Microplastics in Soils

DENSITY SEPARATION OF SOILS AS SAMPLE PREPARATION FOR THE DETERMINATION OF PLASTICS

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1. Method Development

The determination of microplastics in soil using thermogravimetric detection methods (TED-GC-MS) has to deal with three key challenges:

1. Low microplastic content in soil [mg/kg]
   + inhomogeneous distribution
   + formation of agglomerates (mineral and organic)

2. The TED-GC-MS only works with small sample masses [mg] [1] , [2]
   + plastics content of soil below the method's polymer specific limits of detection (LOD)

3. Organic contents falsify the TED-GC-MS results with respect to several polymers

Our developed methods tackles all three challenges, putting additional focus on the representativity of the samples. It addresses:

1. the low microplastic content in soil, the inhomogeneous distribution, and the formation of agglomerates

We use sieves to homogenize the sandy soil sample and to break up existing agglomerates.

We use a sample separator (see Figure 1) to divide the original sample into 8 samples with equal masses. We then combine 3 sub samples and repeat the procedure, until we reach a sample mass of less than 100 g.

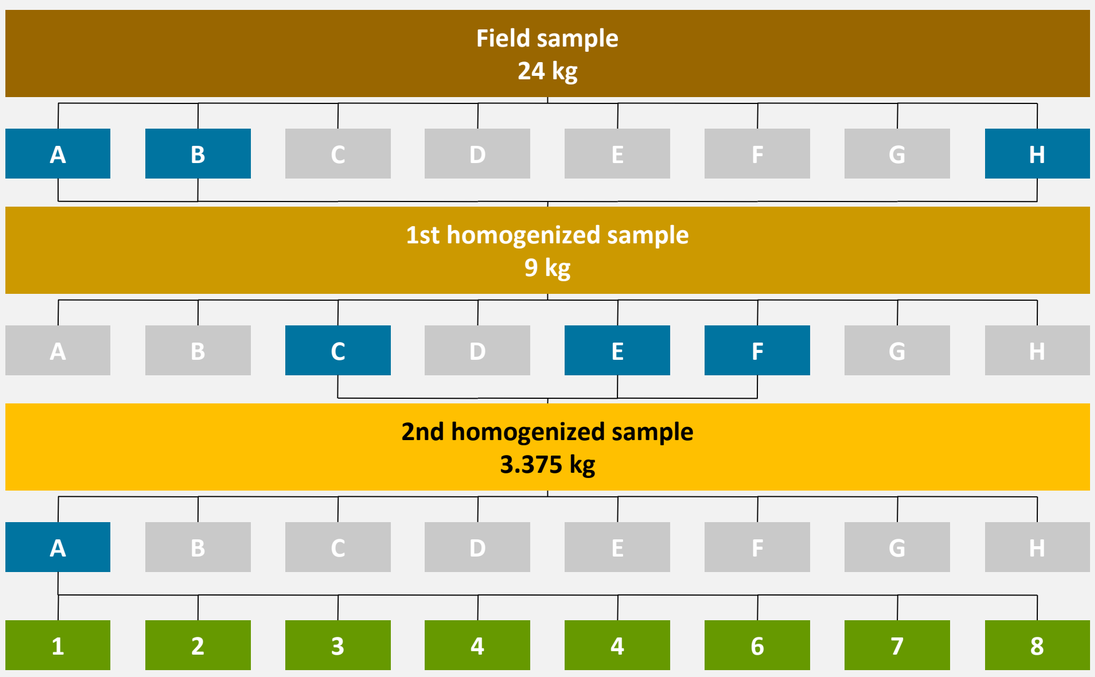

Figure 1: reduction of the sample using a sample separator

The next step is to verify the representativity of our sample division process by comparing the temperature specific weights of our sub samples (8 in total, 3 shown in Figure 2) using a TGA routine (ignition loss).

