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A Method for the Extraction of Microplastics from Solid Samples Using Olive Oil

Scopetani C.[1], Pflugmacher S.[1,2,3], Pellinen J.[1]

[1] Faculty of Biological and Environmental Sciences Ecosystems and Environment Research Programme/Almalab, University of Helsinki, Niemenkatu 73, Lahti FI-15140, Finland.
[2] Korean Institute of Science & Technology (KIST Europe) Environmental Safety Group. Joint Laboratory of Applied Ecotoxicology Campus E 7.1 66123 Saarbrücken, Germany.
[3] Helsinki Institute of Sustainability (HELSUS), Fabianinkatu 33, 00014 Helsinki.

Abstract
Microplastics (MPs) extraction from sediment, sand and soil samples is a challenge that the scientific community is facing nowadays; currently there are no standardized and validated protocols and the most common methods rely on density separation techniques, often unable to separate high density polymers. The aim of the present work was to develop a non-density based, efficient and cost effective method. We tested an oil based extracting technique exploiting the oleophilic properties of plastic. Soil and marine and freshwater sand samples were spiked with 11 different kind of polymers: Polypropylene (PP), low density Polyethylene (LDPE), high density Polyethylene (HDPE), Polyurethane (PU), Polyethylene terephthalate (PET), Polystyrene (PS), Polycarbonate (PC), Polyamide (PA), Polyvinyl Chloride (PVC), Polymethyl Methacrylate (PMMA) and Polytetrafluoroethylene (PTFE). FTIR preliminary results show high recovery rates for almost all polymers except for PTFE. Further developments of this promising method are currently in progress.

Keywords: Microplastics, Sand, Soil, Microplastics extraction, Microplastics quantification

1. Introduction

Microplastic (MP) pollution issue is currently one of the most alarming environmental research topics for scientific community, media and general population (Martellini et al., 2018) especially regarding the aquatic environment where the presence of these materials could represent a threat to the ecosystems (Cole et al., 2011). Among marine and freshwater waste, plastic and MPs seem to be the most abundant and they have been found in all matrices (e.g. surface waters, column waters, sediments, soil and ice) and their ingestion by marine and freshwater biota has been widely detected (Panti et al., 2015; Fossi et al., 2016; Martellini et al., 2018; Renzi et al., 2018; Scopetani et al., 2018). Animals can mistake MPs for food and ingest them directly or via predation of organisms already contaminated (Cincinelli et al., 2018). Within aquatic ecosystems, MPs are not evenly distributed in environmental samples (Horton et al., 2017; Gago et al., 2018; Klein et al., 2018) and this difference could result from the absence/lacking of standardized and validated protocols for sampling, quantification, and characterization of MPs (Eerkes-Medrano et al., 2015; Mai et all., 2018). There are significant differences in methods when quantifying MPs in environmental samples, impeding a comparison between studies (Silva et al., 2018). Thus, it is of outmost importance to get the sampling and quantification standardized.

The most complex matrices for MPs extraction are sediment, sand and soil samples because the separation mostly relies on density separation techniques that have several limitations related to the difficulty to extract high-density polymers and the relative high cost of the brine solutions used in the process (Crichton et al., 2017; Felsing et al., 2018).

Here we present results obtained with a new version of the MP extraction method based on oil (Crichton et al., 2017) that exploits the oleophilic properties of plastic and significantly reduces the amount of harmful or expensive chemicals.

2. Materials and Methods

2.1. Sample collection

In order to test the efficiency of the method, both standard soil samples (LUFA natural standard soil from the Agricultural Investigation and Research Institute, Speyer, Germany) and marine and freshwater sand samples were used. Marine sand was collected on a sandy beach of the Regional Natural Park of Migliarino, San Rossore, Massaciuccoli, Pisa, Italy, while freshwater sand was sampled along Arno River shoreline in proximity of the City of Florence, Italy. To be representative of the different grain sizes, we collected samples from low, medium and high tides.

2.2. Oil extraction

Extra virgin olive oil and olive pomace oil were selected to evaluate their efficiency in extracting MPs.

Soil, marine and freshwater sand samples were spiked with 11 different kind of self-made micro-polymers: Polypropylene (PP), low density Polyethylene (LDPE), high density Polyethylene (HDPE), Polyurethane (PU), Polyethylene terephthalate (PET), Polystyrene (PS), Polycarbonate (PC), Polyamide (PA), Polyvinyl Chloride (PVC), Polymethyl Methacrylate (PMMA) and Polytetrafluoroethylene (PTFE). MPs were obtained grinding plastic items previously analyzed with FTIR spectroscopy.

Soil and sand samples (30g) were put in glass cylinders and spiked with MPs. Ultrapure water was added and after shaking the samples, a 3 ml layer of oil was added. The system was then cautiously mixed, allowing the oil to get in contact with the sediment/sand. After few minutes of settling down, the oil layer on top of the water with the MPs in it was collected and filtered on glass microfiber filter (GF/C, 47 mm diameter, Whatman). The filters were then carefully rinsed with hexane to remove oil traces, and finally with ultrapure water. The filters were dried and analyzed using an Alpha-P FTIR equipped with ATR (Brucker), which allows to detect and identify MPs.

3. Results

The preliminary results show that no traces of oil were left on the filters and that the FTIR analysis was free of interferences. The use of hexane did not modify the size or the FTIR spectra of MPs. High recovery rates were achieved for almost all polymers. On the contrary, the method turned out to be inefficient in extracting PTFE. Further developments of this promising method are currently in progress.

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