Application Note

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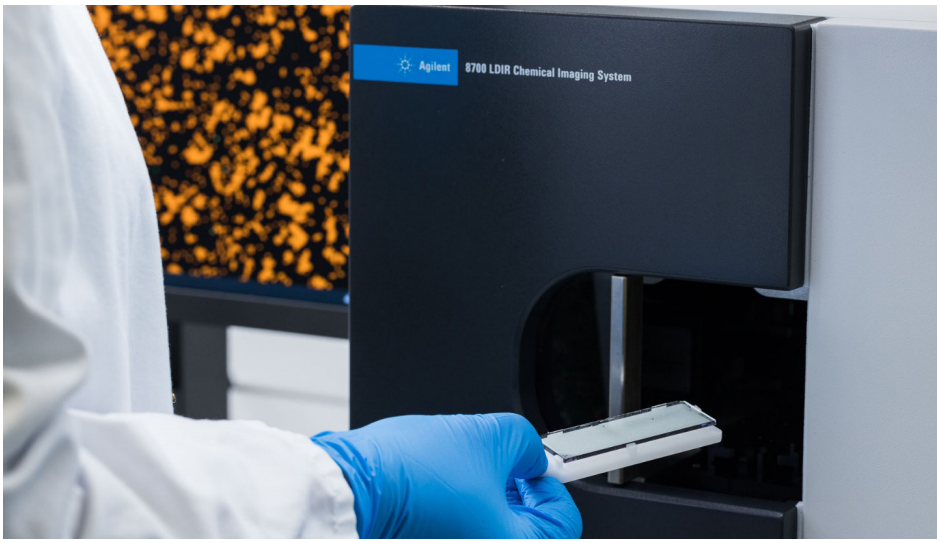
Microplastic Analysis Using the Agilent 8700 Laser Direct Infrared (LDIR) Chemical Imaging System

Accurately distinguishing between polyethylene microplastics and magnesium stearate



Authors

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Abstract

To understand the impact of microplastics on the environment and food chains, accurate and proper characterization of these particles is mandatory.[1] But distinguishing between polyethylene and magnesium stearate, a nonpolymer compound commonly used in food, cosmetics, and latex gloves, using spectroscopic techniques can be a challenge. Samples may contain both polyethylene microplastics and stearates, and there are often only marginal differences in spectra. It can be difficult to differentiate among molecules containing long-chain hydrocarbons, leading to potential false-positive identification.[2,3] This application note demonstrates how the Agilent 8700 Laser Direct Infrared (LDIR) chemical imaging system can overcome this challenge and provide accurate microplastic analysis.

Introduction

Microplastics pollution in waterways is a pervasive issue, creating hazards for wildlife, drinking water, and food systems. But conventional spectroscopic techniques are often inadequate to classify pollutants, a key step in determining their ecological impact. At a molecular level, polyethylene microplastics—which come from diverse sources including personal care products, shopping bags, and other items[4]—can look nearly identical to magnesium stearate, a water-insoluble powder widely used as an additive in a variety of household products.

In this application note, the automated microplastics analysis workflow within Agilent Clarity software was used with the 8700 LDIR chemical imaging system (Figure 1). The scanning mode was first used to rapidly scan the sample area at a single wavenumber (1,442 cm^{-1}) to generate an infrared image. This image was used to both locate particles in the area and describe their size and shape. Once each particle was located, the LDIR then rapidly and automatically moved to each and acquired a full spectrum. The spectrum was then immediately compared to a microplastics spectral library. The best fit match for the spectrum was determined and reported for each particle. The library was derived from well-established sources and included a range of spectra relevant to the analysis of microplastics (e.g., core polymers and natural materials present in samples and minerals).[5, 6]

Experimental

Commercially available clear polyethylene microspheres (Cospheric LLC, CPMS-0.96 38-45 µm–5 g) and magnesium stearate (MilliporeSigma, 415057-25 g, CAS Number: 557-04-0) samples were obtained for the study. Examples of each particle type are shown in Figure 2.

Each sample was uniquely shaped and sized, which made them easily distinguishable from each other and other contaminants, as shown in Figure 2.

