Application Note

Environmental

Accurate Microplastic Analysis of Bottled Drinking Water

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

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Abstract

Microplastics are defined as small plastic particles between 1 µm and 5 mm in size. Due to poor waste management and plastic pollution, they are now documented to be ubiquitous in the environment.[1,2] However, pathways of dietary exposure to microplastics are not yet fully understood. The consumption of bottled water has been calculated to increase by 7% annually, with an estimated mean total global consumption of bottled water to be 513 billion liters by 2025.[3] This study demonstrates how the Agilent 8700 Laser Direct Infrared (LDIR) Chemical Imaging System can accurately identify and quantify the presence of microplastics in bottled drinking water. This study also shows the capability of the Agilent Cary 630 FTIR spectrometer to investigate the source of microplastic contamination.

Introduction

With the exponential increase in plastic production, there has been a related rise in plastic pollution of the environment.[4] Most plastics break down into smaller fragments (also known as microplastics), allowing them to be more easily ingested. Recent studies in which 259 bottled drinking water samples were processed, 93% showed some sign of microplastic contamination.[5] Typically, plastic water bottles are made from polyethylene terephthalate (PET) or recycled PET, with either a polypropylene or polyethylene cap.

In this application note, the automated microplastic analysis workflow within Agilent Clarity software and LDIR (Figure 1) were used to characterize microplastics present in two commercial brands of bottled drinking water. To investigate the source of contamination, the Agilent Cary 630 FTIR spectrometer with a diamond attenuated total reflectance (ATR) module (Figure 2) was used to identify the type of polymer for each brand (bottle and cap).

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, and their size and chemical composition.

Figure 2. The Agilent Cary 630 FTIR spectrometer with a diamond ATR module.

Experimental

Two commercially available bottled drinking water brands (Figure 3) were purchased from a local supermarket.

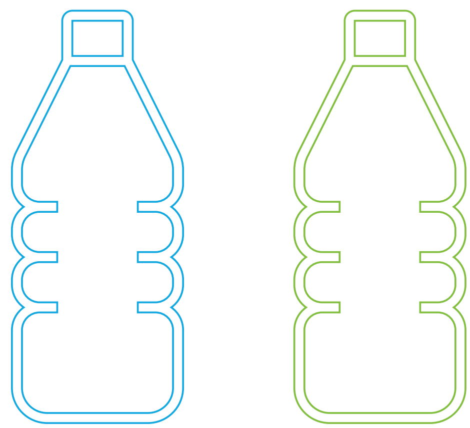

Figure 3. Details of the bottled drinking water brands used in this study.

Sample preparation

The two bottles of drinking water (600 mL) were filtered through Sterlitech polyester (PETG) gold-coated membrane filters, 0.8 µm, 100/0 nm coating, 25 mm. To ensure that all present microplastic particles were collected, the inside of the bottle and funnel was rinsed once using microplastic-free water. To prevent the gold-coated membrane filter from deforming, a small-pore glass frit vacuum filtration was used. As the small-pore glass frit vacuum filtration exhibits a high pore density and solid structure, it ensures that the vacuum pressure spreads more evenly. Once the filtration was complete, the gold-coated membrane was carefully removed using tweezers and placed evenly on the slide holder. The gold ring was then tightened to ensure a uniformly flat filter surface, as shown in Figure 4.

8700 LDIR Chemical Imaging System

An Agilent 8700 LDIR Chemical Imaging System controlled using Clarity software was used in this study. The two polyester (PETG) gold-coated membrane filters loaded with microplastic particles from each sample were analyzed by the Agilent 8700 LDIR using the fully automated Particle Analysis method in the Clarity software. The method setup parameters used for data acquisition are shown in Table 1. Instrument parameters were all set to the instrument default settings.

