



Sampling and sample preparation for analysis of microplastics in soils

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Despite abundant evidence of the occurrence of microplastics (MP) – these are particles smaller 5 mm – in aquatic environments, little is known about the accumulation of plastic in terrestrial environments, especially in soils. Possible major input pathways could be the use of plastic mulching, the use of compost, sewage sludge or residues from biogas facilities as fertilizers, as well as littering in urban areas. To estimate the MP pollution, the development of reliable, fast methods for sampling, sample preparation, and detection is needed. The obtained data must be representative of the sampled environmental compartment and measurements from different environmental compartments must be comparable. A first breakthrough is an application of ThermoExtractionDesorption-Gas Chromatography-MassSpectrometry (TED-GC-MS) for the detection of MP, including tire abrasives. This method allows the determination of mass content within a few hours and only a minimum of sample preparation for samples from aquatic environments is needed.

However, in contrast to filtrate samples from aquatic environments, sediment or soil samples need an enrichment of MP. Whereas MP concentration from marine sediments can be obtained by floatation and density separation techniques using NaCl solutions, the extraction or separation from soils proves to be more difficult, as plastic particles are often part of organo-mineral aggregates within the soil matrix.

The aim of this study is the development of a practicable processing guideline for representatively taken soil samples in order to concentrate microplastics, without complex and time-consuming treatment steps. Dispersants or detergents can be applied to decompose the soil matrix, but each preparation step carries the risk of cross-contamination of the sample and prolongs the preparation procedure.

For this reason, we choose ZnCl₂-solution with a density of 1.7 g/cm³, which include the densities of relevant MP types (0.9-1.7 g/cm³). It was tested to achieve both, disaggregation and separation as it decomposes organic material and dissolves carbonates. Also, ZnCl₂ is inert to the precipitation of undesirable salts and carbonates during the process of density separation, as polytungstate solution does. ZnCl₂ can be reused after stepwise filtering (7 μm, 1.5 μm, 0.7 μm). Thus, disposal costs can be reduced. Efficiency and reproducibility of the sample preparation as well as the degradation behavior of MP under the present conditions were demonstrated with model samples.

Real sampling campaigns were conducted at several agricultural sites and floodplains in south-west Germany. The sampling was performed according to practice for soil sampling, using adequate sampling strategies (pattern of sampling, number of field samples, homogenization, etc). The lab sample was fractioned into three size classes (5-100 μm, 100-1000 μm, and 1-5 mm). The identification and determination of mass fraction were done using TED-GC-MS.