The application of Infrared microscopy for the analysis of microplastics in water-borne environmental samples

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Background

It is estimated that there is in excess of 150 million tonnes of plastic materials in the World's oceans increasing by 6.4 million tonnes per annum1 . Much of this pollution is large items such as discarded drinks bottles or plastic carrier bags. However there is increasing research into the amount of much smaller materials, termed microplastics, in the river and ocean systems as they present a major problem for marine life.

Plastic fragments deposited on beach in Lanzarote after every high tide

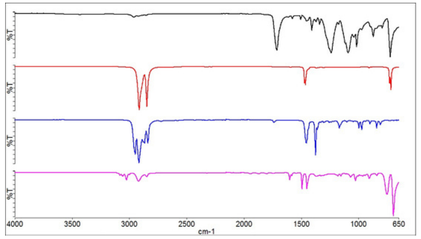
Microplastics are generated from the breakdown of larger plastics by UV degradation combined with wind and wave impact. They are also present in many consumer products such as facial scrubs, toothpastes and shower gels. These microplastics, which are typically sub-millimetre in size, get washed down the sink as they 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 US states banned the use of microplastics in cosmetic formulations and most cosmetic companies are voluntarily phasing out their use , replacing them with more eco-friendly materials.

IR spectroscopy of microplastics

Infrared (IR) spectroscopy is the established technique for identifying polymer materials and has been used extensively for identifying large (over 100 µm) polymer materials.

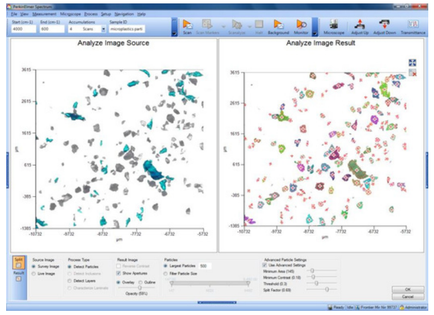
The Spectrum Two is a portable FT-IR spectrometer that can operate from a battery pack and has been used on boats for immediate identification of these polymers1. For microplastics, down to a few µm in size, an IR microscope can be used for the detection and identification of these materials.



ATR spectra of larger (>1mm) plastic fragments Identified as PET, polyethylene, polypropylene and polystyrene

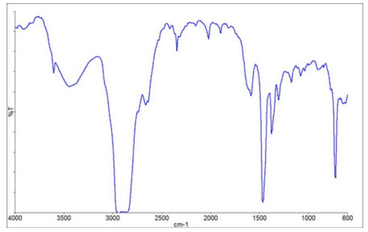
Microplastics in consumer products

A commercially available facial scrub product was tested using the Spotlight 200i FTIR microscope in order to determine if microplastics were present and to identify the types of plastics used. Soluble ingredients were extracted and the non-solubles filtered using a 50 µm metal mesh. The non-soluble materials were placed on a KBr window and placed in the IR microscope for transmission measurement..

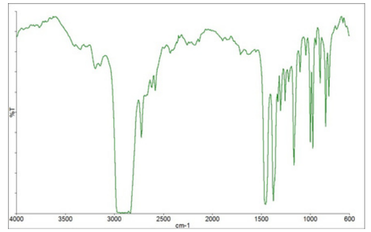


The Analyze Image routine (left) detects the particles and optimises the aperture for the measurement. The particles are then automatically scanned and identified using a polymer search library. Two different polymer types, polyethylene and polypropylene were found to be present in the sample as shown in the spectra and Search results below..

Search Score : 0.959831 Polyethylene



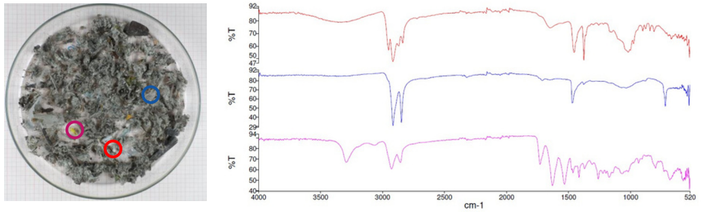
Search Score : 0.98174 Polypropylene, isotactic



PerkinElmer Spotlight 200i FT-IR Microscope

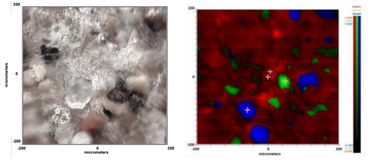
Detection of microplastics in the River Danube

Samples extracted from the river Danube with 500 and 50 µm nets are analysed using the Spotlight 400 FTIR microscope. Fragments larger than 500 µm are measured in ATR mode.



Left: Dried material sampled from the river Danube, mainly of biological origin; some plastic fragments are already visible by the naked eye.
Right: IR spectra of 3 larger plastic fragments (>500 µm, encircled in left figure) identify the materials polypropylene (PP, red), polyethylene (PE, blue), and polyamide (PA, purple).

For the smaller particles the ATR imaging accessory proves very useful. The sample area is covered with particles (below left), and scanned within a few minutes at 6 µm pixel size. The following PCA elucidates the data structure (below right). Spectra extraction from the green and blue regions yields polyolefins as best hits.



Left: Visual image of the sample area covered with microparticles
Right: PCA plot of the sample area, based on ATR imaging. Colored regions are identified as PP (green), PE (blue), and non specific (red).

Conclusions

The correct detection and identification of plastics in the micron range is an emerging issue of interest in the context of oceans, surface waters, and other environmental compartments. The well-established technique of FTIR combines perfectly with the features of microscopy and imaging, and allows reliable detection and identification of these pollutants.

Acknowledgements:

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* Environment Agency Austria, Vienna

References :

1. Labo magazine – Oktober 2010 Wasserverschmutzung durch Mikroplastikpartikel