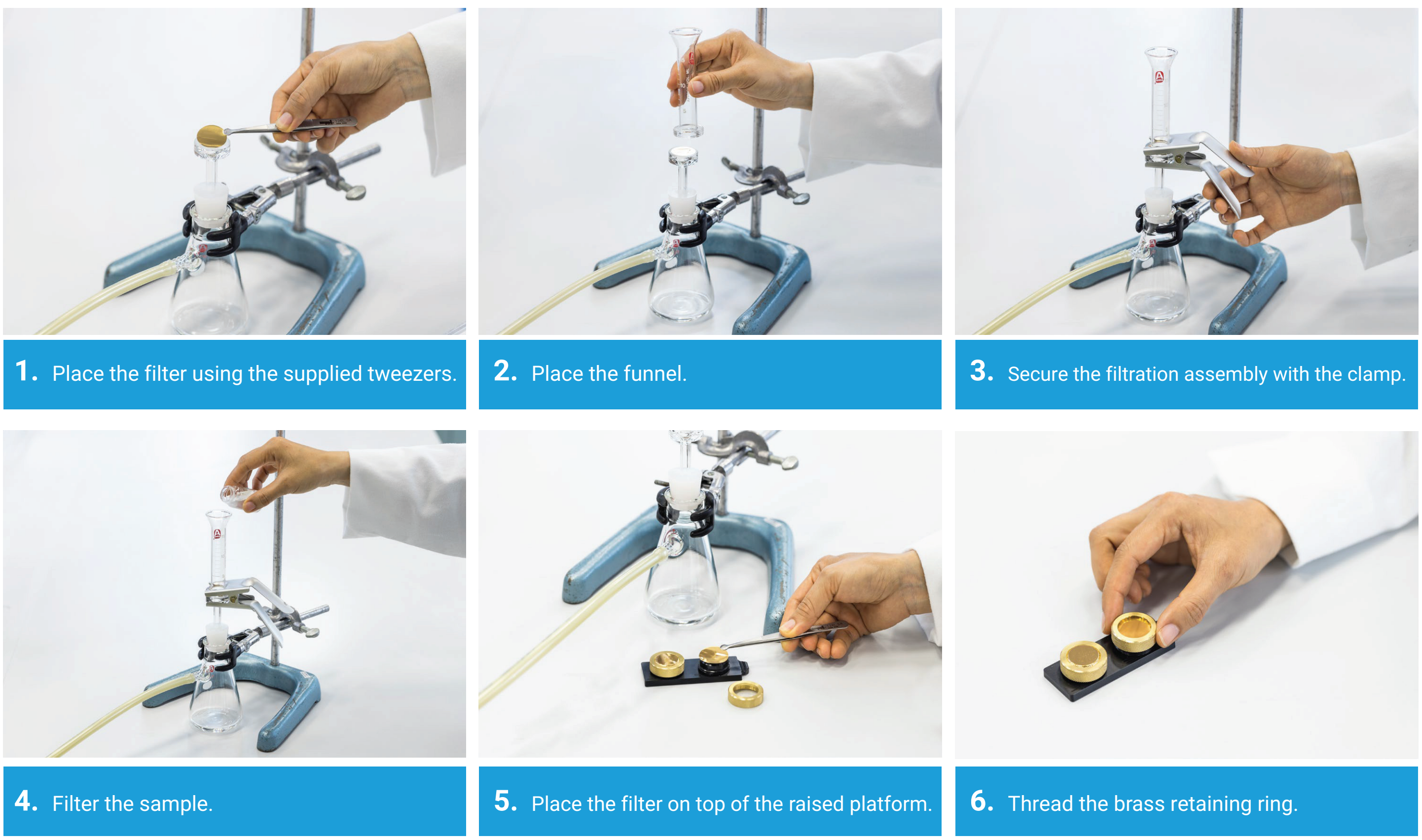

Figure 4. Sample filtration equipment and steps for the preparation of samples for LDIR on-filter analysis.

Cary 630 FTIR with an ATR module

To investigate the source of microplastics found in bottled drinking water, a Cary 630 FTIR spectrometer with a diamond ATR module was used. Samples (~5 mm) of both the bottle and the cap of each brand were measured directly using the ATR, and were identified via a user-generated library of polymers. This spectral library contains ATR spectra of the most common polymers used in the plastic industry. This library was developed using the Polymer Sample Kit (Scientific Polymer Products, Inc.; catalog number 205; LOT number 600801012), which includes polystyrene, polypropylene, high- and low-density polyethylene, polyethylene terephthalate, polyvinyl chloride, polycarbonate, poly (methyl methacrylate), polyoxymethylene, polyamides, and polytetrafluoroethylene. The library search method was used by applying the Similarity search algorithm using the parameters shown in Table 2.