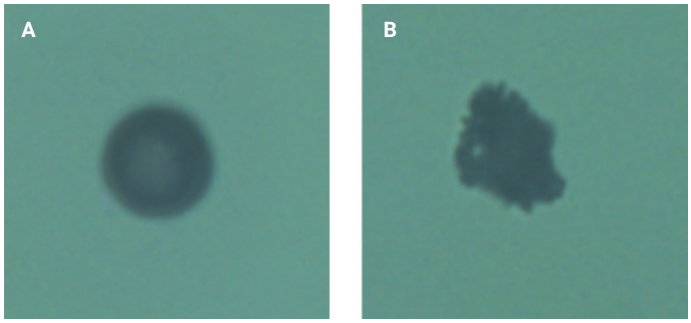

Figure 2. (A) Clear polyethylene microsphere. (B) Magnesium stearate. Both images were obtained using the high-magnification visual camera of the Agilent 8700 LDIR chemical imaging system.

Figure 1. The Agilent 8700 LDIR chemical imaging system allows the high-speed routine analysis of microplastics, including the number of particles present in the sample, their size, and chemical composition.

To prepare samples for analysis, approximately 2 mg of both materials were suspended in absolute ethanol (5 mL) and deposited on a low-e infrared reflective glass slide (7.5 × 2.5 cm, MirrIR, Kevley Technologies). The ethanol was allowed to evaporate at room temperature before analysis (approximately 2 minutes). The automated particle analysis workflow contained within the Clarity software was used for all samples. This workflow sets all the necessary instrument settings automatically, including scan speed, sweep speed, and attenuation. These settings cannot be altered. An analyst can adjust the default settings for several options, including the sensitivity of the particle detection system. The analyst may also set their own hit quality index ranges. Hit quality describes how closely the spectrum of the sample matches that in the reference library. For this experiment, classification ranges (i.e., the characterization of spectral match quality by high, medium, and low) were set to:

- low confidence 0.65 to 0.75
- medium confidence 0.75 to 0.85, and
- high confidence 0.85 to 0.99.

Any particles falling outside this range, i.e., <0.65, were classified as "undefined."

The minimum particle size was set to 20 µm and the maximum particle size to 500 µm as the default setting. A starter library of microplastics (provided with the LDIR) was used to perform the analysis.

Results and discussion

To determine the ability of the LDIR to distinguish polyethylene from magnesium stearate, the samples of polyethylene and magnesium stearate were first assessed separately, then mixed.

Polyethylene microspheres

The first step was to assess the spectra obtained for each polyethylene microsphere sample against the spectral library provided with the Clarity software. In a scanned area (2.88 × 2.83 mm) containing a total of 39 particles, 39 (100%) particles were identified as polyethylene using the automated workflow (Figure 3). All particles were identified as polyethylene with high confidence (hit quality index >0.85), as shown in Figure 4B. The particle analysis workflow generates a statistical overview of particle diameter (µm) versus count automatically at the end of the analysis (Figure 4A). In this analysis, agglomeration of polyethylene microspheres was observed.

Magnesium stearate

In like manner to polyethylene particles, magnesium stearate spectra obtained using LDIR were assessed against the same spectral library provided with the Clarity software. A total of 242 particles were detected in a scanned area of 2.88 × 2.84 mm. All particles were identified as magnesium stearate, 234 (96.7%) particles with high confidence and 8 (3.3%) particles with medium confidence (Figures 5 and 6).

