APPLICATION NOTE

FTIR Microscopy and Imaging

AUTHORS

Ian Robertson
PerkinElmer,Inc., Seer Green, UK

Ella Gardner
PerkinElmer, Inc., Seer Green, UK

FT-IR Imaging Analysis of Microplastic Test Sample

Introduction

The widespread use of plastic materials in human society has led to a huge global presence of microplastics in the environment including the air, soil and water. As microplastics are becoming more of a major environmental pollutant they are receiving an increasing amount of interest from researchers and the public.

Plastics are the most prevalent type of marine debris found in the oceans.[1] Plastics come in all shapes and sizes but microplastics are defined as being any plastic material greater than 1 micron and less than 5 millimetres in size. Almost 400 million tons of plastics are produced every year.[2] Unfortunately, this problem is only going to grow because with an estimated mass of 5 billion tons of plastic in landfill and the environment that will break down over time it means the microplastic levels keep rising.[2] A variety of testing methods have been adopted to determine the prevalence and sources of microplastics contamination in the environment.

FT-IR and FT-IR microscopy have been adopted as standard methods for the detection and identification of microplastics from a wide range of environments and in a wide range of sample matrices. This paper describes the FT-IR microscopy analysis of a standard test sample developed for an interlaboratory study.[3]

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Sample Preparation for IR Microscopy

Using IR microscopy to analyze a range of microplastics requires clean samples avoiding sample matrix interference and the individual particles to be separated from the matrix. Sediment samples usually need a sample cleanup process but in this case a test sample tablet described below was dissolved and the microplastics that had been contained within the tablet were separated from the aqueous matrix. This was achieved by simple filtration of the sample using an appropriate filter that is suitable for IR measurements. Gold-coated filters are the most suitable for IR reflectance measurements. Aluminium oxide (Anodisc) filters can be used for transmission measurements.

Experimental

A test sample tablet was sourced from the Norwegian Institute for Water Research (NIVA) consisting of a mixture of sodium hydrogen carbonate ($NaHCO_3$), citric acid ($C_6H_8O_7$) and a binder (lactose) spiked with a mixture of microplastics. This was dissolved in 200 mL of pure water and then vacuum filtered using a 600 mL Advantec flask fitted with a 13 mm glass filter holder. A 13 mm Anodisc membrane filter (Sterlitech Corp.) with a pore size of 0.2 micron was used to retain the microplastics on the filter. The 13 mm filter was placed directly into the IR microscope sample slide holder of the Spotlight 400 (Figure 1). A Visible Image Survey of the entire filter was recorded followed by IR imaging transmission measurement using the MCT array detector.

Figures 2A and 2B show the complete visible image surveys, using reflectance (2A) on 13 mm diameter gold coated polycarbonate filter and transmittance (2B) illumination of the filtered test sample on 13 mm diameter Anodisc filter.

Figure 1. PerkinElmer Spotlight 400 FT-IR Imaging system.

A range of dark particles can be observed distributed across the filters.

After a visible image survey was collected the filter was imaged at 8 cm^{-1} using 25 microns pixel size. A complete image of the 13 mm filter can be achieved in as little as 40 minutes. The average absorbance image obtained from the test sample is shown in Figure 3.