Results and discussion

The Clarity software Particle Analysis method used both LDIR scan and sweep modes. Scan mode was used first to rapidly scan the sample selected area (~16 mm in diameter for each filter) at a single wavenumber to both locate particles and determine each particle's boundary (Figure 5). Once particles were located, the LDIR then rapidly and automatically moved to each particle and acquired a full spectrum over the wavelength range (sweep mode through the midinfrared fingerprint region). The spectra were then compared to the Microplastics Starter 2.0 spectral library in real time. The best fit match for the spectrum was determined and reported for each particle.

For brand A, a total of 98 particles were detected, however only four microplastics were detected with a hit quality index of >0.8 (size range 20 to 100 µm, Figure 6). The remaining particles were either plastic polymers with a hit quality index <0.8 or non-microplastic particles such as naturally occurring polyamides and cellulosic materials. For brand B, more particles were detected (a total of 1,112), with only 33 identified as microplastics (size range 20 to 703 µm) using the reporting criteria mentioned previously.

Table 1. Parameters used for the Agilent 8700 LDIR Chemical Imaging System automated method analysis of microplastics.

Parameter	Setting
Method	Particle Analysis
Library Used	Microplastics Starter 2.0[6,7]
Auto Scan	On
Collect Visible Images	Yes
Particle Sensitivity	Automatic
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) – High confidence (0.85 to 0.99) Any particles falling outside this range (i.e., <0.65) were classified as "undefined"
Size Classification Range (µm)	0–30 30–50 50–100 100–200 200–300 300–500 >500
Scan Speed	Default (8)
Sweep Speed	Default (3, high speed)
Focus Offset	0
Polarization (Degree)	Default (0)
Attenuation (%)	Default (0)/Auto

Table 2. Agilent Cary 630 FTIR–ATR operating parameters.

Parameter	Setting
Method	Library search
Library Used	User-generated polymers library
Search Algorithm	Similarity
Spectral Range	4,000 to 650 cm^{-1}
Background Scans	64
Sample Scans	64
Spectral Resolution	4 cm^{-1}
Background Collection	Air
Color-Coded Confidence Level Thresholds	Green (high confidence): >0.97 Yellow (medium confidence): 0.95 to 0.97 Red (low confidence): <0.95

