Application Notes AN M153

Reliable Identification of Microplastics of any Dimension, on any Filter

Introduction

Microplastic particles (MPP) are by definition polymer particles ranging in size from 5.000 to 1 µm. They originate from polymer beads added to cosmetic and personal care products as well as abrasion of macroscopic objects such as plastic bottles, synthetic textiles, and car tires. Today, these particles are detected ubiquitously around the globe in aquatic environments. Oceans, rivers, artic ice, lakes, bottled drinking water and even tap water is now contaminated with microplastic particles. They threaten the health of aquatic animals like fish, shells and crustaceans. Furthermore, MPP are good transporters for pathogenic bacteria and persistent organic pollutants (POP) that adhere to the particles surface. Also, they can release toxic plasticizers that were used in the manufacturing process.[1]

Ultimately, these particles make their way in to the human body either through the food chain or by direct intake through our drinking water. Smallest particles can accumulate in organs and are suspected of entering the bloodstream.[2]
Visual microscopy is the method of choice to locate MPP but identification is limited to particles down to 100 µm. This often leads to wrong estimates of the number of plastic particles and does not offer the possibility to identify the type of polymer.

Keywords	Instrumentation and Software
Microplastics	LUMOS II FTIR microscope
Particle analysis	HYPERION FTIR microscope
On-filter analysis	OPUS Spectroscopy Software
Fibers	B-KIMW Polymer spectral library
Sediments	ATR-LIB-COMPLETE spectral library

To overcome this obstacle, Bruker offers the analytical solution for reliable particle identification down to 5 µm through the combination of visual and FTIR microscopy using the ATR technique. ATR stands for attenuated total reflection and is a contact based measurement method.
It is trouble-free and applicable to a wide variety of samples. Unlike transmission or reflection measurements, micro ATR analysis will generate IR spectra of the highest quality, regardless of sample shape and dimension. Moreover, with ATR the MPP can be analyzed on almost any filter material or even without prior separation, e.g. on complex matrices such as sediments. When applying transmission or reflection, dedicated IR transparent or reflective filter materials need to be utilized and the spectral data quality is highly dependent on the size of the MPP.

Figure 1: LUMOS II FTIR microscope.

Automated FTIR ATR microscope

A fully automated FTIR microscope like the LUMOS II guarantees easy handling and a convenient workflow for the analysis of MPP. It is a stand-alone microscope with low space requirements and has all components integrated, including a software controlled fully motorized sample stage and ATR crystal within the objective. It already has found its way into many microplastic laboratories around the globe, like France, [3] the United states [4], China [5] and Turkey [6], from research institutes to numerous partners in industry.

Visual identification

The combination of visual microscopy and FTIR is a powerful tool in MPP analysis. It retains all the advantages of optical microscopy For particle localization while allowing reliable polymer identification.

Crossed visual polarizers

Transparent items are easily overlooked as they are hard to distinguish from the filter material. The automated visual polarizers in the LUMOS II FTIR microscope can overcome this issue by highlighting particles that shift the plain of polarization (see figure 2, top).

Dark field microscopy

Figure 2, a darkfield image recorded with the LUMOS II FTIR microscope is shown. By using the darkfield illumination, light directly reflected from the surface is blocked and only diffusely reflected light is collected. Contrast is greatly improved and particles are lighting up on an otherwise dark background. (see figure 2, bottom)

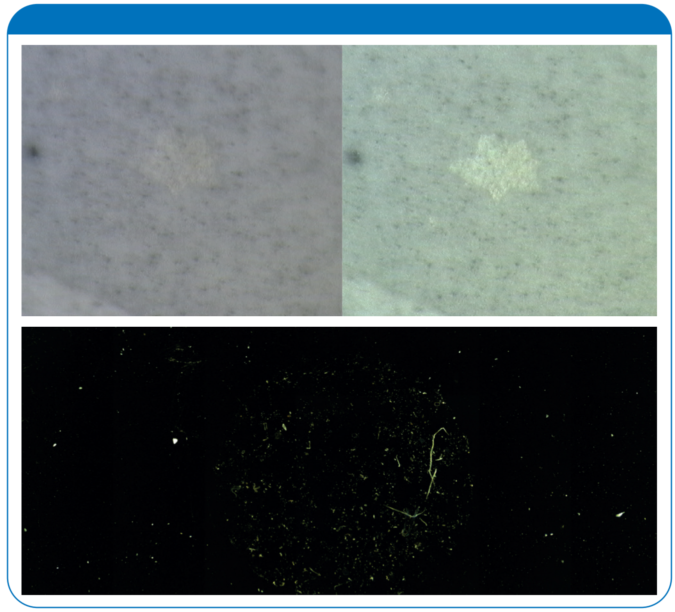

Figure 2: Visual enhancement options offered by the LUMOS II. The contrast improving effect of crossed visual polarizers can clearly be seen (top), while in the darkfield image, the particles light up on a gold filter for easy localization (bottom)

Precise measurement with ATR

To avoid background influence, the LUMOS II FTIR microscope is equipped with freely adjustable, fully automated knife edge apertures that allow defining specific measurement areas that only target the particle itself.