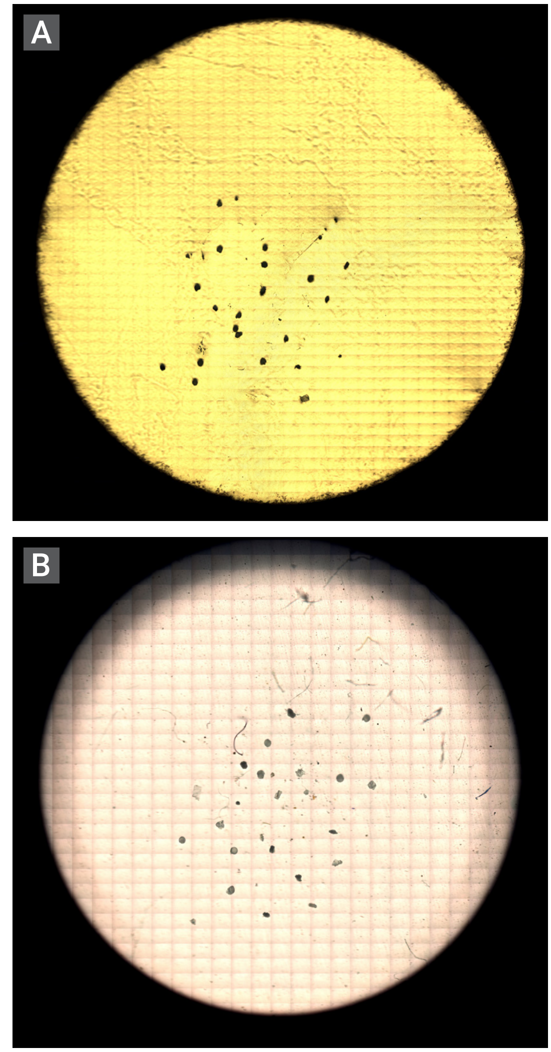

Figure 2. (A) Visible survey of test sample filtered onto gold-coated polycarbonate filter using reflectance. (B) Visible survey of test sample filtered onto Alumina oxide membrane filter in transmittance.

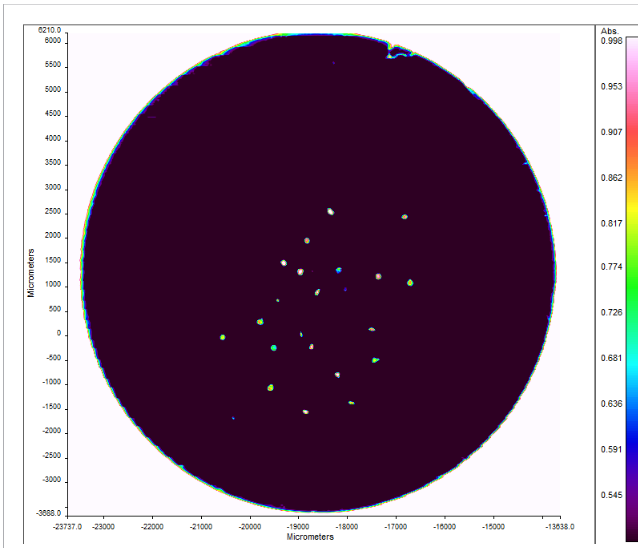

Figure 3. Average Absorbance image for test sample tablet dissolved in pure water.

The samples were initially analyzed, as filtered, on the Anodisc filter. The particles were then manually transferred onto the gold coated polycarbonate filter to allow for reflectance measurement with an increased spectral range. The Anodisc filters can only measure down to 1250 cm^{-1} due to the spectral characteristics of the filter itself, thereby missing many spectral features of the microplastic materials. The reflectance measurements on the gold coated polycarbonate filter offer the full spectral range down to 700 cm^{-1} using the MCT detector. Many laboratories standardize on transmission measurements using the Anodisc filters, others prefer the full spectral range of the gold coated polycarbonate (or alternatively silicon) filters. Figure 4 shows the spectra of polystyrene obtained from the two different filters with significant additional spectral information in the spectrum obtained from the reflectance measurement on gold coated polycarbonate compared to the transmission measurement on the Anodisc filter.

