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APPLICATION NOTE

No. 52921

Microplastics in the environment

Key words: Environment, Exfoliants, Filters, FTIR Microscopy, MicroATR, Microbeads, Microplastics

Abstract

Several classes of consumer products, including shower soaps and toothpastes, contain tiny pieces of plastic, called microbeads, which enhance the scrubbing or exfoliating power. Unfortunately, these materials can also accumulate in the environment – especially marine environments – where they can enter the food chain. Cycling affected water through filters enables capture and analysis of the microbeads. The microbeads are roughly 25 to 100 microns in size and are easily analyzed by basic FTIR microscopy. A reference set of samples and beads from a bath scrub are analyzed here after extraction via filtration. Using a tip-ATR, the entire analysis takes less than a minute with no sample preparation required.

Figure 1: Thermo Scientific Nicolet iN5 FTIR microscope

Introduction

The outer layers of skin contain a significant number of dead cells. Removal of these cells, called exfoliation, produces a cosmetically cleaner appearance, making exfoliation a common practice in facials and other treatments. This can be done aggressively, using sandpaper-like boards, or in a more subtle manner using exfoliating creams or soaps. With a surge of interest in this practice, many cosmetic producers began using microbeads and other materials to enhance exfoliation. Some, such as pumice and salts, are natural products, but they also include synthetic materials such as polymeric microbeads.

The microbeads are typically around 25-100 microns in size – making them visible to the unaided eye. Concerns have been raised about the large number of microbeads released and the slow or negligible breakdown of these materials, and their subsequent entry into the food chain through microorganisms and filter feeders.

FTIR microscopy is an excellent tool for detecting and identifying these materials. The Thermo Scientific™ Nicolet™ iN™5 FTIR microscope shown in Figure 1 was designed for exactly such an application, with extreme simplicity in design and function and powerful analysis software. This is enhanced by the microATR capabilities which enable the analysis with no sample preparation. Here, we use the Nicolet iN5 microscope and Germanium (Ge) ATR to analyze reference materials and samples extracted from a consumer product.

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Experimental

A Nicolet iN5 FTIR microscope was configured with a Ge-tip ATR. A solution containing a commercial standard of 25 micron diameter polystyrene beads was evaporated onto a glass slide to serve as a reference sample. A sample from an exfoliating cleanser was analyzed after filtration through a standard paper filter without further treatment. Finally, a set of microbeads used for X-ray diagnostic procedures were similarly filtered and analyzed. The Ge-tip ATR and 100-micron aperture in the Nicolet iN5 microscope yield an effective spatial resolution of 25 microns, perfectly gauged to match these samples. The visual images were acquired using the built-in camera of the Nicolet iN5 system. The analysis was carried out using the Thermo Scientific™ OMNIC™ and OMNIC Specta™ spectroscopy software and standard polymer libraries.

Results

Figure 2 shows a relatively large field of view image of particles dried from a solution of reference standard 25 micron microbeads – the consistency of the microbeads is clearly visible. Figure 3 shows a sparsely populated region where a single, isolated particle could be selected. The circular image is taken through the 100 micron aperture of the Nicolet iN5 microscope. The Ge-tip ATR was inserted and contact made as indicated by a simple red-green light on the Nicolet iN5 system. The resulting spectrum (40 second acquisition) is also shown in Figure 3, indicating clearly that the Ge-tip ATR is contacting the particle accurately. The high level match to polystyrene (> 92) further confirms the excellent performance of this combination.

To demonstrate the 4X magnification provided by Ge-ATR, spectra from a crowded region and from a single microbead are compared in Figure 4, on a common y-axis scale. The similarity in intensity indicates there is no additional signal from the cluster of beads, only a slight baseline tilt due to scattering from the multiple particles. The spectrum from the single particle is actually the cleaner of the two.

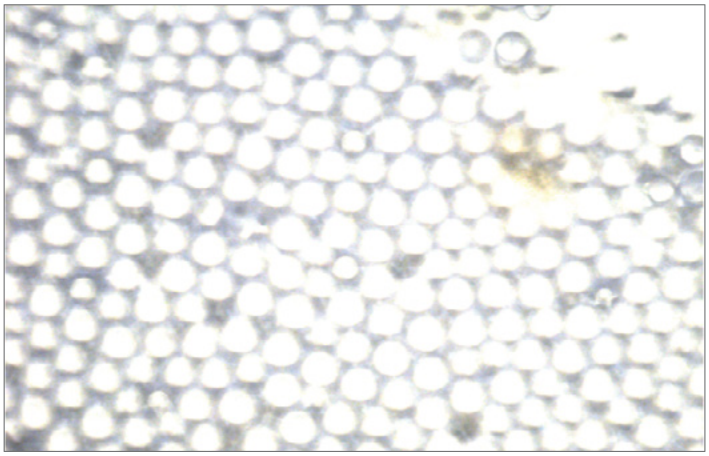

Figure 2: Plastic microbeads evaporated from solution onto glass slide.

