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Sample preparation plays a pivotal role in simplifying and expediting microplastic determination and helps reducing the overall analysis time.

BACKGROUND

The increasing use of plastics in our daily lives and in manufacturing processes often culminates in a slow degradation of the materials and the subsequent contamination of our environment and food chain.

Scientists define microplastics as plastic fragments less than 5 mm, or about 0.2 inches, in diameter. Nanoplastics are even smaller, with diameters less than 0.001 mm.

Recent studies demonstrated that the human ingestion of MPs is about 120 thousand particles annually, which comes from water, food or even air[1]. Research in wildlife and animal models has linked micro- and nanoplastic exposure to infertility, inflammation and cancer, but health outcomes in people are currently under assessment, with effects mainly associated with the blockage of the digestive tract.

While regulators are trying to understand the extent and toxicity of the problem, researchers and analytical methods bodies are working towards standardized analytical solutions to best characterize these particles.

In this white paper, we will present **two new methods for microplastic determinations** that aim to simplify and expedite this type of analysis. Featuring scientific publications by two experts in the field, prof. Cezar A Bizzi of **University of Santa Maria**, and prof.

Jacopo La Nasa, of the **University of Pisa**, this paper will underline how the use of microwave technology has been applied to microplastic determinations by two different analytical techniques.

DETERMINATION OF MICROPLASTIC CONTENT IN SEAFOOD: AN INTEGRATED APPROACH COMBINED WITH THE DETERMINATION OF ELEMENTAL CONTAMINANTS[2]

Several methods are available for the identification and characterization of MPs in fish tissue, but few of them can be applied for quantification. They often require long reaction times, up to 72 h, a high reagent consumption and lead to the degradation of MPs during the process.

Microwave sample preparation has been successfully and widely applied for both **elemental and molecular analysis** thanks to its efficiency and selectivity achievable with volumetric heating.

A selective microwave assisted sample preparation protocol has been developed for the gravimetric determination of MPs in fish tissue. The innovative microwave protocol ensures a selective digestion of fish tissue without affecting the composition of the MPs. The protocol was performed with Milestone's ultraWAVE[3] with the aim to develop a fast and easy-to-apply protocol for routine high throughput analysis (Figure 1).

WHAT ARE THE PRINCIPLES OF THE METHOD?

A simple approach was developed using **diluted acid with microwave-assisted wet digestion**.

To develop a reliable method for the determination of MP amount, up to 2 g of an in natura seafood sample were spiked with a known amount of MP (100 mg of mixed MP). Suitable conditions were obtained using 1 mol L–1 HNO3 at 200 °C (10 min holding time). Digests were filtered and the plastic content was gravimetrically determined. The heating program was 20 min, which represents a significant reduction in the time normally reported in the literature for MP analysis (from few hours up to 3 days). The proposed method allowed gravimetric determination of eight plastic types (polyethylene terephthalate, polystyrene, expanded polystyrene, polypropylene, high and low density polyethylene, polycarbonate and polyvinyl chloride) with particle size ≥0.3 mm. Up to 2 g of an in natura seafood sample

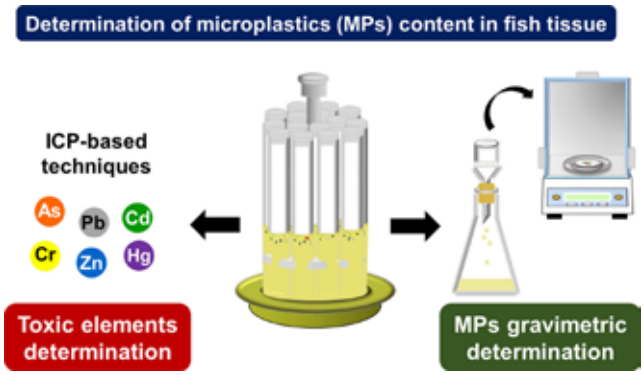

Figure 1. Determination of MPs content in fish tissue

(shark species, acoupa weakfish, tuna fish, trahira, and pink shrimp) were efficiently digested, which opened the possibility of using the proposed digestion method for determining elemental contaminants (Al, As, Ca, Cd, Co, Cr, Cu, Fe, Hg, La, Mg, Mn, Mo, Ni, Pb, and Zn). Thus, as the main feature of the proposed digestion method is the **possibility of determining MP and elemental contaminants using the same digestion protocol**, saves time and reagents and provides accurate and precise information about different classes of marine pollutants (MP and elemental contaminants).