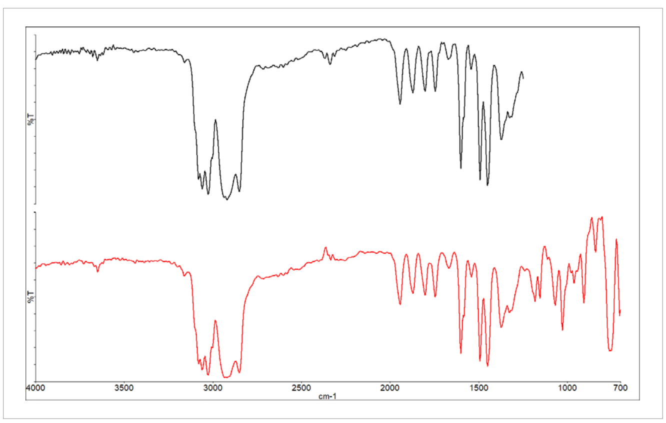

Figure 4. Polystyrene spectra obtained from Anodisc (top) and gold coated polycarbonate (bottom) filters.

Four main polymer types were found from inspection of the spectra of the particles observed in the average absorbance image, identified as polyethylene (PE), polystyrene (PS), polyvinyl chloride (PVC) and polyethylene terephthalate (PET).

The IR image is based on the collection of nearly 270,000 spectra. Manually sorting through the data to find individual chemical species would take hours. However, data processing routines allow for rapid extraction of information. The "Show Structure" command in the Spectrum Image software uses Principal Components Analysis (PCA) to extract the information for the different chemical types present within the data collected.

Figure 5 shows the overall mixed scores plot and then Figures 6-9 show 4 single Scores plots. Score 1 is PE, Score 2 is PS, Score 3 is PVC, and Score 4 is PET. Each score shows clear isolated particles from the total of the particles seen in the Total Absorbance plot shown in Figure 3.

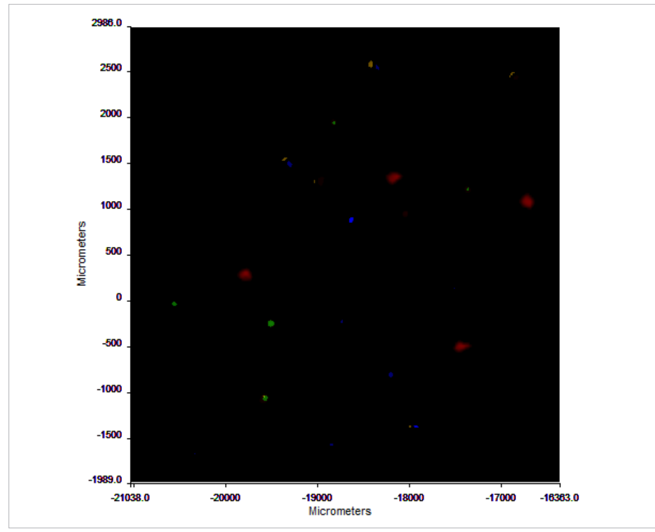

Figure 5. Mixed scores plot. Different colors represent different polymer types.

