White Paper

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Microplastics Analysis Doesn't Need to Be So Hard

Infrared chemical imaging systems can significantly simplify microplastics analysis through a rapid automated workflow

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Overview

Microplastics in the environment are rapidly coming into focus as scientists begin to understand their penetration into our ecosystems and food chains. Researchers and analytical methods organizations are working to develop standardized analytical solutions to best characterize microplastics in terms of chemical identity, size, shape, and total mass.

A new approach in infrared chemical imaging that uses quantum cascade laser (QCL) technology greatly simplifies microplastics analysis through a fast, automated workflow. Its streamlined measurements provide effortless microplastics characterization in a fraction of the time of traditional analytical techniques.

Analytical techniques for microplastics analysis

While the size of microplastics of analytical interest ranges from less than 10 µm up to 300 µm, it is believed that the smaller the particle, the more risk it may pose. Visual microscopy is useful for inspection of microplastic particles, but it cannot identify the myriad types of plastics that are observed in the environment. In contrast, spectroscopic techniques can reveal specific chemical identities of plastics. Infrared spectroscopy (IR) involves shining infrared light on a particle to see which wavelengths it absorbs. Fourier transform infrared (FTIR) spectroscopy is the traditional choice for plastics analysis. To evaluate small analytes, FTIR is typically combined with a microscope. However, the large incoherent light source can be difficult to focus onto a small microparticle and because only a tiny fraction of the light can be absorbed, the resulting signal is very weak. As such, data must be acquired over a relatively long period of time in order to capture enough signal. Raman spectroscopy is a laser-based alternative in which the light can be focused onto a particle. Still, the response is very weak, making data acquisition extremely slow.

An infrared laser-based technology is now available in the Agilent 8700 Laser Direct Infrared (LDIR) chemical imaging system. This instrument features a bright infrared laser source with proprietary Quantum Cascade Laser (QCL) technology. It can focus all of the laser power onto a particle, so that a strong infrared signal can be acquired in only one second. The laser is easily tunable across the mid-infrared region, enabling custom data collection at specific wavelengths, if desired.

Laser Direct Infrared Imaging system

The workflow for the Agilent 8700 LDIR is remarkably simple. Being an infrared spectroscopy technique, sample preparation methods are largely the same as for traditional IR experiments. Once a clean sample is prepared, it is introduced to the instrument on an IR reflective slide. The LDIR performs a quick IR scan of the selected analysis area (in as little as one minute for a typical area) to locate all of the particles within a selected sample region and measure their dimensions. The instrument will then automatically acquire a spectrum for each individual particle, which will be compared in real time to the standard infrared library embedded in the software. A report is produced with identities, sizes, and statistics for all of the microplastic particles in the region.