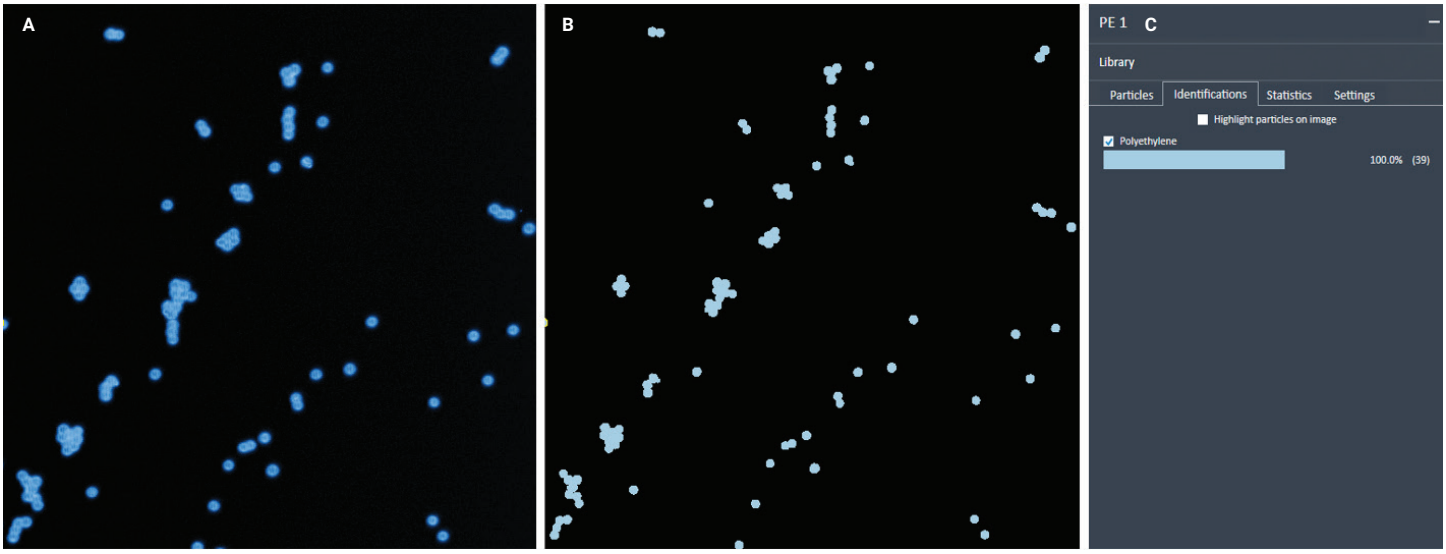

Figure 3. Polyethylene microspheres automated workflow analysis using Clarity software. (A) Infrared image scanned at 1,442 cm^{-1}. (B) Highlights of particles found; the particles are colored based on the identification of the type of microplastic. (C) Automatic statistical data generated based on the identification of microplastics.

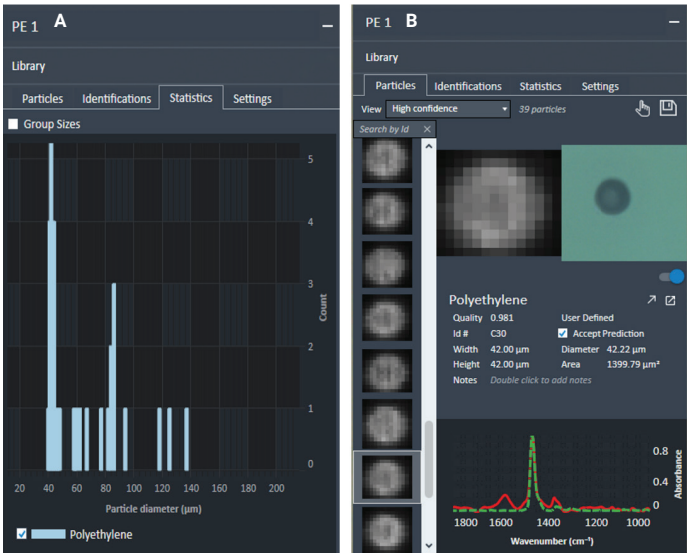

Figure 4. (A) Statistical data of microplastic particles based on various size ranges. (B) Polyethylene particle example; particle information such as infrared image, visible image, hit quality index, size, and overlap of spectrum (solid red line) with matched library spectrum (green dashed line) can be displayed.