MICROWAVE-ASSISTED SOLVENT EXTRACTION & DOUBLE-SHOT ANALYTICAL PYROLYSIS FOR THE QUALI-QUANTITATION OF PLASTICIZERS AND MICROPLASTICS IN BEACH SAND SAMPLES[4]

Pollution from microplastics (MPs) needs to be evaluated by deploying reliable analytical techniques that provide qualitative and quantitative data on the extent of contamination in the various environmental matrices.

Solvent extraction of MPs followed by pyrolysis-gas chromatography-mass spectrometry (Py-GC–MS) provides data that not only regard the soluble plastics, but also the organic additives contained in MPs and the low molecular weight degradation products of insoluble plastics. The potential of microwave-assisted solvent extraction and double-shot Py-GC–MS was investigated to obtain quali-quantitative information on polystyrene and on phthalate plasticizers in environmental samples. The method was validated and provided **recoveries higher than 96 %**, and detection limits lower than 1 ng and 1 µg for phthalates and polystyrene, respectively. They used the method to analyze samples of sand collected from a shoreline in Tuscany (central Italy) and thereby determine the content of phthalates and polystyrene at different depths and distances from the coastline. Qualitative data were also obtained regarding the presence of oxidation products derived from polyethylene and polypropylene. The different contents of plasticizers, plastics, and degradation products in the investigated samples are discussed in relation to how environmental agents affect the leaching and degradation processes of the polymers.

The Teflon vials used for the microwave extraction and the glass vials used to store the extracts were cleaned in Milestone's traceCLEAN acid steam cleaning system[5] using nitric acid (65%) at 500 °C for 30 min, and then washed with bidistilled water and acetone.

Solvent extractions were performed using a microwave ETHOS system[6]. Extractions tests were carried out for 60 min at 60 and 80 °C with 600 W irradiation power. The best extraction yields were obtained at 80 °C for 60 (Figure 2).

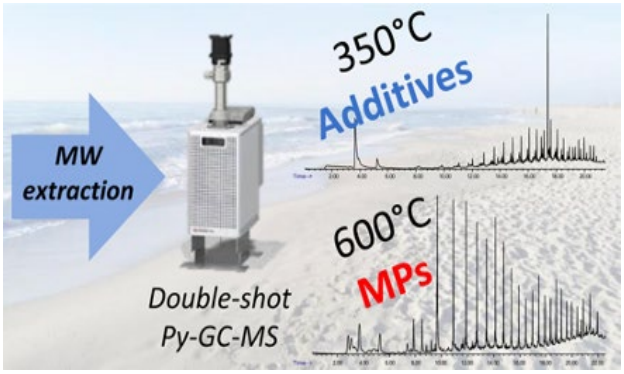

Figure 2. Milestone extraction for quali-quantitation of plasticizers and microplastics in beach sand samples

CONCLUSIONS

Based on the above considerations, the evaluated methods proved two different approaches for the determination of microplastics in environmental samples. Both studies shown the **pivotal role that sample preparation** plays in this analysis and how it helps in reducing the overall analysis time.

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With over 50 patents and more than 20,000 instruments installed in laboratories around the world, Milestone has been widely recognized as the global leader in metals prep technology for the past 30 years. Committed to providing safe, reliable and flexible platforms to enhance your lab's productivity, customers worldwide look to Milestone for their metals digestion, organic extractions, mercury analysis and clean chemistry processing needs.

REFERENCES

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