Detection of microplastic particles (1-10µm) in soil matrices

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Quantifying the number of small microplastic particles (MP) in complex matrices is challenging. We, therefore, developed a method based on automated scanning electron microscopy (SEM) to assess MP between 1 and 10µm. We suspended 1-10µm sieved polyethylene (PE), polyvinylchloride (PVC) and Lufa 2.4. soil in different proportions as proxies for contaminated soils. These suspensions were filtered through 40nm gold-coated membrane filters (Nuclepore, Whatman) and imaged using an SEM operated at an acceleration voltage of 3kV. These experimental conditions were derived from Monte Carlo simulations of the interaction of the electron beam with solid materials using the software code CASINO (v. 3.3.0.4) [1] (Figure 1). Particle detection, based on the backscattered electron signal and elemental analysis, using a windowless energy dispersive x-ray (EDX) analysis system (X-TREME, Oxford Inst.), were automated using the software code 'feature' (Oxford Inst.). The classification was based on the carbon to oxygen ratios and on the presence of elements such as silicon or chlorine (Cl).

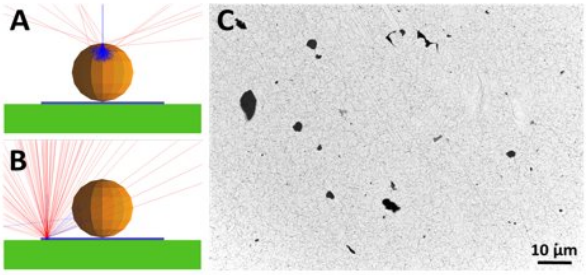


Figure 1: A-B) Monte Carlo simulations of the interaction of the electron beam (blue, vertical line) with a 1µm polyethylene particle (PE, orange) deposited on a membrane filter (Nuclepore (NP), green) coated with 40nm of gold (horizontal blue bar). The backscattered electrons (BSE) are given in red. C) BSE image of PE particles deposited on a gold-coated (40nm) NP filter. The acceleration voltage for the simulations and the experiments was set to 3kV.

Pure suspensions: In the PVC sample, 70% of the particles were identified as PVC and 15% as PE and soil particles, each. In the PE sample, 85% were identified as PE and 15% as soil particles and in the pure soil sample, 93% of the particles were classified as silicates and 7% as PE.

MP mixtures in soil: The dynamic range of the method was assessed by mixing MP (stock suspension containing equal amounts of PE und PVC MP) with soil suspensions in a 1:1 and a 1:10 ratio. The quantified ratio between PE and PVC in all experiments (pure MP mixtures, 1:1 and 1:10 mixtures with soil particles) ranged between 0.7 and 1.2. The obtained ratios between soil and MP in 1:1 and 1:10 mixtures were 1.1 and 8.5, respectively. These results demonstrate that the MP were correctly identified also in the presence of a 10-fold excess of soil particles. The reduced accuracy of the PVC identification is probably caused by the volatility of Cl under the electron beam. We are currently developing sample preparation protocols to enrich the MP content in soils, which will allow assessing the contents of small MP in field samples.

[1] Demers, H., et.al. (2011), Three-dimensional electron microscopy simulation with the CASINO Monte Carlo software. Scanning, 33: 135-146