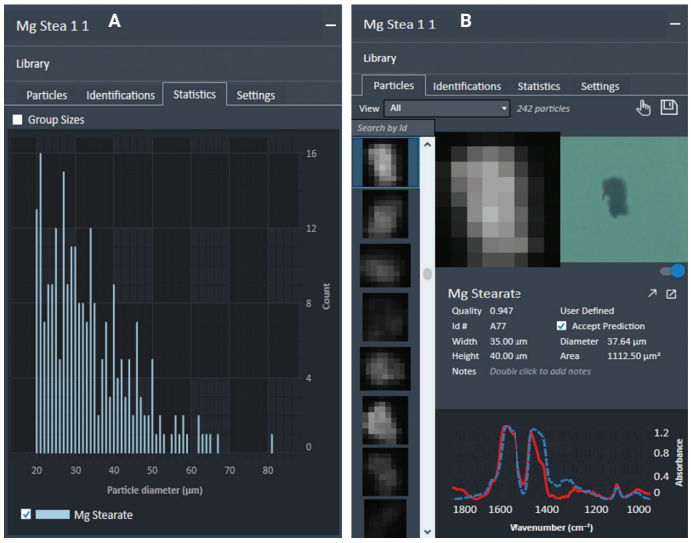

Figure 5. (A) Statistical data of microplastic particles based on various size ranges. (B) Magnesium stearate particle example, particle information such as: infrared image, visible image, hit quality index, size, and overlap of spectrum (solid red line) with matched library spectrum (blue dashed line) can be displayed.

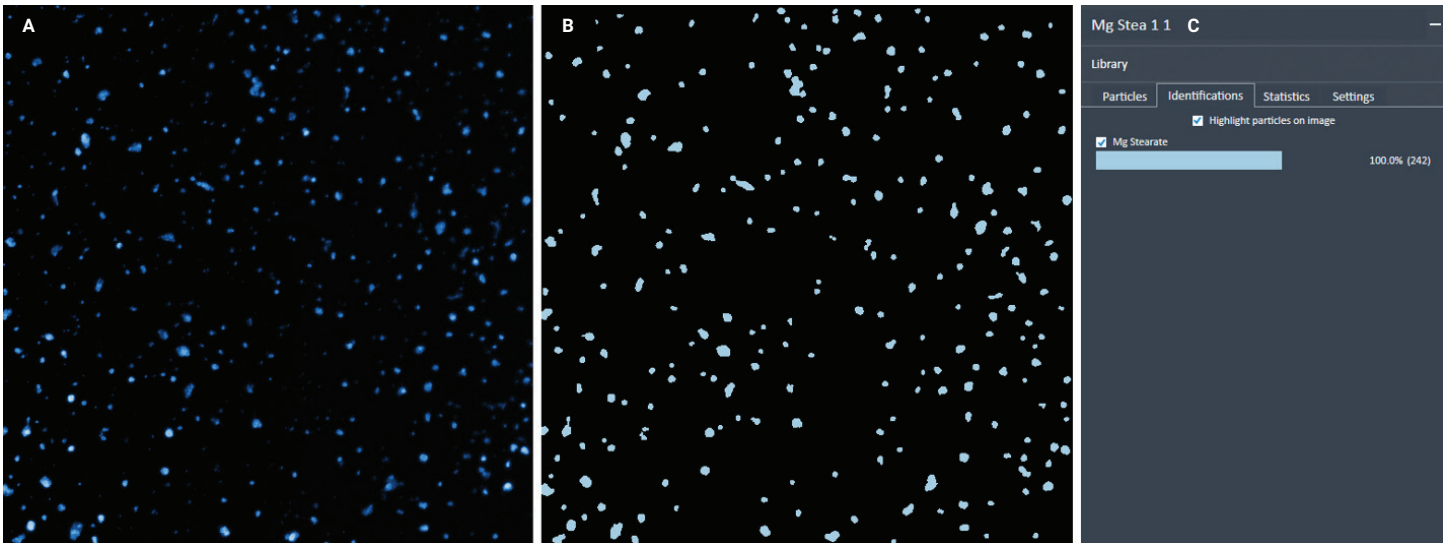

Figure 6. Magnesium stearate automated workflow analysis using Clarity software. (A) Infrared image scanned at 1,442 cm^{-1}. (B) Highlights of particles found: The particles are colored based on the identification of the type of microplastic. (C) Automatic statistical data generated based on the identification of microplastics.