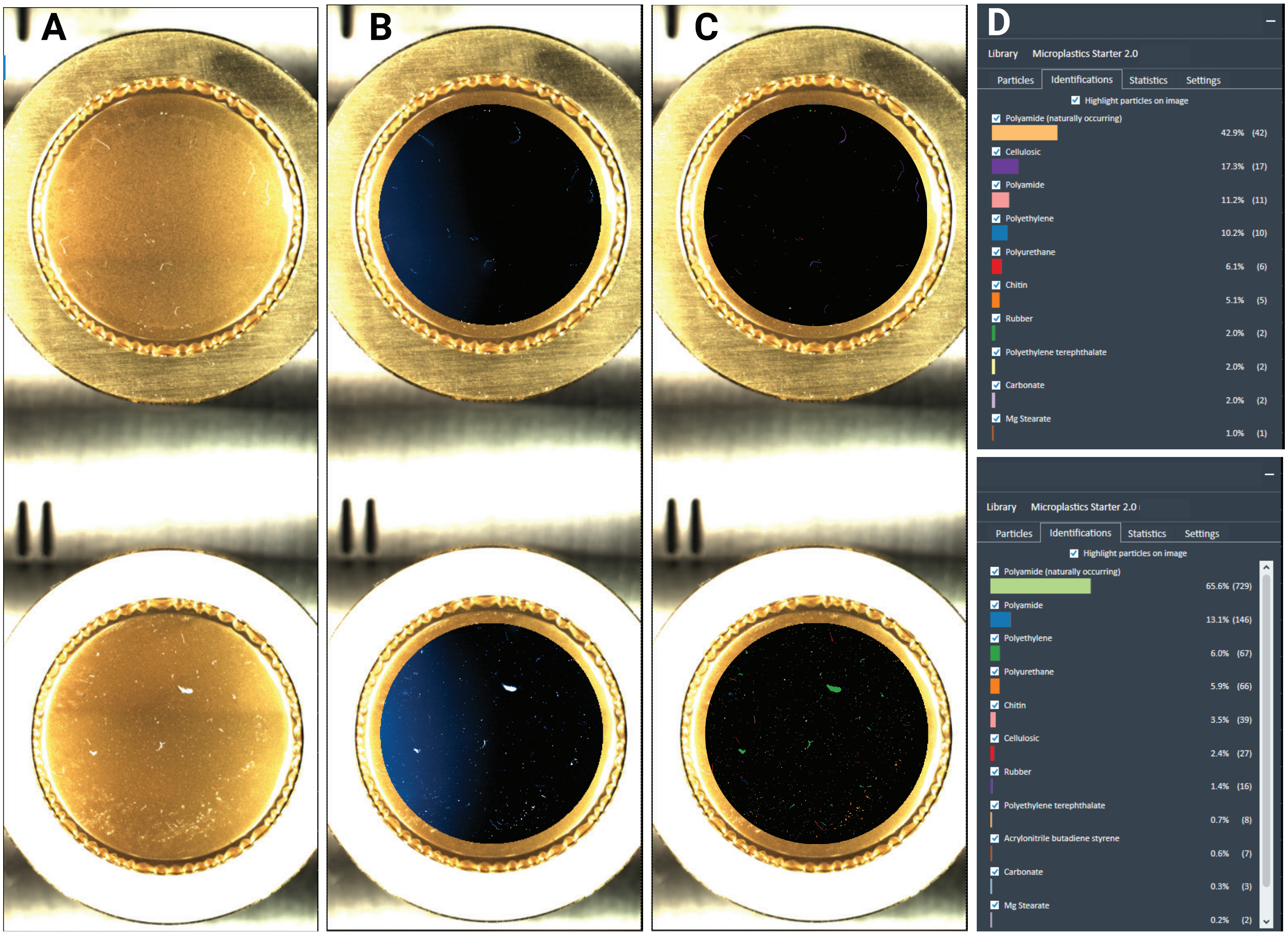

Figure 5. Identification and classification data of microplastics in brands A (top) and B (bottom), analyzed directly on gold-coated polyester membrane filters using the Agilent 8700 LDIR system. (A) Visible image of both filters. (B) IR image scanned at 1,442 cm^{-1} of both filters. (C) Highlights of particles found—the particles are colored based on the identification of the type of microplastic on both filters. (E) Automatic statistical data generated based on the identification of microplastics in both brands.

