Invisible-Light Labs

from proteins to stars

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NANOPLASTICS ANALYSIS WITH EMILIE™

Invisible-Light Labs GmbH

October 17th, 2024

Nanoplastics are ubiquitous in the environment. However, a methodological and technological gap exists in their sampling and characterization. Nanoplastics have sizes below the limit of microscopy hyphenated with spectroscopy techniques such as Fourier-transform infrared spectroscopy (FTIR), Raman microspectroscopy, and focal plane array (FPA)-FTIR. 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. Still, the process is extremely time-consuming, and the required instrumentation is 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.

The measurement principle of EMILIE™ is based on the photothermal heating of the sampled nanoplastics, causing a measurable frequency detuning of the EMILIE™ resonator chip [2,3]. The frequency detuning, measured by the frequency tracking electronics PHILL™, is a direct measure for the absorbed light power [4]. With the photothermal sensing principle, NEMS-IR has a limit of detection in the picogram range [5].

METHODOLOGY

Polypropylene (PP) (54 nm average diameter, Lab 261, USA) and polystyrene (PS) (Sigma-Aldrich, Germany, 100 nm average diameter) dispersions were prepared in distilled deionized water. Dispersions were nebulized for 1 minute with a sampling flow of 1 L/min (10 µL/min flowrate 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. Alternately, ~20nL sample aliquots were deposited on the surface of the EMILIE™ nanomechanical sampling and sensing chips using a nanodrop dispensing tool (PIPEJET™, Hamilton Freiburg GmbH) corresponding in 1 ng of sample deposited (Figure 1).
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.

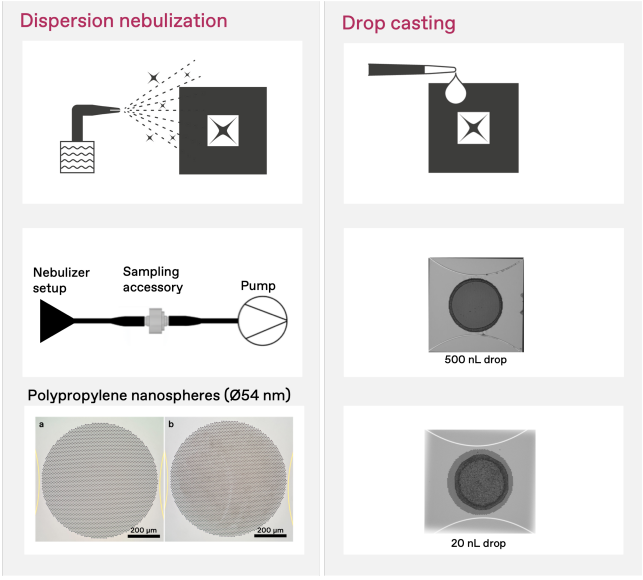


Figure 1. Nanoplastic solutions nebulized and sampled by impaction directly on the EMILIE™ nanomechanical sampling and sensing chip with the help of the EMILIE™ sampling accessory (left). Alternately, a nanoplastic solution can be dropcasted directly on the surface of the EMILIE™ nanomechanical sampling and sensing chip (right).

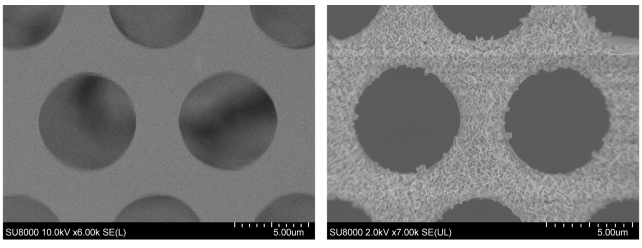


Figure 2. SEM micrograph of the perforation of the EMILIE™ nanomechanical sampling and sensing chip before (left) and after (right) sampling of PP nanoparticles by nebulization.