Mixed sample

As a final step in the process to assess the capability of LDIR in distinguishing between polyethylene and magnesium stearate, a mixed sample of both materials was analyzed using the automated workflow. Each type was easily distinguished visually using the built-in high-magnification camera, which allowed system-generated results to be verified, as demonstrated in Figures 4B and 5B. An area of this sample (4.66 × 5.58 mm) was scanned, in which 346 particles were detected. Of these particles, 200 (57.8%) were classified as magnesium stearate and 146 (42.2%) as polyethylene (Figure 7).

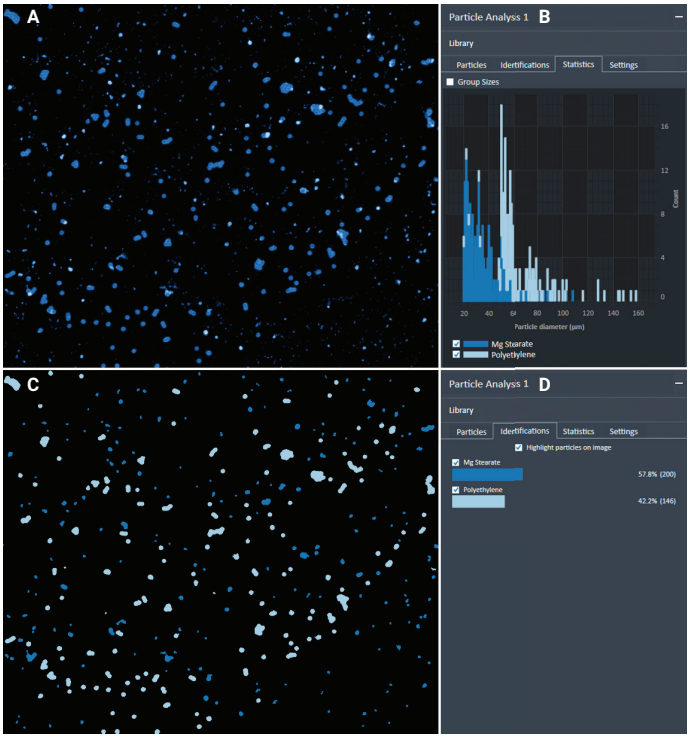

Figure 7. Mixed sample (magnesium stearate and polyethylene) automated workflow analysis. (A) Infrared image scanned at 1,442 cm^{-1}. (B) Statistical data of microplastic particles based on various size ranges. (C) Highlights of particles found; the particles are colored based on the identification of the type of microplastic. (D) Automatic statistical data generated based on the identification of microplastics.

Following full visual verification (manually assessed 346 particles), it was noted that:

1. Agglomerates of magnesium stearate and polyethylene have been formed and been identified as magnesium stearate. This happened due to the LDIR relying on acquiring a single point spectrum from the highest absorbance point (magnesium stearate) within the agglomerate.
2. Other than agglomerates, no particles of either magnesium stearate or polyethylene were visually identified that had not been classified correctly; i.e., there were no false negatives.

Typical spectral region of interest of polyethylene is the C-H bending at 1,480 to 1,440 cm^{-1}. Both polyethylene and magnesium stearate showed this absorbance band. However, magnesium stearate had another characteristic strong signal in the region of 1,500 to 1,660 cm^{-1} (Figure 8). Since Clarity software uses first derivative spectral treatment as a matching algorithm, the absorbance band at 1,500 to 1,600 cm^{-1} helped with accurate identification of magnesium stearate and clear differentiation from polyethylene.