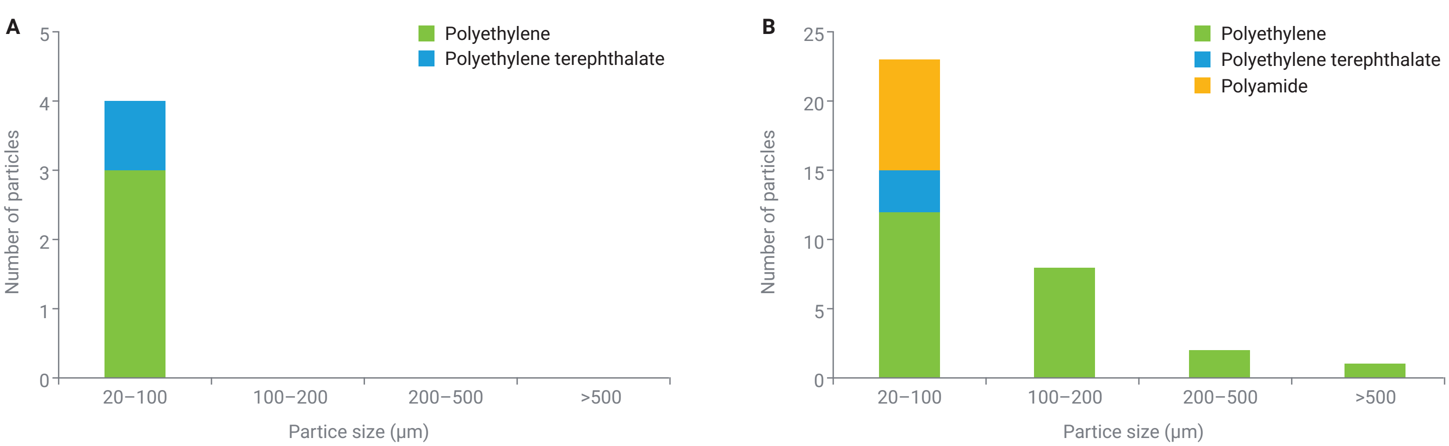

Figure 6. Number of particles, size ranges, and their types identified in (A) brand A and (B) brand B using the Agilent 8700 LDIR Chemical Imaging System.

PE and PET microplastics were identified as contaminants in both brands. Brand A had three PE particles (75%) and one PET particle (25%), and brand B had 23 PE particles (69%) and three PET particles (9%), as shown in Figure 7. In addition, brand B showed contamination with eight particles of PA (24%). Also, non-microplastic particles were detected in both samples with a high quality index (Figure 8).

Microplastic source investigation with Cary 630 FTIR

As described in the experimental section, the Cary 630 FTIR spectrometer with a diamond ATR was used to investigate the source of microplastics present in both brands. The bottle polymer type of both brand A and B was identified as PET, with a hit quality index of 0.98027 and 0.97496 respectively, as shown in Figure 9. Results are color coded based on user-defined confidence level thresholds (see Table 2). While the caps for brands A and B were identified as high-density PE, with a hit quality index of 0.98571 and 0.98914 respectively, as shown in Figure 10. The Cary 630 FTIR spectrometer is controlled using Agilent MicroLab PC software, which uses a pictorial interface to guide users through the steps of the analysis, from sample introduction to reporting (Figure 11).

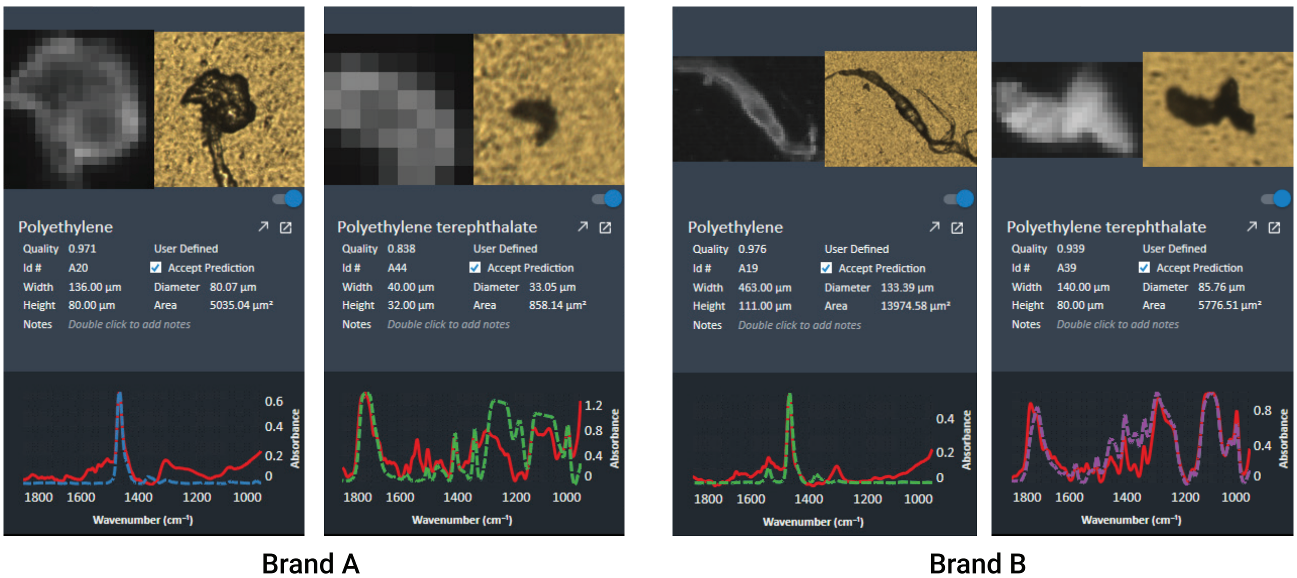

Figure 7. Examples of microplastics (PE and PET) identified in both brand A and B.

