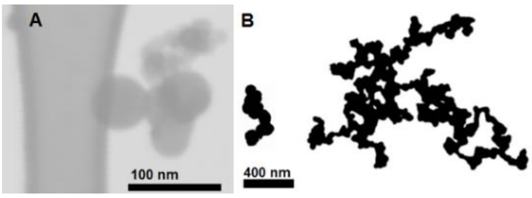

Figure 1: **A.** STEM image of soot agglomerate. **B.** Binarized images from STEM recordings [1].

STEM was practical for aggregate measurements and rapid screening due to the faster handling of multiple samples. STEM and TEM recordings were analyzed by manual and semi-automated techniques (Fig.1). Nonetheless, 2D measurements can lead to errors in aggregated media. 3D morphology was then analyzed by TEM (Fig.2) thanks to the volume data provided by tomography reconstruction. Advantages and disadvantages of techniques will be emphasized such as the limitations of STEM resolution, of TEM reconstructions, downsides of the image computation for overlapping particulates in aggregated media (i.e. both physical overlapping and projection artifacts [2]).

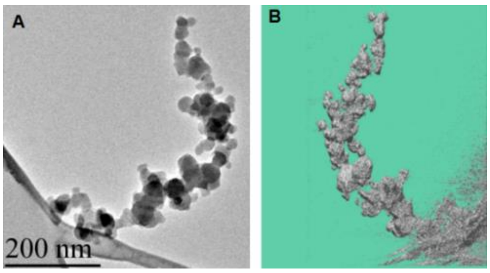

Figure 2: **A.** TEM image of soot. **B.** Corresponding tomography reconstruction from stack of rotated images (-60° to +60°).

Finally, some high resolution assessments will be made (Fig.3), based on our previous studies on nanoscale order indicating promising results in terms of scenario identification for bench-scale fire [1], further details on subject is given in the published article [1] and in the literature references therein.

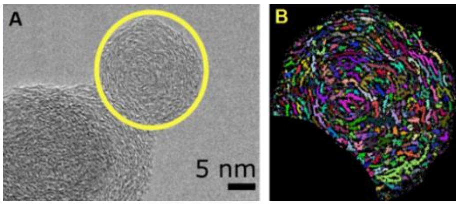

Figure 3: **A.** TEM recording of particles at high magnification. **B.** Corresponding analysis to highlight the nanoscale order [1].

To conclude, we will introduce some numerical modeling aspects (material-radiation interaction) which are fed by microscopy measurements [2] and discuss the unknowns. The work will allow the discussion of prospective insights with the community, on the measurement of carbonaceous materials and on the implications for other techniques used, e.g. for particle counting and monitoring in complementary domains as environment and toxicity.

References:

1. Okyay, G., Bellayer, S., Samyn, F., Jimenez, M., & Bourbigot, S. (2018). Characterization of in-flame soot from balsa composite combustion during mass loss cone calorimeter tests. *Polymer Degradation and Stability*, 154, 304-311. http://doi.org/10.1016/j.polymdegradstab.2018.06.013.

2. Okyay, G.. (2016) *Impact of the morphology of soot aggregates on their radiative properties and the subsequent radiative heat transfer through sooty gaseous mixtures.* PhD diss., Univ. Paris Saclay - CentraleSupélec, France.

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