APPLICATION NOTE

Infrared, IR Microscopy

Author:

Ian Robertson

PerkinElmer, Inc.
Seer Green, UK

# Detection and Identification of Microplastic Particles in Cosmetic Formulations Using IR Microscopy

## Introduction

It is estimated that there is in excess of 150 million tons of plastic materials in the world's oceans[1]. Much of this pollution consists of large items such as discarded drink bottles and plastic bags. However, there is increasing research into the amount of much smaller materials, termed microplastics, in the river and ocean systems which present a different type of problem for marine life.

Many cosmetic products, such as facial scrubs, toothpastes, and shower gels, currently contain microplastic beads as abrasive materials. These microplastics, which are typically sub-millimetre in size, get washed down the sink and are too small to be filtered by sewage treatment plants consequently ending up in the river systems and ultimately in the oceans. These microplastics can be ingested by marine organisms and fish and end up in the human food chain.

In 2014 a number of U.S. states banned the use of microplastics in cosmetic formulations and most cosmetic companies are voluntarily phasing out their use.

Infrared (IR) spectroscopy is the established technique for identifying polymer materials and has been used extensively for identifying large (over 100 micrometer) polymer materials. The Spectrum Two™ is a portable FT-IR spectrometer that can operate from a battery pack and has been used on boats for immediate identification of these polymers.[1] For microplastics, down to a few micrometers in size, an IR microscope can be used for the detection and identification of these materials.

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Two commercially available products were tested using the Spotlight™ 200i IR microscope system in order to determine whether microplastics were present as the exfoliant and to identify the types of plastics used.

Product 1 is a commercially available facial scrub. Product 2 is a commercially available body scrub. Each of these products was mixed with hot water in order to dissolve the soluble ingredients in the formulation. The resulting solution was filtered through a 50 micrometer mesh, capturing any insoluble components greater than 50 micrometers in size. The filter was then allowed to dry in air prior to IR microscopy measurements. The samples were measured both directly on the mesh and also after transferring the residual particles onto an IR transmitting window on a microscope holder. Visible images of the collected microplastics are shown as Figures 1a and 1b.

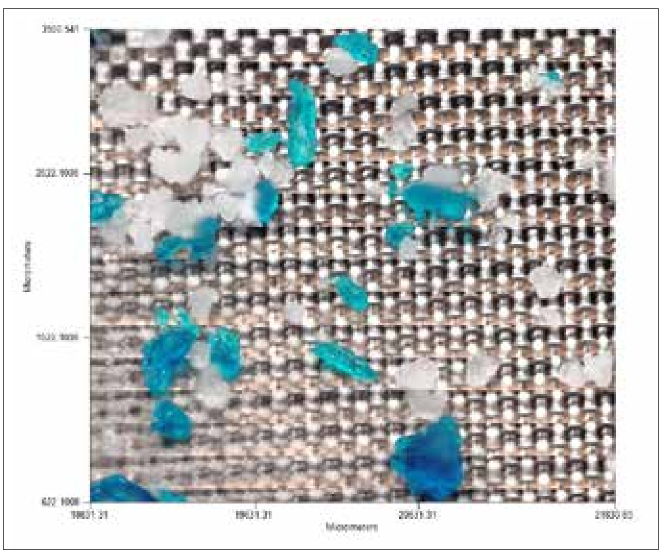

*Figure 1a:* Microplastics in Product 1 (facial scrub) collected on mesh.