Figure 2 shows an EMILIE™ sampling and sensing chip before and after sampling of nanoplastics by nebulization. The perforation enables the efficient filtering and collection of airborne nanoplastics.

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RESULTS

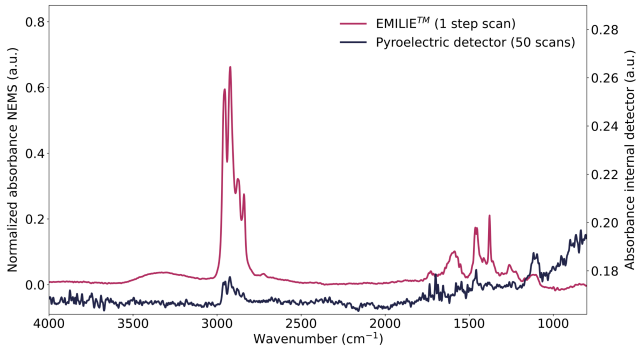


Figure 3. FTIR spectra of nebulized polypropylene nanoplastic spheres (54 nm average diameter) acquired with EMILIE™ (pink) and the FTIR's internal detector (dark blue).

Figure 3 shows the comparison of the spectrum of a nanomechanical resonator sampled with polypropylene nanoplastic particles 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 3, 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. Figure 4 shows NEMS-FTIR spectra for dropcasted solutions of PP and PS.

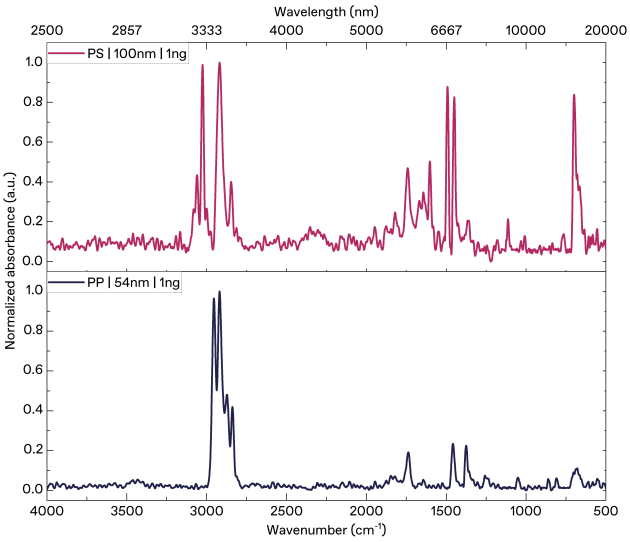


Figure 4. NEMS-FTIR spectra of 1 ng of dropcasted 100 nm PS nanoplastic particles (top) and 1 ng 54 nm PP nanoplastic particles (bottom).

BENEFITS OF USING EMILIE™

- Rapid collection of nanoplastics directly on the surface of the nanomechanical sensing element
- Rapid bulk chemical characterization
- High sensitivity

REFERENCES

[1] Schwaferts, Christian, et al. "Methods for the analysis of submicrometer-and nanoplastic particles in the environment." *TrAC Trends in Analytical Chemistry* 112 (2019): 52-65.

[2] Andersen, Alina J., et al. "Nanomechanical IR spectroscopy for fast analysis of liquid-dispersed engineered nanomaterials." *Sensors and Actuators B: Chemical* 233 (2016): 667-673.

[3] Yamada, Shoko, et al. "Photothermal infrared spectroscopy of airborne samples with mechanical string resonators." *Analytical chemistry* 85.21 (2013): 10531-10535.

[4] Schmid, Silvan, Luis Guillermo Villanueva, and Michael Lee Roukes. "Responsivity and Sensitivity." Fundamentals of Nanomechanical Resonators. Springer International Publishing, 2023. 175-203.

[5] Luhmann, Niklas, et al. "Nanoelectromechanical Infrared Spectroscopy with In Situ Separation by Thermal Desorption: NEMS-IR-TD." *ACS Sensors* 8.4 (2023): 1462-1470.

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