

NANOPLASTICS ANALYSIS WITH EMILIE™

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Nanoplastics and sub-microplastics are ubiquitous in the environment. However, there exists a methodological and technological gap for their sampling and characterization. As shown in Figure 1, sub-microplastics and nanoplastics have sizes below the limit of common microscopy hyphenated with spectroscopy techniques such as Fourier-transform infrared spectroscopy (FTIR), Raman microspectroscopy, and focal plane array (FPA)-FTIR [1]. AFM-IR (atomic force microscope - infrared spectroscopy), TEM-EDX (transmission electron microscopy with energy dispersive X-Ray spectroscopy), and SEM-EDX (scanning electron microscopy with energy dispersive X-ray spectroscopy) allow for the chemical characterization of individual nanoparticles, but the process is extremely time-consuming and the required instrumentation expensive. As an alternative, we propose nanoelectromechanical Fourier-transform infrared spectroscopy (NEMS-FTIR) with EMILIE™, allowing for the rapid bulk chemical characterization of sub-microplastics and nanoplastics.

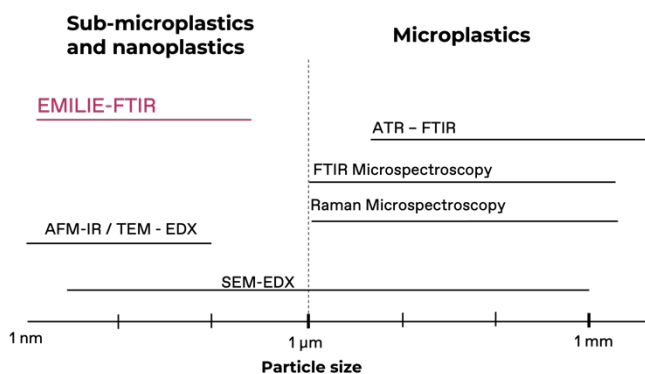


Figure 1. Available methods for nano- and microplastics analysis and their applicable particle size range.

The measurement principle of EMILIE™ is based on the photothermal heating of the sampled nanoplastic, causing a measurable frequency detuning of the EMILIE™ resonator chip [2,3]. The frequency detuning, which is measured by the frequency tracking electronics PHILL™,

is a direct measure for the absorbed light power [4]. With the photothermal sensing principle, EMILIE™ has a limit of detection in the lower picogram range [5].

METHODOLOGY

A polypropylene nanoparticle dispersion (54 nm average diameter, 15.75 ppm) was nebulized for 1 minute with a sampling flow of 1 L/min (10 μL/min flow-rate at the syringe pump, 10 L/hour nebulizer gas, 30-35 L/h make-up flow, 65 L/hour flow rate at the chip site) and the nebulizer output sampled by impaction on the nanomechanical resonator chip with the help of our EMILIE™ sampling accessory [6]. A schematic of the sampling setup is shown in Figure 2. As an alternative to the spray nebulization, the aerosol can also be produced by electrospray [2,3] or with a piezoelectric mesh nebulizer.

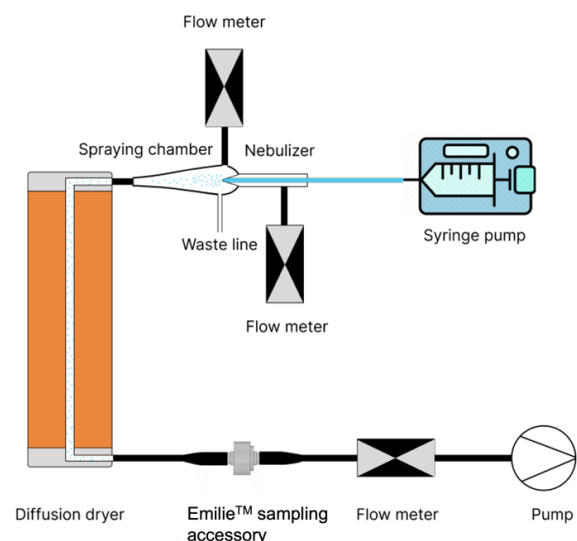


Figure 2. The nanoplastic solution is nebulized and dried prior to impaction on the nanomechanical resonator chip with the help of the EMILIE™ sampling accessory.