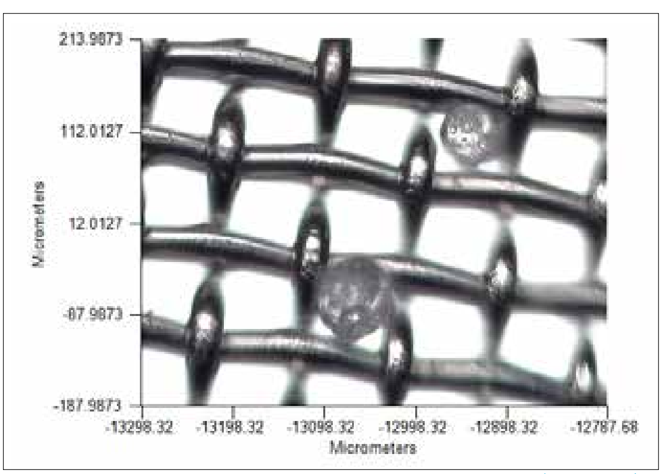


*Figure 1b:* Magnified view of microplastics collected from Product 2 (body scrub).

It is clear from these images that Product 1 has irregular-shaped microplastics with particles of two different colors. The particles from Product 2 are regular spheres with those visible in Figure 1b being approximately 50 and 80 micrometers in diameter. Infrared spectra of these materials can be measured in either transmission or reflectance on the IR microscope. Spectra measured on one of the particles in Figure 1a, in-situ on the mesh, are shown as Figure 2.

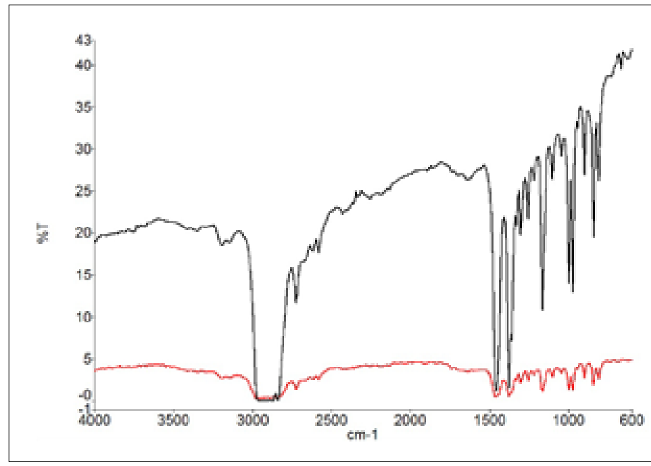


*Figure 2:* Spectra from a microplastic particle in Product 1. Transmission spectrum (black) and reflectance spectrum (red).

The transmission spectrum has a much higher signal than the reflectance spectrum and gives better sensitivity for this measurement. In addition, the bands in the reflectance spectrum are more intense due to the fact that the IR beam is effectively passing twice through the sample, known as transflectance. For smaller particles this does not cause any problems; but for larger particles the path length may be too large leading to totally absorbing bands, thus making identification more difficult. However, in this case, it would be possible to identify the material from either the transmission or reflectance spectrum. The mesh may interfere with the transmission measurement, slightly decreasing the amount of energy reaching the detector. This explains the baseline slope observed in the spectrum, but it does not significantly impact the overall measurement. To obtain the best quality spectrum of the material, the sample can be transferred onto an IR-transmitting window material, such as potassium bromide (KBr). A KBr window was placed onto the mesh containing the sample and the mesh inverted thereby transferring the microplastic particles directly onto the KBr window.

A "Visible Image Survey" was collected over the area containing the majority of the particles in Product 1. Selecting the "Analyze Image" function in the Spectrum 10 software invokes the intelligent automated routine for detecting particles within this Visible Image Survey, which is displayed as "analyze image result" shown in Figure 3.

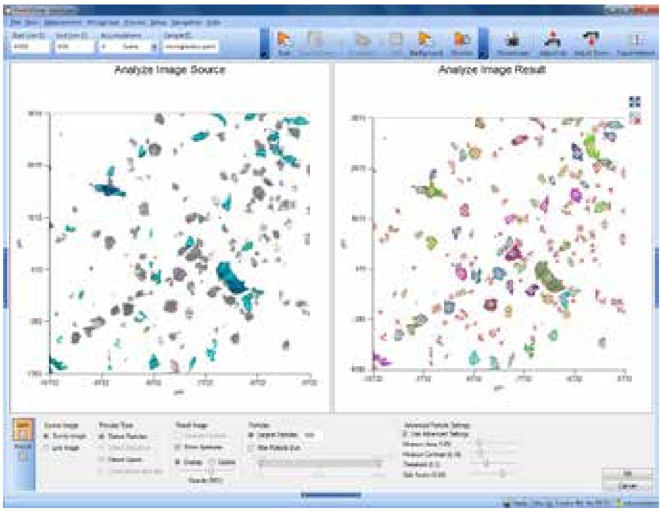


*Figure 3:* The Analyze Image software routine detects the particles in Product 1.

