Density Separation of Microplastics from Solid Sample Matrices

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Aim of the Study

A reliable method for the analysis of MPs in environmental samples, especially in Solid Sample Matrices (e.g. soil), was not established until today, leading to a lack of comparability of studies. The aim of this study was to examine and improve previously applied methods based on Density Separation to isolate conventional and biodegradable MPs (> 500 µm) from different Solid Sample Matrices with solutions of varying density.

Material & Methods

Mixing of dried proportions for sand substrate artificial soil (OECD 222) sieving of compost < 1 mm

Cryo milling of plastic products
Preparation of two polymer composite samples:
conventional polymers: PE, PP, PET, PVC
biodegradable polymers: PLA, PBS, MB

Addition of microplastic composite samples and water to substrates
Drying (60 °C, 24 h)

Stirring (400 rpm, 30 min) with density solutions (1000 mL):
H₂O, NaCl, SHMP, NaBr
Setting (24 h)
Removal of top layer with spoon, vacuum filtration
Second removal of top layer with peristaltic pump**

Digestion with hydrogen peroxide (60 °C, 24 h)**
Vacuum filtration**
Second density separation with applied density solution (500 mL), manual stirring and removal of top layer after few minutes**

Analysis of microplastics with stereomicroscope and ATR-FTIR

Results

• Highest recovery rates were achieved with NaBr (ρ = 1.53 g/cm³)
  - Conventional polymers (colored):
    94.7 % ± 10 %
  - Biodegradable polymers (translucent):
    57.6 % ± 32 %
• Influence of the method on some polymers (PE, MB) by treatment with H₂O₂ at 60 °C and by mechanical influence
• Density of density solution recovery rates
• Content of organic matter recovery rates

![FIG 1: Method procedure for the recovery tests of MPs in Solid Sample Matrices by Density Separation.](image)

![FIG 2: Results for the recovery tests of MPs in different Solid Sample Matrices by Density Separation with NaBr.](image)

Conclusion

Complex solid matrices with increasing organic content (Compost > OECD 222 > Sand) make separation of Microplastics (MPs) more challenging. Consequently, removal of organic matter is a crucial step for sample preparation. Furthermore, recovery rates were higher for conventional than for biodegradable polymers. Due to the results of influence tests, it is concluded that lower recovery rates of biodegradable polymers mainly depend on appearance of polymers with reduced visibility during analysis of samples and not on chemical properties of the polymer. An automated chemical analysis could improve the method by decreasing visual dependency. Besides, beakers were not detected to be suitable for density separation as stirring led to fragmentation of Microplastics and corrosion of beakers. For further experiments, application of density solutions with ρ ≥ 1.5 g/cm³ are recommended.

![FIG 3: Results for the recovery tests of MPs in artificial soil (OECD 222) by Density Separation with different solutions of varying density.](image)