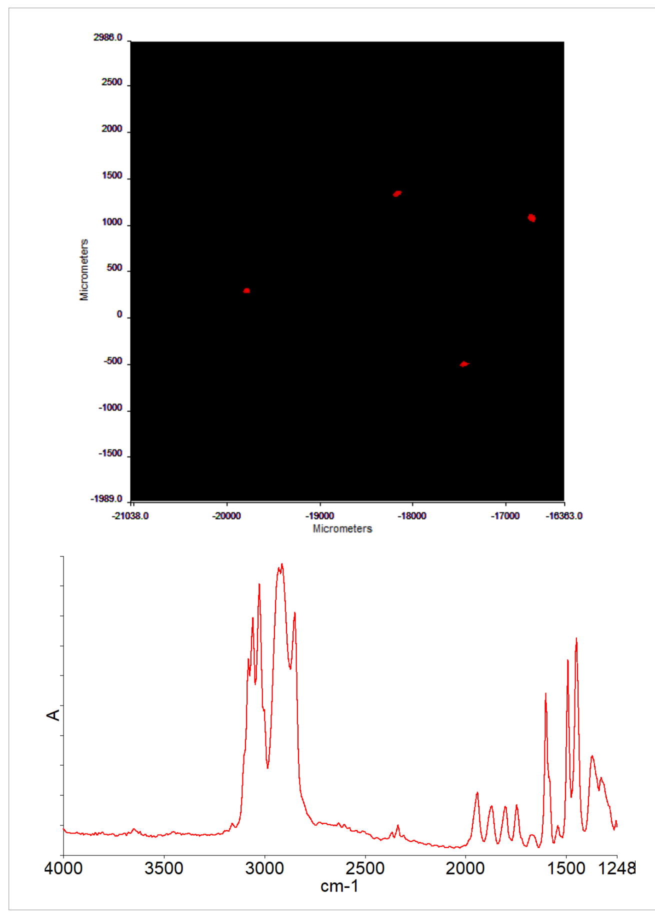

Figure 6. PS Score and IR spectrum.

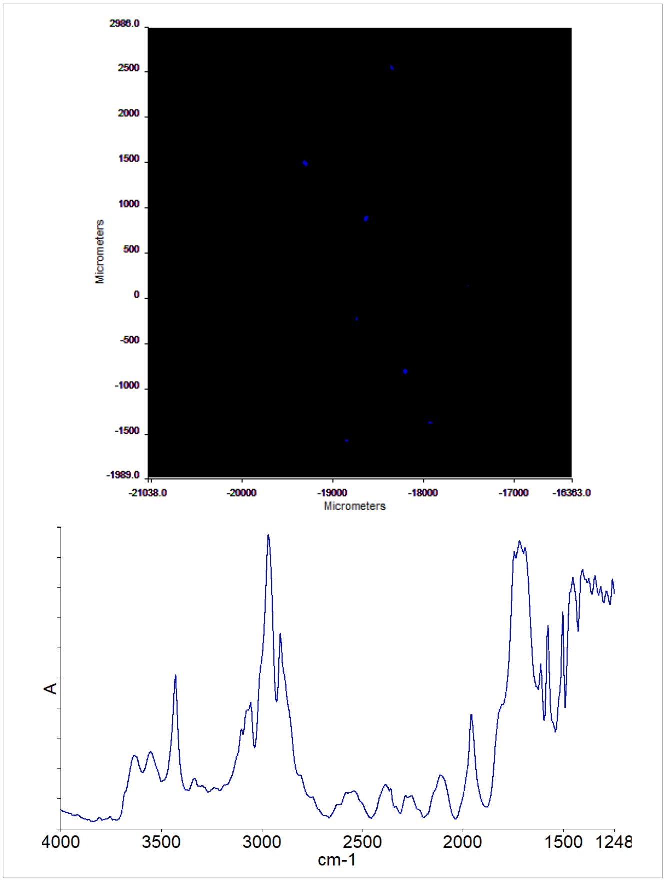

Figure 7. PET Score and spectrum.

