# CASE STUDY: UNIVERSITY OF BIRMINGHAM TEAM USES AGILE TGA-FTIR-GC/MS WORKFLOW TO ADVANCE MICROPLASTICS RESEARCH

## Introduction

Microplastics are defined as pieces of plastic that are less than 5 mm (0.2 inch) in length. Over the past decade or so, these minute structures have been discovered in marine, freshwater, terrestrial, and atmospheric environments in alarming abundance.

PerkinElmer
*For the Better*

In aquatic environments, microplastics often enter freshwater systems within which they migrate to marine environments where it can take up to 600 years for them to degrade. They are also ingested by aquatic fauna and absorbed by aquatic flora, which can result in their bioaccumulation within food chains. Finally, toxic chemicals can be adsorbed by microplastics and harmful pathogens can adhere to particle surfaces. The resulting impacts of microplastics on aquatic ecosystems are not fully known at this time, but there is growing interest and research focused on gaining a better understanding of this and how to address the impacts.

Aquatic biologists require comprehensive data on microplastics chemical composition, size-distribution relationships, tissue and cellular fates, and effects on aquatic biota. To obtain such data, scientists rely upon analytical instrumentation and methodologies that are able to generate accurate, sensitive, reproducible data for a range of sample types. To-date, analytical methods used in microplastics research have been plagued by poor polymer identification rates due to an array of issues such as polymer color, biofouling or environmental aging. The biggest challenge is the lack of standardized methodologies that articulate best practices and approaches to ensure high-quality, sensitive, reproducible methods within standard frameworks.

A UK-based research team is making great strides in characterizing and understanding microplastics in aquatic environments. The team includes experts in aquatic ecology and analytical methodology, a perfect match for microplastics research.

### THE UNIVERSITY OF BIRMINGHAM RESEARCH TEAM

Dr. Holly Nel and Dr. Andrew Chetwynd are part of a microplastics research team in the University of Birmingham's (UoB) School of Geography, Earth and Environmental Sciences in England. Their collaboration addresses the microplastic pollution problem from different, but complimentary, angles.

Dr. Nel is an expert at in-situ sampling and sample preparation techniques for microplastics in fresh and marine water, sediment, and biota. Her research focuses on understanding the movement of plastics through aquatic systems, the drivers of and barriers to that movement, the source-sink dynamics of a range of MP particle sizes, and the impacts of MPs on aquatic organisms.

Dr. Nel spends a lot of time in the field collecting samples and preparing them for analysis. The data generated from the samples is used in conjunction with lab-based studies and models to understand various transport and fate scenarios. Generating accurate and reliable data is, then, one of Dr. Nel's primary goals and is crucial for developing an understanding of the movement and effects of different particle sizes at the full ecosystem level. To gain that understanding, she requires improved detection capabilities for a range of particle sizes as well as identifying the effects of different sample preparation techniques on particle detection.

After collecting field samples, Dr. Nel needs advanced analytical capabilities to extract the chemical and physical data that will propel her research forward. That is where Dr. Chetwynd's work comes in. Dr. Chetwynd is an expert in the development of novel analytical methods that push the boundaries of analyte detection and characterization. His current research focuses on developing enhanced analytical methods for the characterization of nanomaterials, microplastic particles, individual polymers, and proteins and metabolites that adsorb to micro- and nanomaterials.

Dr. Chetwynd is developing methods that provide the greater sensitivity, reproducibility, and reliability needed to advance microplastics characterization, and to do so using less sample mass. He is also designing methods that allow the detection of particles within organelles—an important progression from detecting particles within cells.

### ADVANCED, AGILE WORKFLOWS

Dr. Chetwynd uses several PerkinElmer, Inc. (PerkinElmer) instruments and software to develop and evaluate the advanced analytical methods needed for microplastics research. He describes PerkinElmer's modular workflow of thermogravimetric analysis (TGA), Fourier-transform infrared spectroscopy (FTIR), and gas chromatography/mass spectroscopy (GC/MS) as one of his most versatile and valuable tools. "I can use each module individually or in different hyphenations," he explains, "meaning I can customize the platform for each specific project or analysis."

One example of this customizability is Dr. Chetwynd's use of the TGA-FTIR-GC/MS hyphenated workflow to accurately distinguish polypropylene, polyethylene, and polystyrene within a sample. He uses TGA analysis to initially determine if a sample contains polypropylene, polyethylene, and/or polystyrene, which have very similar TGA profiles. FTIR is then used to confirm or rule out the presence of polystyrene, followed by GC/MS to distinguish between polypropylene and polyethylene. PerkinElmer's FTIR also identifies polymer additives, further helping to avoid mischaracterization of microplastics. The hyphenated workflow provides Dr. Chetwynd with the detailed, accurate data needed to identify each polymer type contained in a sample.

Another successful application of the TGA-FTIR-GC/MS hyphenated workflow is distinguishing between different pyrolysis products of a polymer. Dr. Chetwynd explains that polystyrene samples tend to expand in volume, limiting the size of sample that can be loaded into TGA crucibles and, therefore, the level of detail that can be achieved from the reduced sample mass. This is a problem in polystyrene identification because it relies upon styrene dimer and trimer data, not just total styrene. Styrene is distinguishable using GC/MS, but the TGA and FTIR modules provide the detailed thermal and chemical information needed to accurately identify the styrene dimer and trimer. The sensitivity of the workflow provides Dr. Chetwynd with the specificity needed to accurately identify polystyrene in a microplastic sample.