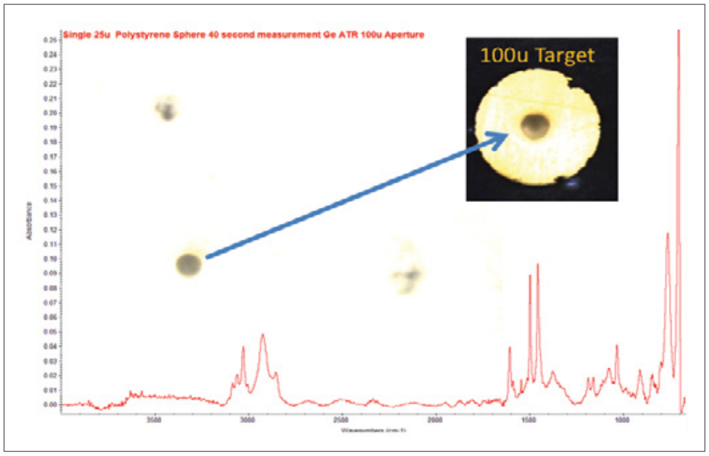

Figure 3: Single 25µ polystyrene bead on glass slide.

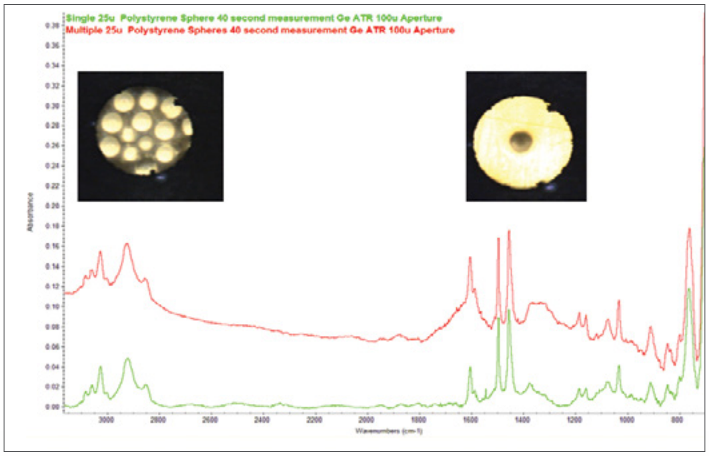

Figure 4: Beads through 100 micron aperture demonstrate 4X magnification with Germanium.

Figure 5 shows spectra obtained from a filtered sample of exfoliating cream. There is a residual layer of cream over the filter, so the spectrum from the microbead shows multiple components. A spectrum from a nearby region free of microbeads was subtracted, yielding a clean spectrum of the bead. This matches low molecular weight polyethylene, with some oxidation. Further, the technique allows the soap residue itself to be analyzed with a strong match to propylene glycol ether, as seen in Figure 6.

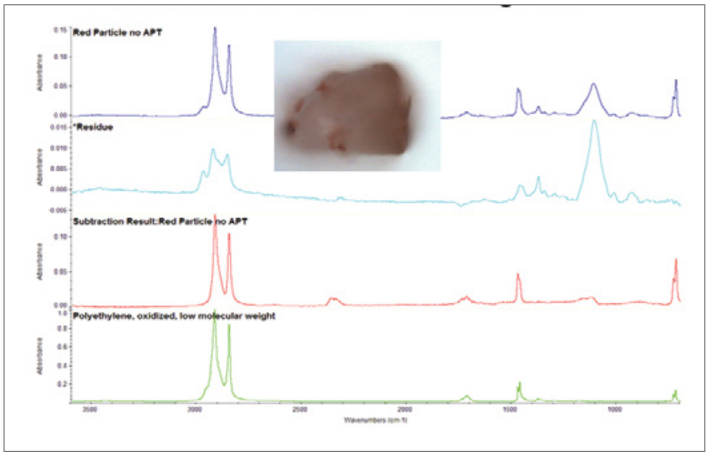

Figure 5: Particles filtered from an exfoliating scrub.