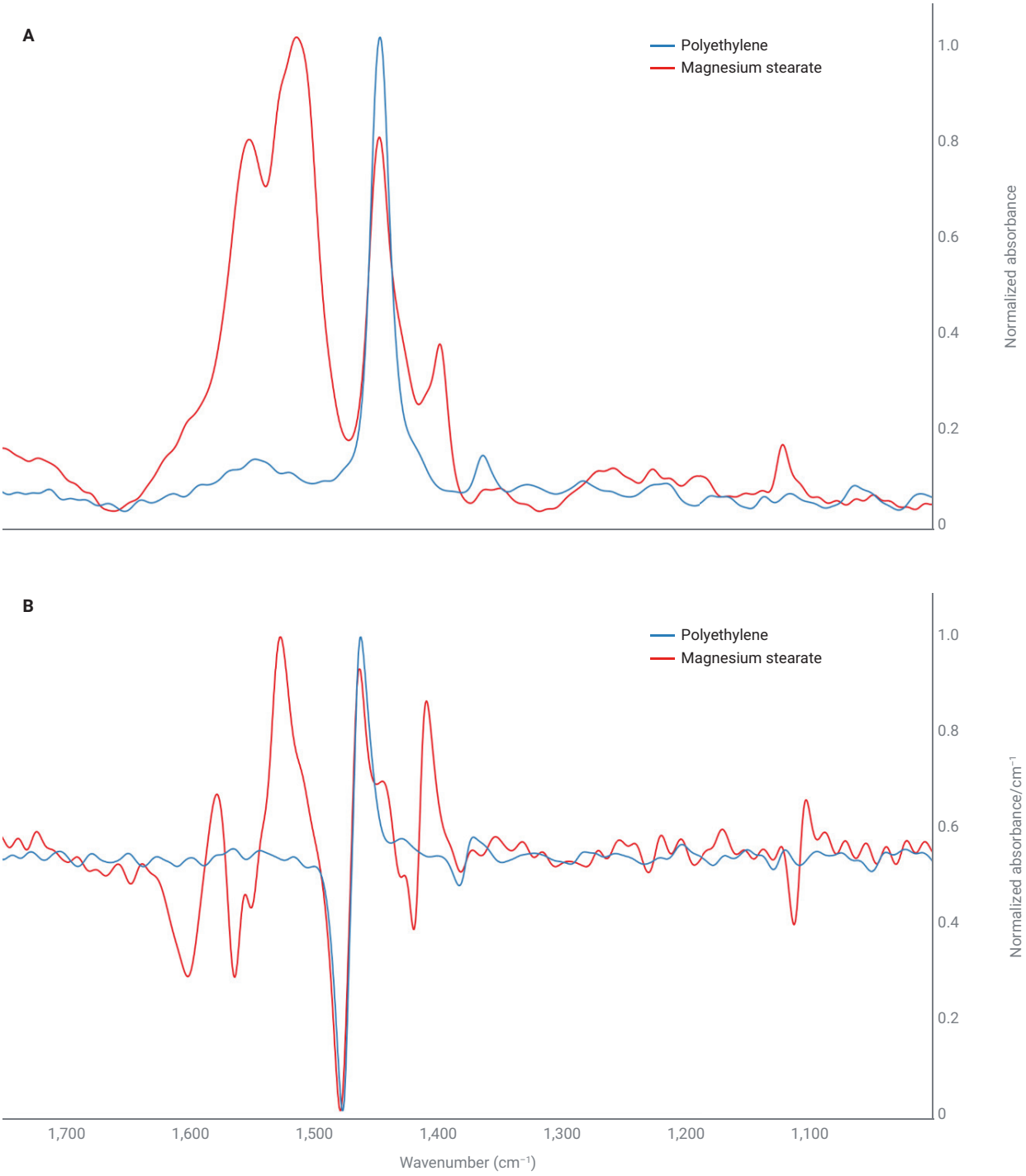

Figure 8. An overlay of polyethylene spectrum (blue) with magnesium stearate (red). (A) normalized absorbance; (B) first derivative.

Conclusion

Many synthetic and natural microparticles exist in the environment. Laboratory results can be confusing, as nonpolymers can act as a potential source of contamination. This application note demonstrates that the Agilent 8700 LDIR could successfully classify and differentiate magnesium stearate from polyethylene in a mixed sample of both. LDIR hardware and software capabilities ruled out misinterpretation of magnesium stearate and polyethylene in the automated particle analysis workflow. The fully automated particle analysis method within the Agilent Clarity software is also an efficient way for users to obtain information on particle sizes, distribution, and identification of microplastics.

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