Application Note

Environmental

Analyzing Colored Microplastics with the Agilent 8700 Laser Direct Infrared (LDIR) Chemical Imaging System

Authors

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Abstract

Plastics are produced in various colors to serve different manufacturing purposes, such as protection, usability, and aesthetic aspects. The presence of dyes/pigments in microplastics analysis using spectroscopic techniques such as Raman microscopy can lead to inaccurate results. This application note demonstrates how the Agilent 8700 Laser Direct Infrared (LDIR) Chemical Imaging System can overcome this challenge with colored polyethylene terephthalate (PET) microplastics. The system provided accurate microplastic analysis that was not compromised by the color of the particle.

Introduction

Widespread plastic pollution has become one of the world's most pressing environmental issues. According to a 2022 report, the amount of global plastic waste will nearly triple by 2060, with 15% leaking into ecosystems.[1] Some of this pollution takes the form of microplastics, tiny particles that can contaminate waterways and food chains.

Understanding the impact of microplastics requires characterizing them properly. But conventional spectroscopic techniques such as Raman microscopy cannot reliably classify colored microplastics, a key step in determining their ecological impact. In a Raman microscope, dyes and pigments can generate sharp peaks and higher intensity compared to the polymers themselves, which can lead to inaccurate identification of microplastics.[2-5] To overcome this issue, time-consuming measurement optimization and library creation for common dyes and pigments is required.

This application note demonstrates that colorants (dyes and pigments) added to polymers have no negative effect on microplastics characterization using the automated microplastics analysis workflow of the 8700 Laser Direct Infrared (LDIR) Chemical Imaging System (Figure 1).

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.

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 wavenumber was selected to ensure accurate detection of microplastics (most plastic polymers absorb at this wavenumber due to the presence of carbon-hydrogen bonds). For the rare polymer types without carbon-hydrogen bonds, the particle detection is based on the scattered light coming back from the particle. This image was used both to 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 match for the spectrum was determined and reported for each particle. In this study, the Microplastics Starter 2.0 library supplied with Agilent Clarity software was used. This 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).[6,7] To verify microplastics identification, an Agilent internal library was used as needed.

Experimental

Commercially available colored polyethylene terephthalate (PET) bottles (brown, white, and cobalt blue 100 mL PET round bottles, product number: BOPBRB100, New Directions Australia) were obtained for the study (Figure 2).

Figure 2. Colored polyethylene terephthalate plastics bottles used in this study.

To prepare samples for analysis, part of each colored PET bottle was ground into a fine powder using a metallic file that is available at most hardware stores. The particles were collected into a vial containing 5 mL of absolute ethanol (Scharlau ET00052500; CAS No: 64-17-5) and shaken vigorously without further processing to create a microplastic suspension. Multiple 10 µL aliquots of the microplastic suspension were transferred onto a low-e infrared reflective glass slide (7.5 × 2.5 cm; MirrIR, Kevley Technologies, Ohio, USA) using pipettes. The slide was then left to dry at room temperature to allow the ethanol to evaporate 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, size range, and size classification. 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 0.80, and

– High confidence 0.80 to 0.99.

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

In this study, the minimum particle size was set to 10 µm and the maximum particle size to about 2,000 µm (to cover agglomerated particles).

Results and discussion

To determine the ability of the LDIR to accurately identify colored PET, samples of each color were assessed separately. Results obtained are summarized in Table 1.

In each area scanned, more than 150 particles were detected. This was followed by accurate identification of colored PET (brown, white, and blue) microplastics (>95%) achieved via LDIR (Figure 3). In addition to the correct identification of those particles, the largest number of particles were identified with high hit quality index (high confidence), at 76.6% (brown PET), 89.7% (white PET), and 84.9% (blue PET). The LDIR high-magnification visible camera helped visually differentiate the colored PET, as shown in Figure 4. LDIR demonstrated that colorants (dyes and pigments) added to PET bottles had no effect on accurately characterizing PET-generated microplastics (Figure 4).