Figure 3 shows the center perforation of the EMILIE™ chip before and after sampling of nanoplastics. The perforation

enables the efficient filtering and collection of airborne nanoparticles on the EMILIE™ chip.

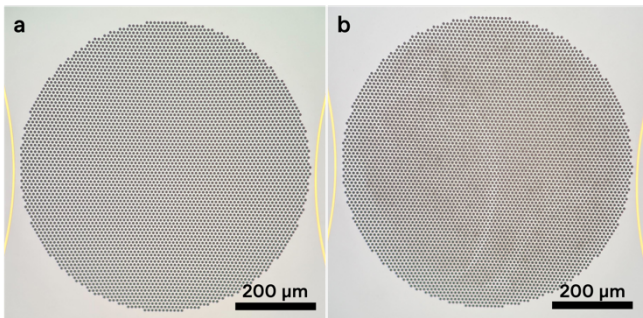


Figure 3. Micrograph of the perforation of the EMILIE™ nanomechanical sampling and sensing chip before a) and after b) sampling of 100 nm polystyrene nanoparticles.

After sampling, the chip is removed from the sampling apparatus and transferred to the EMILIE™ nanomechanical infrared analyser detector chamber, which is placed inside the sample compartment of a commercially available FTIR spectrometer (Vertex 70, Bruker optics, USA) for NEMS-FTIR analysis as illustrated schematically in Figure 4.

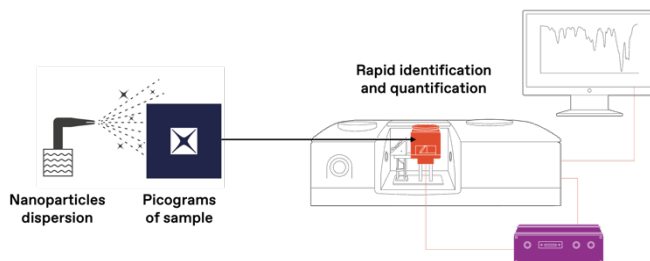


Figure 4. Nanoplastics analysis with EMILIE™.

RESULTS

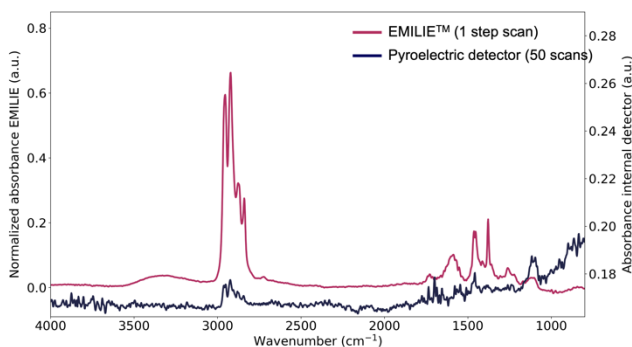


Figure 5. FTIR spectra of polypropylene nanoplastic spheres (54 nm average diameter) acquired with EMILIE™ (pink) and the FTIR's internal detector (dark blue).

Figure 5 shows the comparison of the spectrum of a nanomechanical resonator sampled with polypropylene nanoplastic recorded in transmission mode using the FTIR's internal detector and using EMILIE™. The nanomechanical sensor chip made from silicon nitride is transparent to infrared radiation but for a strong peak at 866 cm⁻¹, which is used as an internal standard to normalize the signal obtained from EMILIE™. As shown in Figure 5, while it is quasi-impossible to identify polypropylene by transmission mode using the FTIR's internal detector, EMILIE™ provides a clear signal with clearly identifiable characteristic peaks.

BENEFITS OF USING EMILIE™

- Rapid and easy collection of nanoplastics with sizes ranging from 10 nm to 500 nm directly on the surface of the nanomechanical sensing element.
- No sample transfer necessary.
- Rapid chemical characterization.
- High sensitivity without cryogenic cooling.

REFERENCES

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[6] Schmid, Silvan, et al. "Real-time single airborne nanoparticle detection with nanomechanical resonant filter." *Scientific reports* 3.1 (2013): 1-5.