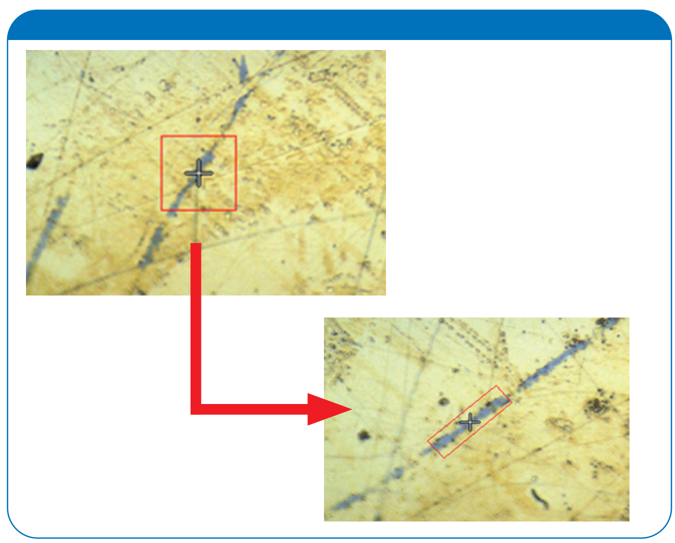

Figure 3: Example for the adjustment of the knife edge aperture (red box) to the desired measurement area.

Chemical identification

To identify microplastic particles with utmost reliability, the obtained spectrum is compared to a comprehensive polymer library. Bruker and the Kunststoff Institut Lüdenscheid (Germany) offer a specialized, constantly updated library containing all important polymers, additives and fillers to provide reliable and easy identification of any found particle. Additionally the general Bruker "ATR-LIB-COMPLETE" database with >26,000 entries is searched for comprehensive identification of any non-polymeric material.

Example 1: Particles in bottled drinking water

In our first case study, it was suspected that a problem in the bottling process led to water contamination. To investigate possible issues with the production batch, the bottled water was passed through a filter and the collected residue subjected to FTIR microscopic analysis.

A red fiber was found on the filter surface and measured with an aperture (red rectangle, figure 5) matching the fiber to avoid influence of the filter. The fiber was identified as polyester, giving indications of the source of the contamination.

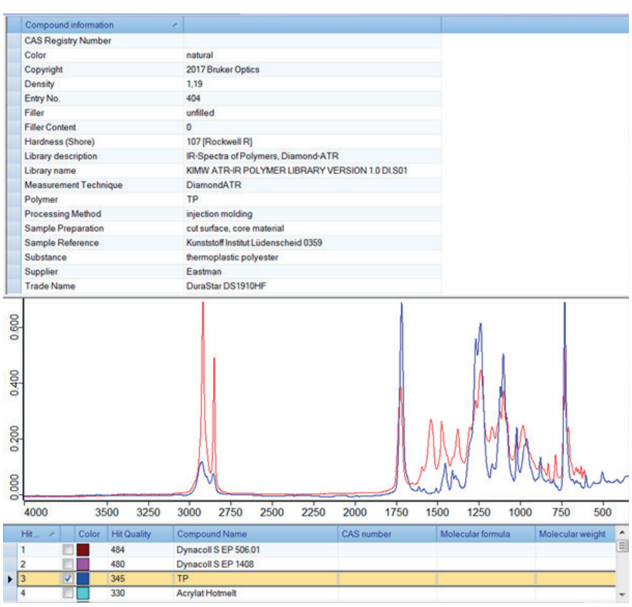

Figure 4: Library search result of the red fiber. The spectrum of the fiber is shown in red and the library spectrum in blue.