In Raman spectroscopy, the presence of these pigments might increase the fluorescence background and create extra peaks, disrupting the Raman spectrum. LDIR would be advantageous in such a case, since the added colorant signal is less substantial compared to the PET polymer signal, as shown in Figure 5. Colored PET exhibited almost similar spectra, with no additional peaks reflecting the added pigments/dyes.

Table 1. Colored PET results obtained using LDIR.

Sample	Area Scanned (mm)	Total Number of Particles	Particles Identified as PET	Quality of Identification	Figure
Brown PET	19.08 × 19.71	201	193 (96.0%)	High confidence: 154 (76.6%) Medium confidence: 23 (11.4%) Low confidence: 24 (11.9%)	3A
White PET	10.27 × 10.66	156	152 (97.4%)	High confidence: 140 (89.7%) Medium confidence: 10 (6.4%) Low confidence: 5 (3.2%) Undefined: 1 (0.6%)	3B
Blue PET	13.24 × 18.30	319	310 (97.2%)	High confidence: 271 (84.9%) Medium confidence: 32 (10.0%) Low confidence: 15 (4.7%) Undefined: 1 (0.3%)	3C

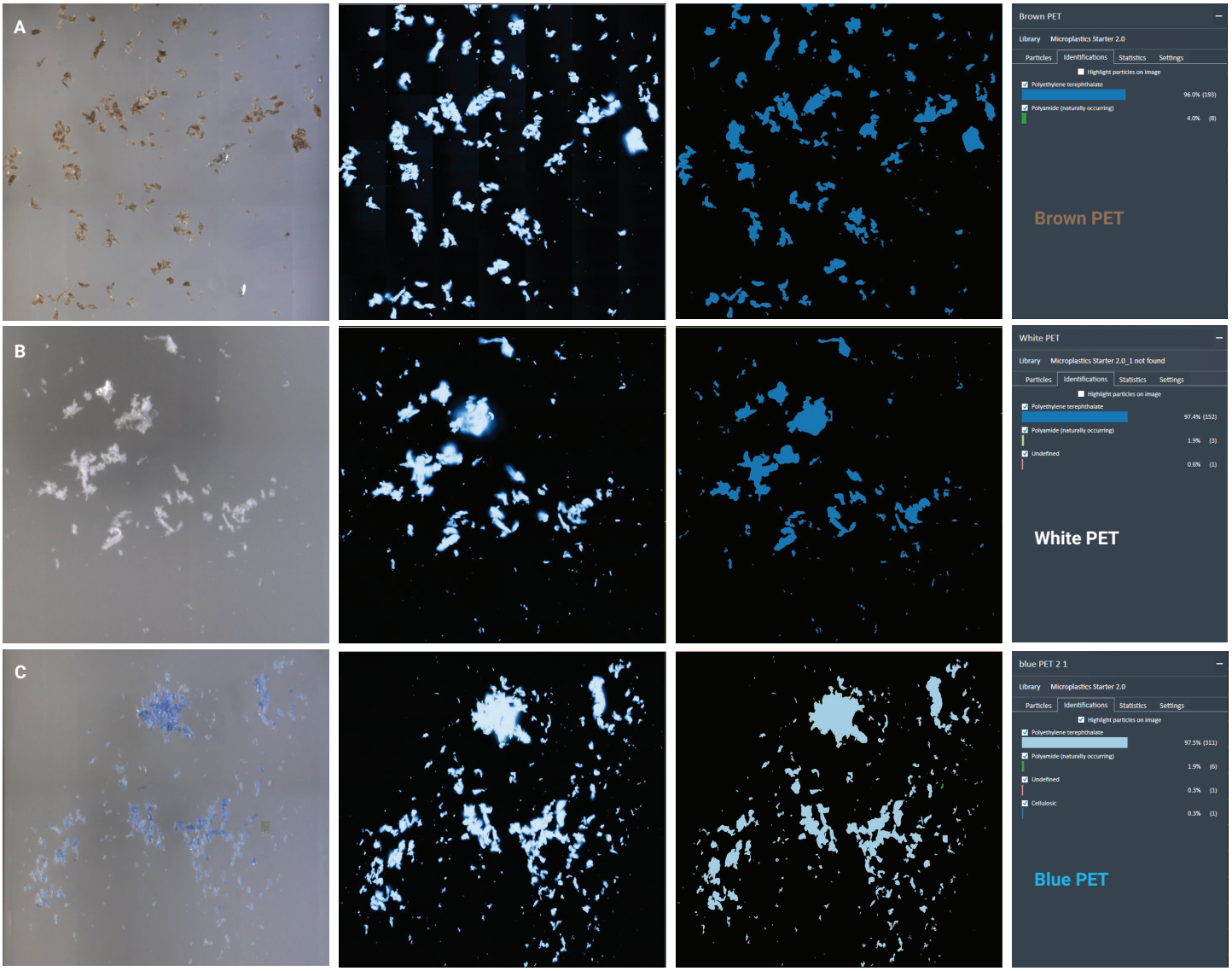

Figure 3. Colored PET microplastics automated workflow analysis using Agilent Clarity software: (A) brown; (B) white; and (C) blue PET. For each color, from left to right: visible image obtained for the selected area; infrared image scanned at 1,442 cm^{-1}; highlights of particles found, based on the type of microplastic identified; and automatic statistical data generated based on the microplastics identified.

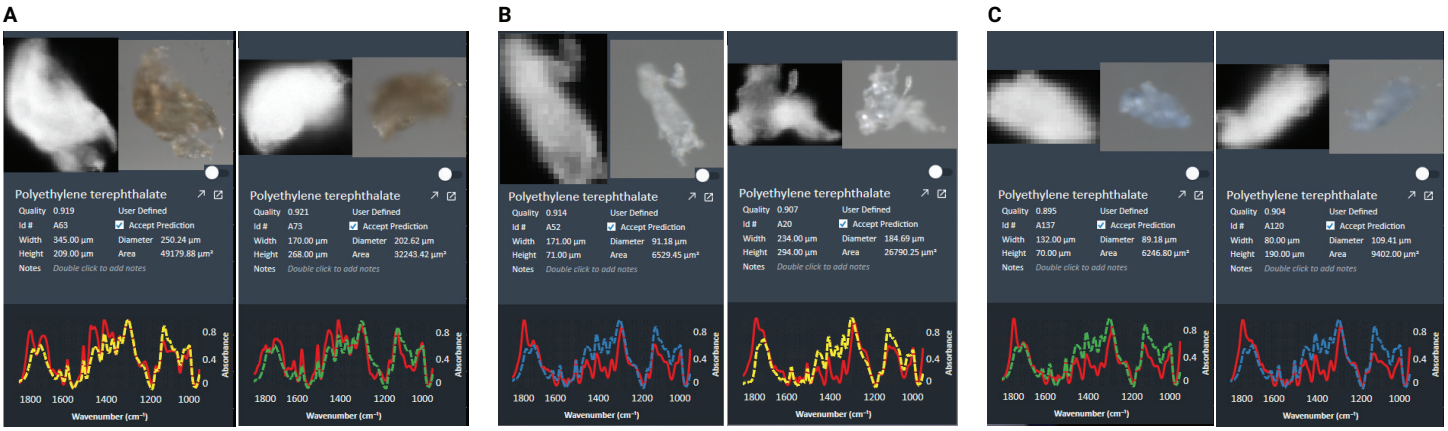

Figure 4. Colored PET microplastics examples: (A) brown; (B) white; and (C) blue PET. Particle information such as infrared image, visible image, hit quality index, size, and overlap of spectrum (solid red line) with matched library spectrum (dashed lines) can be displayed.