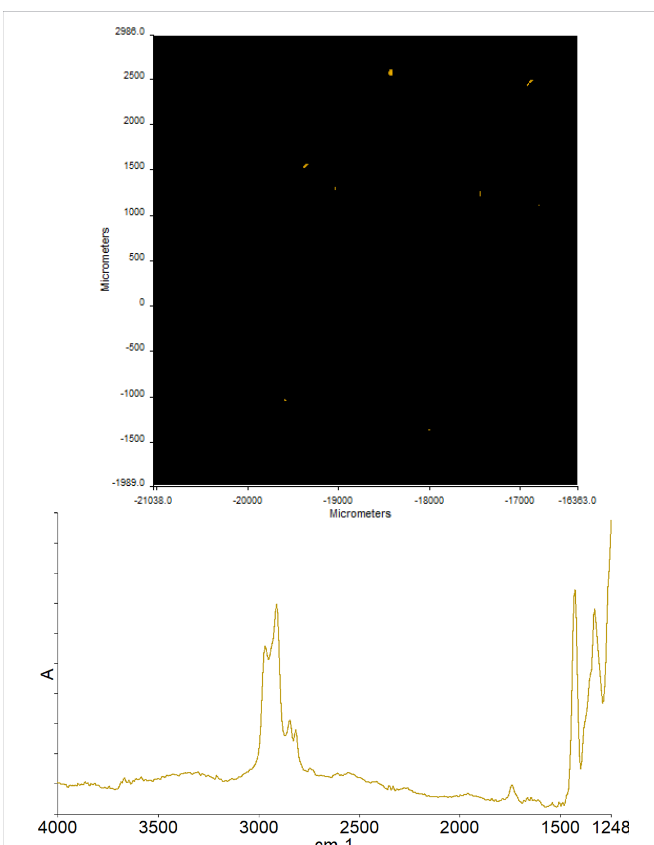

Figure 8. PVC Score and spectrum.

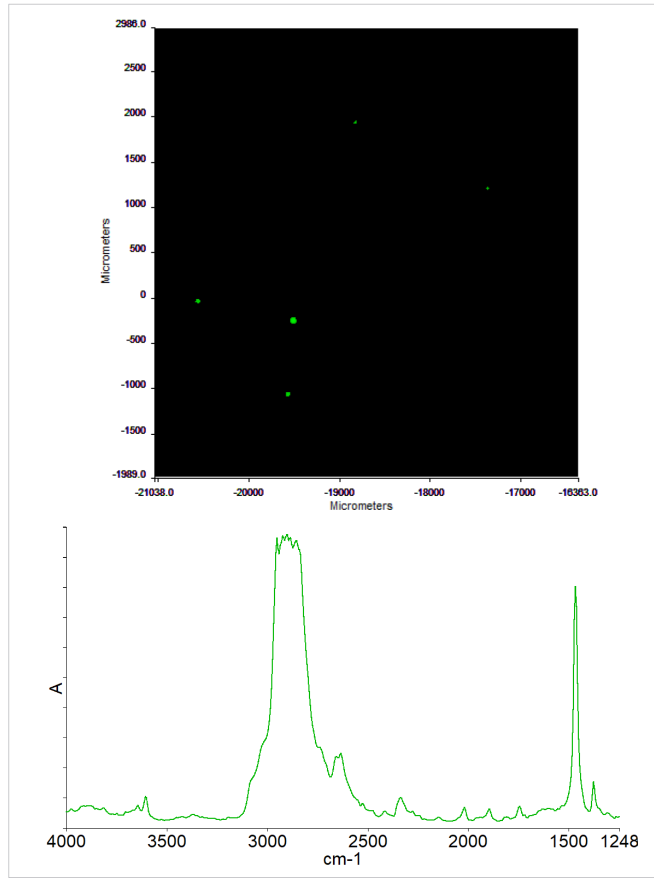

Figure 9. PE Score and spectrum.

Table 1. Summary of the particles detected in the analysis.

Polymer Type	Number of Particles
Polystyrene (PS)	4
Polyethylene (PE)	5
Polyethylene Terephthalate (PET)	8
Polyvinyl Chloride (PVC)	8
TOTAL	25

Summary

The spiked test sample utilized in this study provides an excellent method for the creation of a standard test sample for microplastics analysis, appropriate for a range of different analytical techniques.

IR imaging has been shown to be an excellent analytical technique for the detection and identification of microplastics present in an unknown sample and can be applied to a much larger range of samples containing microplastics using appropriate sample collection and clean-up.

Advanced data analysis techniques simplify and speed up the extensive processing of the data, easily detecting and identifying different polymer types present. This can be achieved using routines within the Spectrum Image software or the data is directly compatible with 3rd party microplastics analysis software, such as Purency[4] or siMPle.[5]

References

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PerkinElmer, Inc.
940 Winter Street
Waltham, MA 02451 USA
P: (800) 762-4000 or
(+1) 203-925-4602
www.perkinelmer.com

For a complete listing of our global offices, visit www.perkinelmer.com/ContactUs

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