Microplastics analysis has historically struggled with inconclusive results and false identifications. The exceptional sensitivity, accuracy, and versatility of the TGA-FTIR-GC/MS hyphenated workflow is helping Dr. Chetwynd overcome those challenges and provides Dr. Nel with the detailed data she needs for her work. Dr. Nel also appreciates that the workflow's data output and analysis reports are easy to understand for researchers like her who focus on measuring the impacts of microplastics in marine ecosystems.

### INTEGRATED SOFTWARE AND POLYMER REFERENCE LIBRARIES

The PerkinElmer software specific to each instrument enable researchers like Dr. Chetwynd and Dr. Nel to evaluate their data efficiently and accurately. As Dr. Chetwynd describes, "the TGA's Pyris™ software has all the required calculations built into the different steps, so it is easy to interrogate the data and get results quickly." He also appreciates that "the FTIR data is easily exportable to a spreadsheet so it can be shared with colleagues in the microplastics arena."

The PerkinElmer GC/MS software includes a polymer reference library that is used to confirm the identity of a polymer. The software also acknowledges a polymer that does not correspond to any within the existing polymer library, thus preventing false identifications. As microplastics research expands, more polymers can be added to the library, which is something that both Dr. Chetwynd and Dr. Nel appreciate. "The software platform is very helpful for building a plastic-specific or location-specific polymer library," explains Dr. Nel, which "facilitates future analyses and supports sharing and collaboration with other scientists" says Dr. Chetwynd.

### THE TEAM'S NEXT STEPS

The data obtained from the UoB team's research are being used to identify the specific plastics present in location-specific and media-specific samples. So far, they have found polyethylene and polypropylene to be the most dominant polymers in their samples.

Dr. Nel explains that "being able to accurately identify the specific

CASE STUDY

microplastic chemicals in our samples helps to identify hot spots and vulnerable areas, pinpoint their sources, and aid in the development of targeted mitigation strategies." To get to that point, she sees the importance of developing faster sample preparation methods and faster data availability, possibly through the use of in-situ sensors.

Dr. Chetwynd continues to pursue analytical methods that provide improved microplastic characterization with lower sample mass requirements. He also anticipates moving toward methods that can further distinguish copolymers, decipher plastic mixtures, and quantify the components of microplastic particles. He even envisions future techniques that can be used to characterize microplastics in-situ within the original sample matrix.

## THE FUTURE OF MICROPLASTICS RESEARCH

The long-range goals of microplastics research are diverse and include:

- Obtaining long-term monitoring data for individual systems so accurate trends can be identified
- Determining the effects of microplastics and individual polymers, additives, and fillers on aquatic ecosystems
- Being able to identify or trace the source of location-specific microplastic contamination
- Designing and implementing targeted remedial strategies for existing contamination
- Making changes in plastic production to eliminate problematic polymers
- Enabling targeted minimization of microplastic releases from production, waste disposal, and recycling operations

As these goals are pursued and eventually achieved, Dr. Chetwynd and Dr. Nel see the need for standardization of methods and criteria in order to effectively address the worldwide microplastics pollution problem. Dr. Chetwynd points to the need for standardization of analytical methods used in microplastics research and monitoring. He believes the sample preparation methods will differ by medium, but the subsequent analytical methods will be consistent. Regarding regulatory criteria for microplastics, Dr. Chetwynd sees the need for a regulatory framework that is similar to those already in place for drinking water, air, and so forth.

Dr. Nel explains that "standardized methods and harmonized practices will be needed for tracking and reporting microplastic pollution so that global research can be integrated and effective." She sees such integration beginning to occur in the growth of a research database that identifies researchers, instruments, and polymer libraries that are available internationally. In her home continent of Africa, a similar database is being developed to help unify and support microplastics research across African nations. PerkinElmer is proud to support the work of this dynamic research team at the University of Birmingham.

## CONCLUSION

Pairing Dr. Nel's ecological research with Dr. Chetwynd's analytical methodology research creates a holistic approach to understanding the lifecycle and impacts of microplastics in aquatic ecosystems. This research team will undoubtedly continue hammering away at the microplastics pollution problem until real solutions are found and implemented. Despite the pandemic of 2020, Dr. Nel and Dr. Chetwynd were able to continue their work with the help of PerkinElmer's instrumentation, software, and technical support. Just before the pandemic emerged, the team had analyzed a large collection of samples and generated a substantial amount of data. Suddenly, they found themselves prohibited from going to the lab to use their PerkinElmer software for data analysis. Dr. Chetwynd reached out to PerkinElmer for help. "Within two days, they got us the software suites we needed to continue our data analysis at home," he reports, "instead of having to wait what ended up being a year to find out what the data revealed." They were relieved to be able to write reports of their work for publication and present their findings at online workshops and conferences, such as the SETAC Europe 30th Annual Meeting in May of 2020.

**For more information please visit www.perkinelmer.com/microplastics**