This routine will automatically detect any particles present in the visible image and mark them as regions of interest. It will then calculate the maximum rectangular aperture size that can fit wholly inside each of the particles, thus maximizing signal-to-noise when the data is scanned. In the past, manual selection of the regions of interest and setting of apertures took a considerable amount of time. Clicking "Scan Markers" initiates the collection of transmission spectra (using equivalent apertures for the background) for each particle, displaying ratioed sample spectra in real time as they are collected. Automatic processing of the spectra, using software routines such as Search, Compare, or Verify, can be performed during data collection. In this case, the analysis of the microplastics, a spectral search was performed against a library of polymer spectra to give the identity of each of the particles as shown in the results screen in Figure 4.

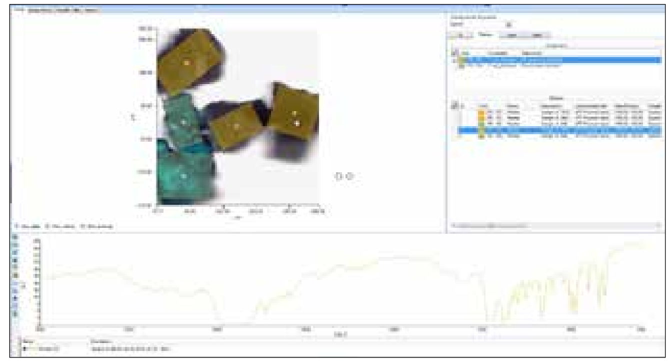

*Figure 4.* Results screen for the detection and identification of particles.

The results show that Product 1 has two different types of polymers present, polypropylene and polyethylene. Product 2 contains only particles of polyethylene. Representative spectra are shown in Figure 5. Small differences are observable in the spectra of the polyethylene between the two different products, most likely due to additives present.

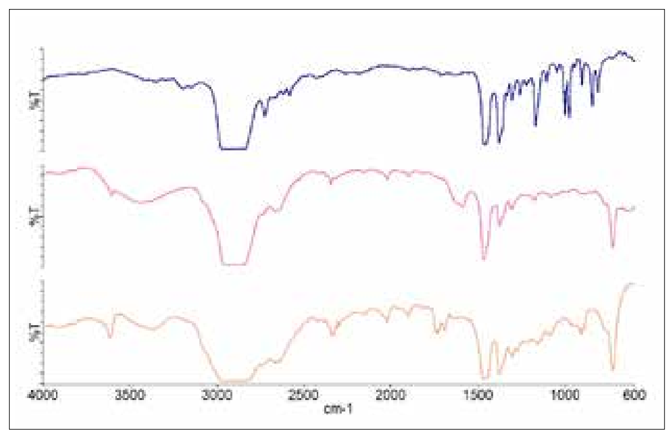


*Figure 5:* Top – spectrum of polypropylene in Product 1. Middle – spectrum of polyethylene in Product 1. Bottom – spectrum of polyethylene in Product 2.

## Summary

Microplastics are a major concern regarding their impact on the environment and as such their use in consumer products is increasingly being prohibited. An automated IR microscopy system has been shown to be an invaluable method for the detection and identification of a source of microplastics in cosmetic formulations. The work presented here will be extended to analyze samples of microplastics collected from European river systems to illustrate how widespread this pollution problem is within marine environments.

## References

1. Labo magazine – Oktober 2010 Wasserverschmutzung durch Mikroplastikpartikel, www.labo.de

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



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