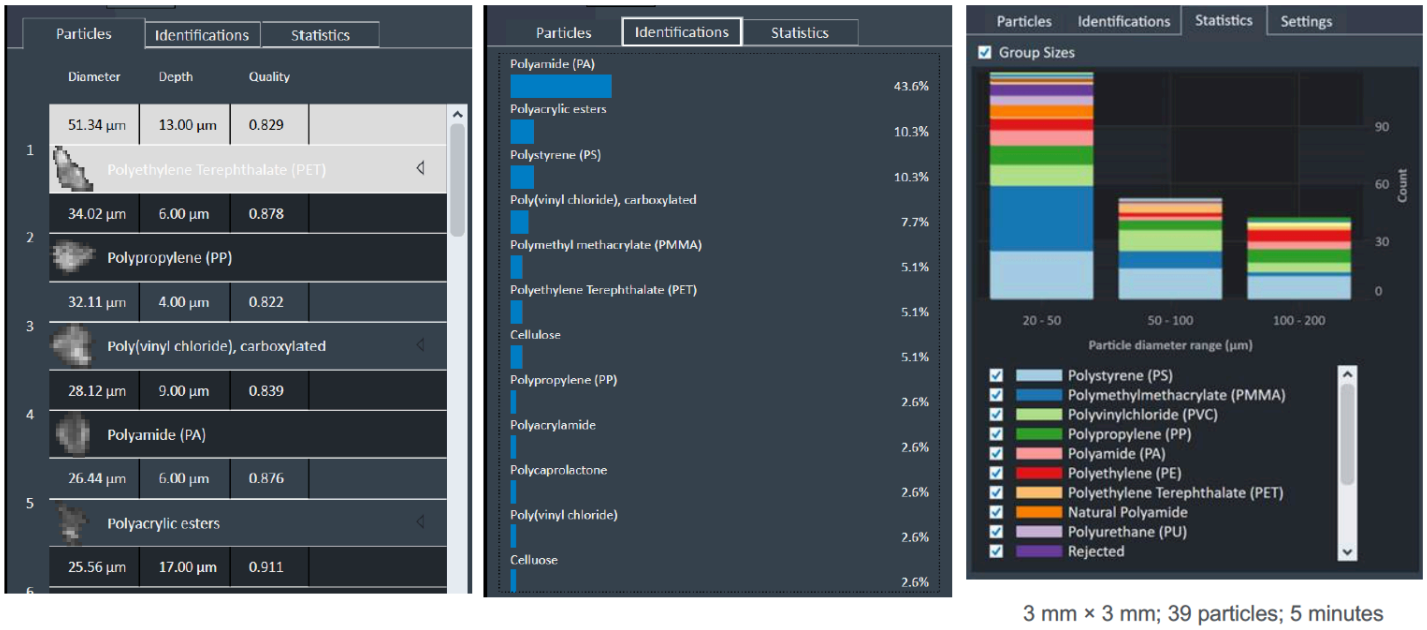


Figure 1. Reported results – Particle breakdown and statistical analysis.

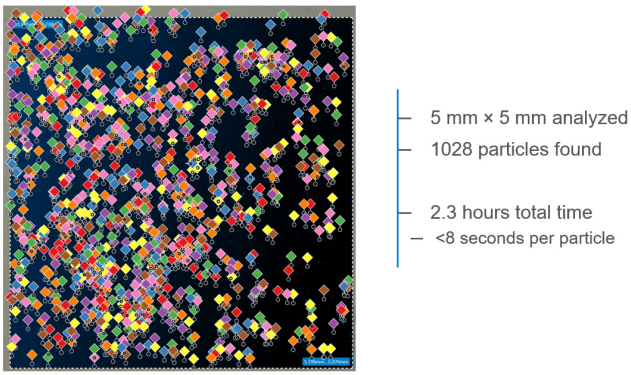


Figure 2. LDIR analysis of wastewater sample.

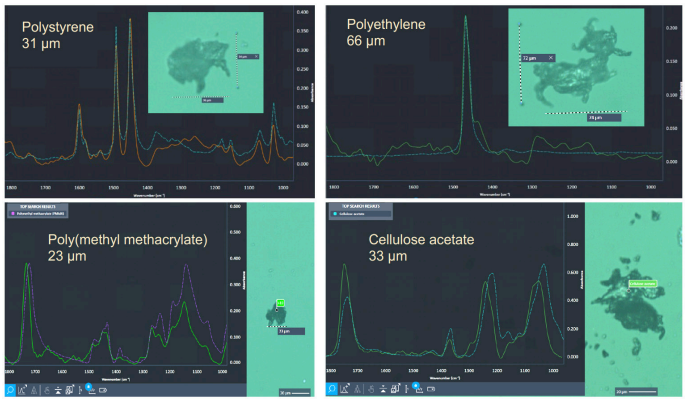


Figure 3. The software allows the data for individual particles to be examined.

Figure 1 shows a report for 39 particles that were detected and identified in a 3 mm x 3 mm area. The entire experiment, with real-time data analysis, was completed in only five minutes, which is less than eight seconds per particle.