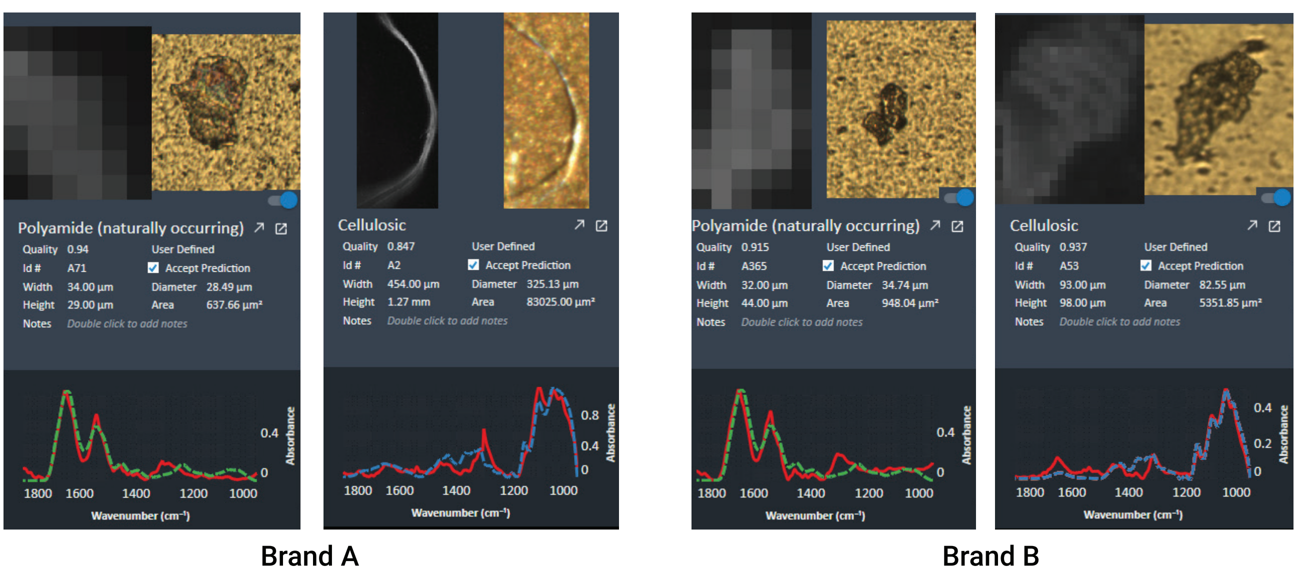

Figure 8. Examples of non-microplastics (naturally occurring polyamide and cellulosic materials) identified in both brand A and B.

Figure 9. Agilent Cary 630 FTIR spectrometer qualitative analysis of bottles (red trace) and library hit (blue trace). The table shows the hit quality and library used, and the hit name for both brand A and B.