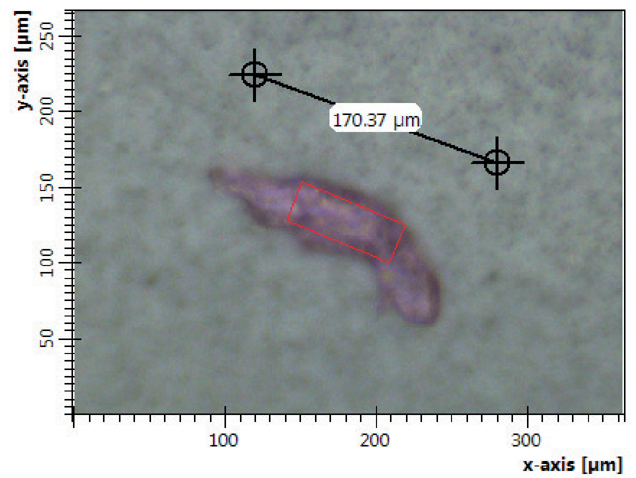

Figure 5: Red particle on a filter with indication of particle length and aperture size.

A similar sample required a different filter material. In figure 6, the particles are easily visible using 8x objective of the LUMOS II FTIR microscope.

They were measured using the ATR technique and the knife edge aperture (red rectangle) was adjusted to the particle size to avoid influence from the filter material. The collected high quality spectra were compared with a spectral reference library. Nomicroplastic was found and the particles were unambiguously identified as inorganic silica.

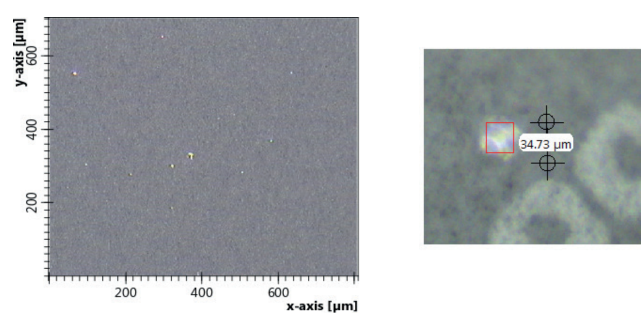

Figure 6: 800 x 800 µm cutout of a drinking water filter (left) and magnified particle together with its size and the fitted aperture (right; red box).

Example 2: MPP in environmental sediment sample

Sediment samples from a river bed were collected on site, and directly analyzed using the LUMOS II FTIR microscope. Once more aperture was adjusted to fit a thin fiber found on the sediment's surface.

A visual image and the resulting spectrum of the measurement can be seen in figure 7. Spectra of pristine quality were received and the fiber subsequently identified as polyamide.

Conclusion

Bruker offers a complete solution to tackle the challenging task of analyzing microplastic particles of any dimension and shape on virtually any filter substrate, or even in complex matrices such as sediments. With the fully automated LUMOS II FTIR microscope, particles down to 5 µm are readily located by visual microscopy, then measured by ATR FTIR microscopy and finally identified using powerful spectral libraries. The presented approach is very simple to apply and results in reliable high quality data.

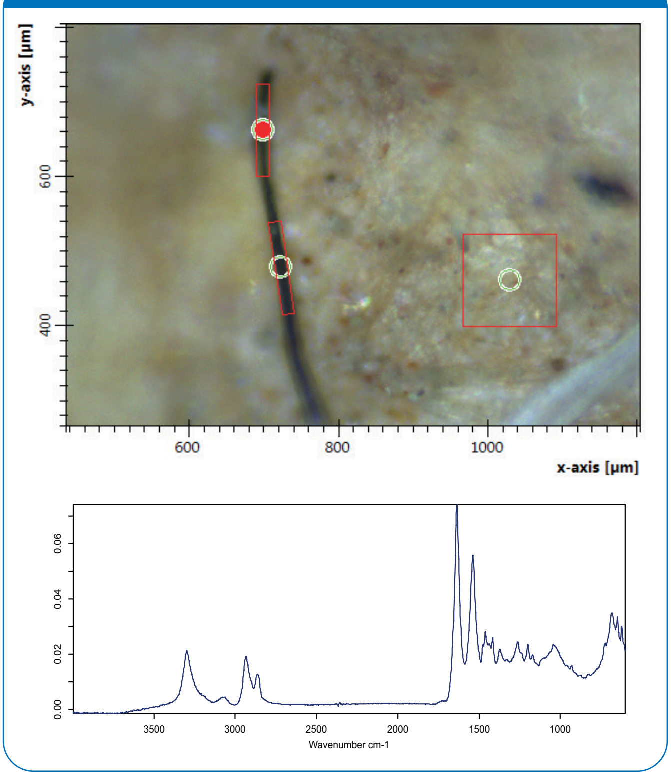

Figure 7. Fiber found in a river sediment and adjusted aperture (red rectangle) of the measurement. Bottom right: acquired spectrum of the fiber.

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