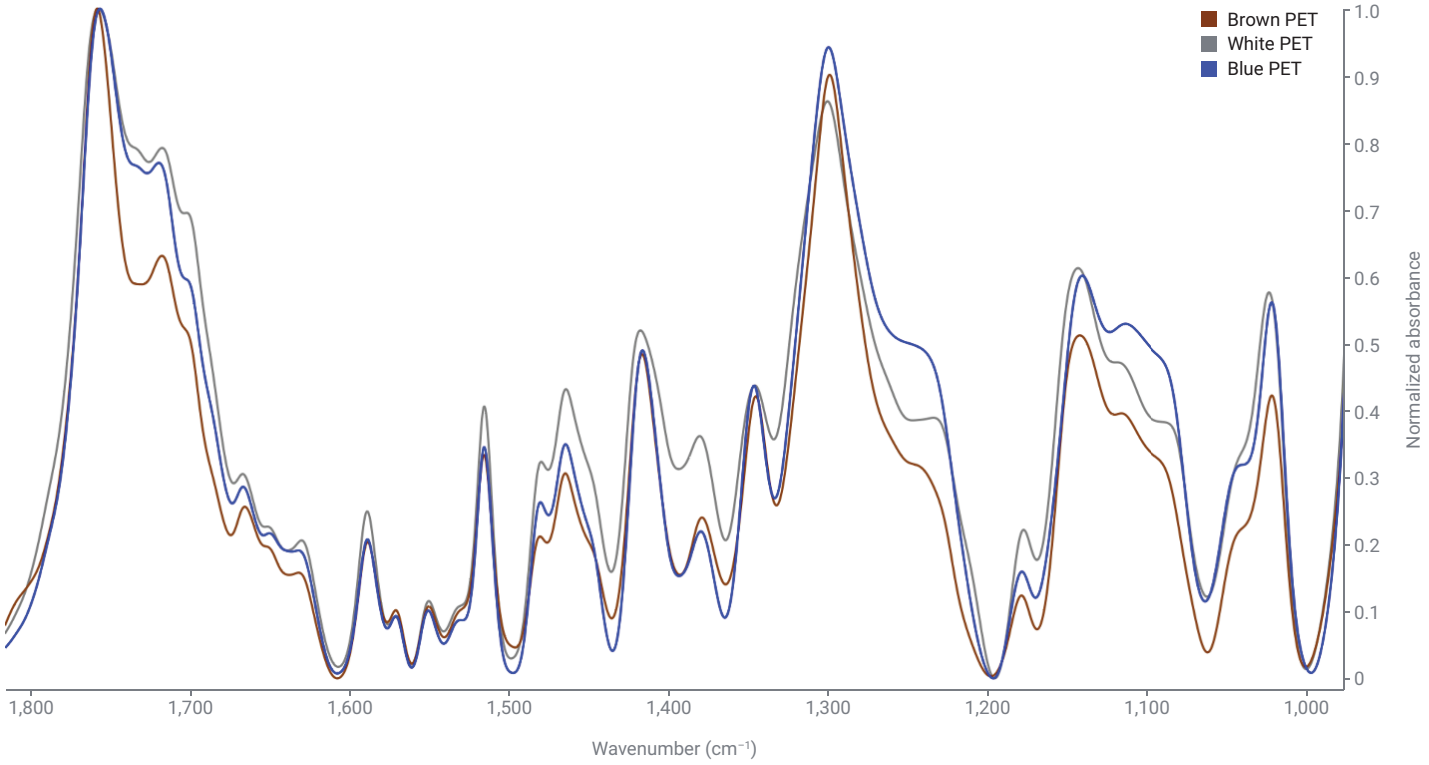

Figure 5. An overlay of colored PET microplastics spectra.

Conclusion

Many colored synthetic microparticles exist in the environment. Added pigments/dyes can introduce challenges to microplastics analysis using Raman spectroscopy (e.g., microplastics misidentification, method optimization, library creation of colorants, and cumbersome processes). This application note demonstrates that the Agilent 8700 LDIR can successfully identify colored PET microplastics. Added colorants (dyes and pigments) had no effect on characterizing PET microplastics accurately. Using this system can save significant time and reduce complication in microplastics analysis. The fully automated particle analysis method within the Agilent Clarity software is also an efficient way for users to obtain information on particle sizes, visible and infrared images for each particle, size distribution, and identification of microplastics.

References

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Further information

- Agilent 8700 LDIR Chemical Imaging System
- Agilent Clarity Software
- Microplastics Technologies FAQs
- Microplastics Analysis in Water

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