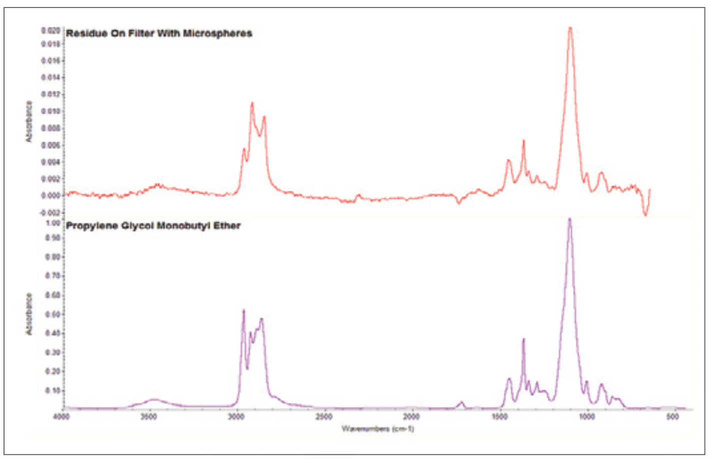

Figure 6: Even after several washes of the filter with water, some residual cream remains from the lotion as well as the particles.

Figure 7 shows spectra of microbeads from a sample containing multiple sizes and colors of beads, also on a filter. All of these appear to be polyethylene. However, the blue beads appear to have a second component, indicated by the additional peaks between 1000 and 1200 cm-1. The OMNIC Specta multi-component search function identifies both components simultaneously, the polyethylene and a barium-sulfur compound, as seen in Figure 8. These beads are used in X-ray diagnostics, especially in veterinary medicine, and Barium is added as an X-ray tracer, with the resulting material called 'Barium Impregnated Polyethylene Spheres.'

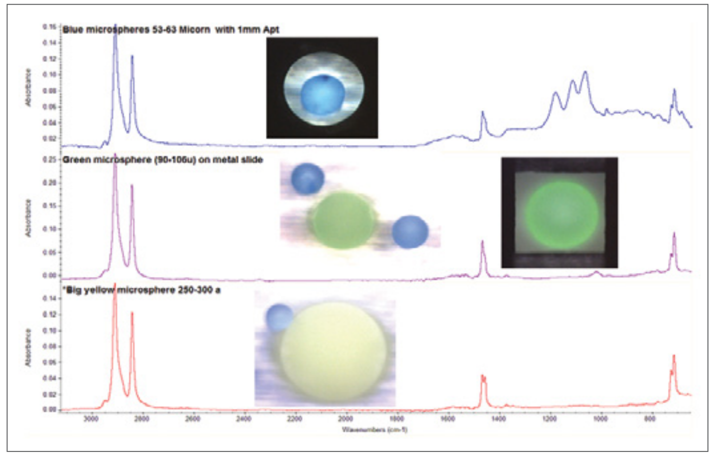

Figure 7: All three spheres are polyethyelene. However the spectrum from the small blue spheres contains a second component with strong peaks above 1000 cm-1. A spectral search of this region produces a good match for Barium Sulfate.

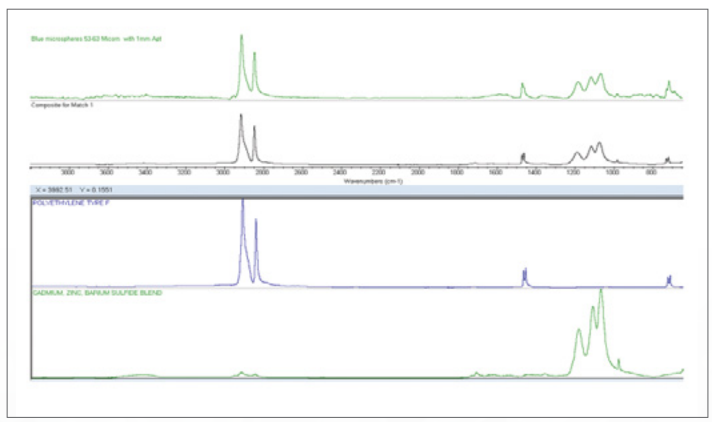

Figure 8: Barium sulfate is added to microspheres to be used to calibrate x-ray imaging devices. Barium Sulfate Polymer Microspheres are created for scientists searching for X-ray opaque microspheres with high sphericity and tight particle size distribution. Barium Sulfate is embedded into the microspheres and acts as a radiocontrast agent for X-ray imaging and other diagnostic procedures. We recommend the higher density 1.5g/cc product for best contrast, these are sometimes call barium impregnated polyethylene spheres (BIPS) used in veterinary medecine.

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Conclusions

The Nicolet iN5 FTIR microscope is ideally suited for the analysis of microbeads on filters. The simplicity of operation targets this at laboratories with minimal microscopy experience. The most common microbeads are around 25-100 microns in size, for which the microscope and its Ge-tip ATR are perfectly suited. Coupled with the OMNIC and OMNIC Specta software, a typical analysis can require no more than a minute, with visual positioning using manual operations, simple contact-and-analyze ATR and the most trusted software in spectroscopy.

Find out more at **thermofisher.com/iN5**

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