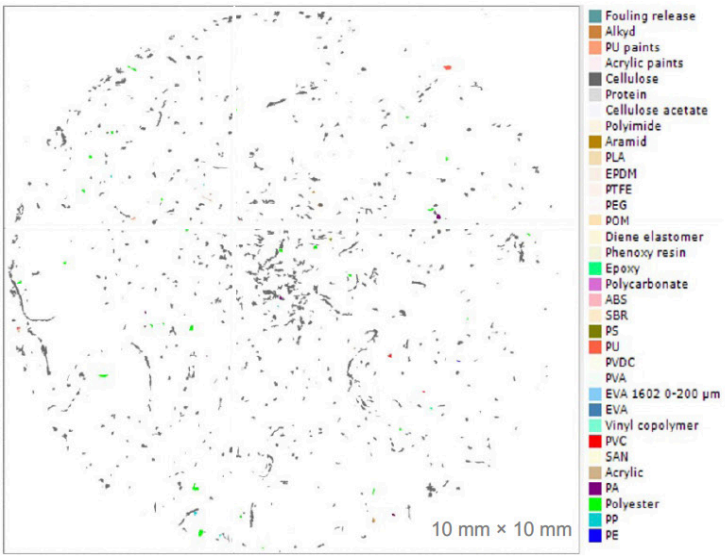
Figure 2 shows a sample of sewage sludge that was used as fertilizer—a possible route to human consumption of microplastics—and was prepared and deposited onto a slide for analysis. The 8700 LDIR detected 1028 microparticles in a 5 mm x 5 mm area at a rate of approximately eight seconds per particle. The map of particles, which was automatically generated by the software, can be seen in Figure 2. Color coding indicates the different classifications of plastics. In addition to the particle map and the type of report shown in Figure 1, the software allows the data for individual particles to be examined, such as the information shown in Figure 3. Solid lines in the spectra represent measured data, while dotted lines represent references from the library. Additionally, the instrument can "zoom in" on individual particles for more detailed interrogation by infrared spectroscopy, if necessary.

The 8700 LDIR can also be used for rapid screening to locate particular types of plastics using individual wavelength images. The laser can be tuned to a wavelength that is characteristic of a specific plastic, which significantly speeds up the analysis. For example, polystyrene (PS) has a strong band with large contrast between 1490–1508 cm^{-1}. Images can be collected with the laser at the appropriate wavelengths in order to screen specifically for PS. A 5 mm x 5 mm region can be screened in just two minutes, which is a dramatic improvement in speed. This is a major advantage of the tunable laser in the 8700 LDIR system. In addition to the speed of data collection, this targeted method reduces the need for intensive sample preparation due to the specificity of acquisition.

FTIR vs. LDIR

State-of-the-art FTIR imaging uses an incandescent light source with an array detector. Every part of the selected region is measured, including pixels where particles are absent. The region of interest is divided into a mosaic, then each segment is scanned, and all of the pieces of data are electronically stitched together to create a complete image. The application described in Figure 4 included the analysis of filtered sediment from a retention pond using FTIR. The 10 mm × 10 mm area was broken up into a 16 × 16 mosaic and spectra were collected every 5.5 µm. After three hours of data collection, 4.2 million spectra were generated for 871 particles. The 33 GB data file took eight hours to process, resulting in a total of 11 hours expended for the complete study.

An analogous experiment using LDIR takes approximately two hours, including data analysis. The tremendous improvement in ef ficiency stems from the LDIR data collection strategy, as well as its strong signal from the QCL laser. The instrument only collects data where particles are present and swiftly produces one spectrum per particle. Thus, LDIR produces the same reliable spectra as FTIR but utilizes more judicious acquisition.



- Imaging FTIR acquires many spectra in parallel using an array detector.
- Example analysis of filtered sediment from a wet retention pond in Denmark[1]:
  - 16×16 mosaic using a 128×128-pixel array
  - A spectrum every 5.5 µm over 10×10 mm^2
  - 3 hours collection + 8 hours data processing (custom software)
  - 33 GB of data
  - 4.2 million spectra
- … for only 871 particles

Figure 4. How does LDIR compare to state-of-the-art FTIR?

Conclusion

The accurate identification and quantification of microplastic particles in terms of chemical identity, size, and shape is of growing interest. The Agilent 8700 LDIR offers a streamlined approach that accelerates microplastics analysis to deliver real-time results in only a few seconds per particle. Relative quantitation of sample constituents is achieved without complex method development and statistical data is automatically generated for fast compositional assessment. Minimal instrument interaction is required, as simple load-and-go methods with high automation make operation simple even for nonexperts. The targeted analysis strategy facilitates imaging of a large sample area much quicker than any other technique. The breakthrough technology of the 8700 LDIR system has eliminated the burden of microplastics analysis and substantially modernized the workflow.

For More Information

To find out more about this system, visit www.agilent.com/chem/8700-ldir.

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