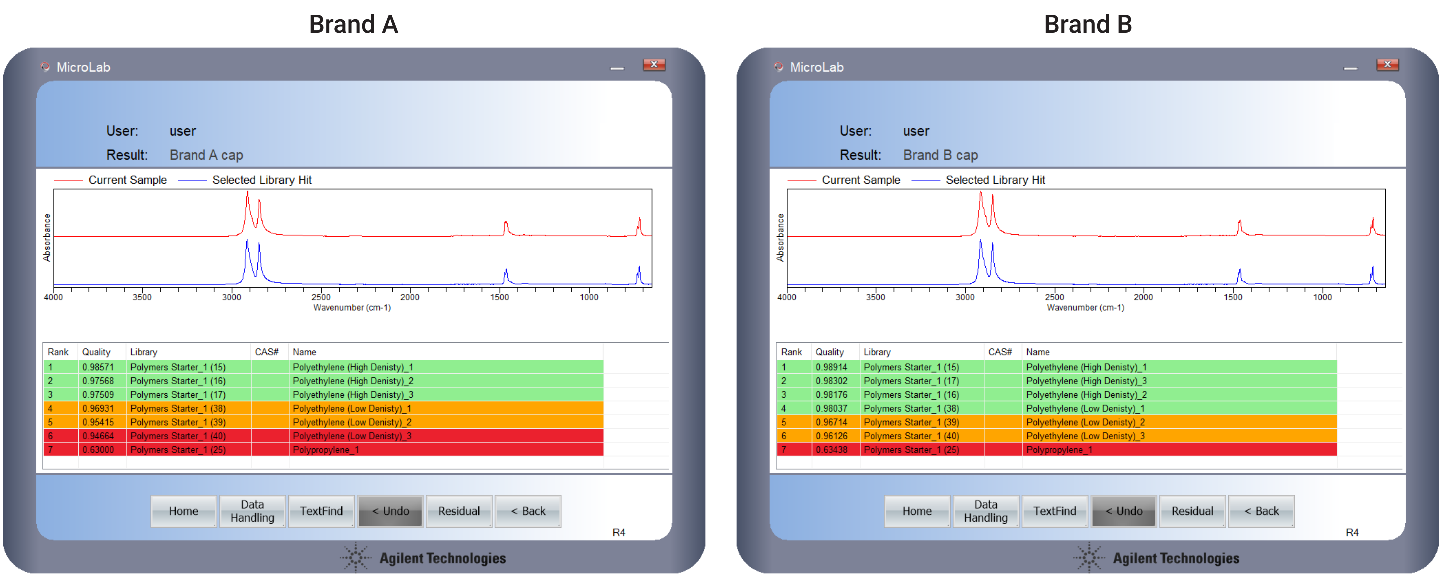

Figure 10. Agilent Cary 630 FTIR spectrometer qualitative analysis of caps (red trace) and library hit (blue trace). The table shows the hit quality and library used, and the hit name for both brand A and B.

1 Start the analysis.

2 Follow picture-driven software guidance.

3 Instantly receive color-coded, actionable results.

Figure 11. The intuitive Agilent MicroLab PC software workflow makes finding answers with the Agilent Cary 630 FTIR spectrometer simple. The picture-driven software also reduces training needs and minimizes the risk of user-based errors.

The ID obtained for bottles and caps for both brands agreed with the data obtained using the 8700 LDIR Chemical Imaging System. Therefore, it can be assumed that the high presence of PE in both samples could be the result of opening and closing the water bottle. This was also reported as a potential source by Samandra *et al.* (2022).[8] The probable source of PA could not be identified, but the source could be the bottle packaging, the source of water, or contamination occurring during sample extraction. However, further investigations should be made to determine its true source.

Conclusion

Using the on-filter analysis capability of the Agilent 8700 LDIR Chemical Imaging System, a total of four and 33 microplastics particles were detected with a hit quality of greater than 0.80 in brands A and B respectively. PE and PET were the most frequently detected polymers in both brands. High levels of identification accuracy and confidence were achieved for PET, PE, and non-microplastic particles on the gold-coated filters.

This work demonstrates that the vacuum filter sample preparation procedures and LDIR methods provided efficient microplastic analysis and significant time saving. The direct-filter LDIR method requires less sample handling, reducing the potential for sample contamination and offering excellent accuracy and higher sample throughput. The automated workflow of the 8700 LDIR Chemical Imaging System will help in accurate characterization of microplastics in different matrices that involve high numbers of samples and fast sample throughput.

Also, the Agilent Cary 630 FTIR spectrometer provided an easy solution for investigating the source of microplastics by performing a simple polymer identification analysis. This application note showed the flexibility of the Cary 630 FTIR for polymer identification, using a sampling module and method tailored to the application. The intuitive, picture-guided Agilent MicroLab PC software is quick and easy to learn, reducing training needs and operator errors, and is easily used to investigate the source of microplastics in various matrices.

References

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Further information

- Agilent 8700 LDIR Chemical Imaging System
- Agilent Clarity Software
- Microplastics Technologies FAQs
- Microplastics Analysis in Water
- Agilent Cary 630 FTIR Spectrometer
- Agilent MicroLab Software
- ATR-FTIR Spectroscopy Overview

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