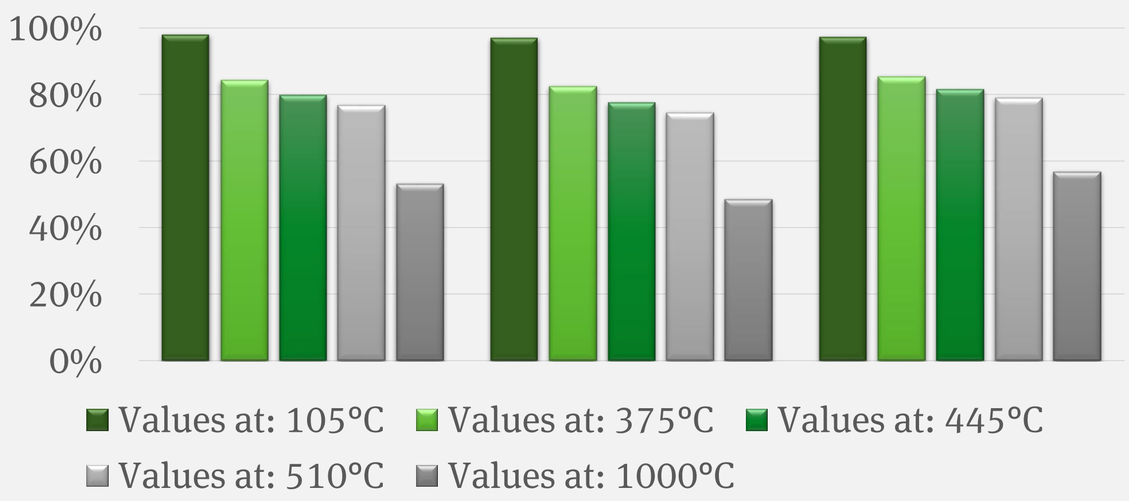

Figure 2: temperature specific masses of 3 samples (out of the 8)

After the generation of a representative sample in the first step, we now focus on challenge

2. small sample masses and a plastics content that is usually below the TED-GC-MS LOD

We use a density separation method to increase the plastics content in the flotation tailings, also further reducing the sample mass. Figure 3 shows the density separation process. We use a sodium iodide based density solution and the sample generated in the first step. After an ultrasonic treatment and a 24 h resting time, we remove the generated sample using a novel suction apparatus (details in appendix). We use a 5 µm stainless steel filter to separate the solid sample components from the density solution, the latter may be reused.

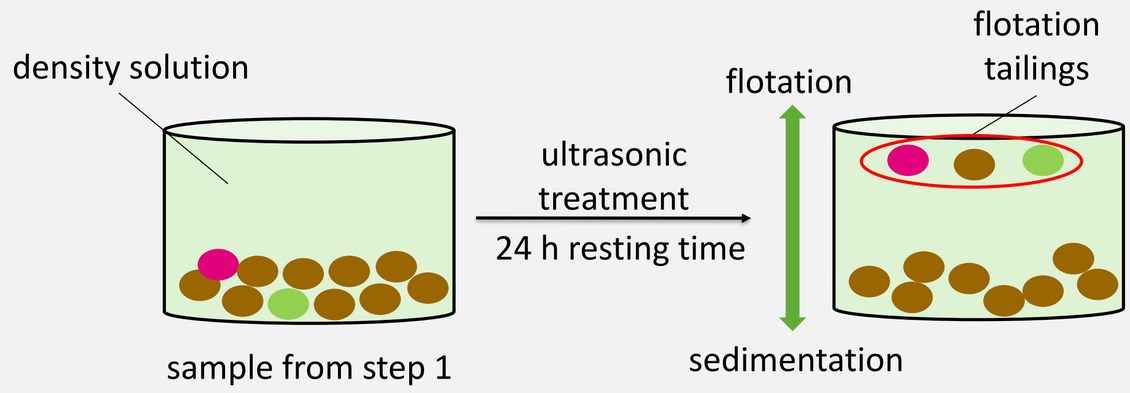

Figure 3: overview of the density separation method

After drying the flotation tailings, we can tackle the final challenge

3. the organic content falsifying detection results with respect to selected polymers

We use a 20% H2O2 solution on the flotation tailings for a maximum of 1 hour, carefully monitoring the temperature (see Figure 4). Then, we use the 5 µm stainless steel filter to separate the solids from the solution and rinse it thoroughly with deionized water.

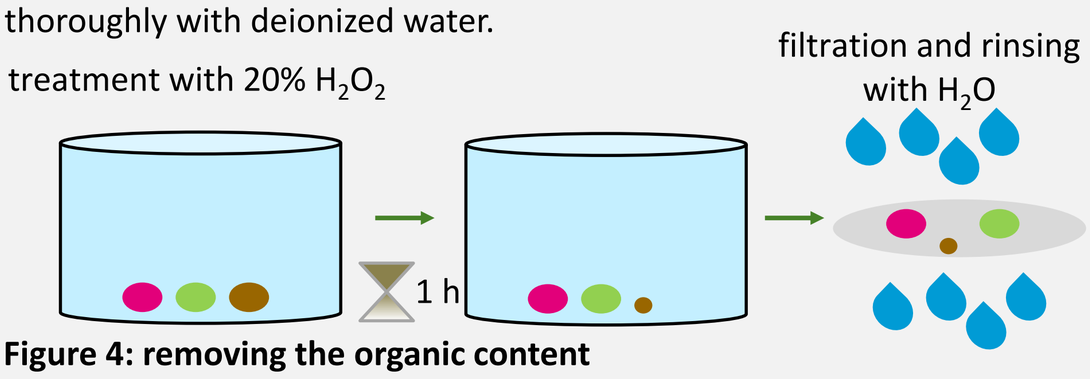

Figure 4: removing the organic content

After carrying out all three steps we may analyze the sample using thermogravimetric methods. Our generated sample is representative for the total sample, has enough microplastics to match the LODs of the TED-GC-MS and has less organic compounds, which falsify the measurements.

2. Validation

We validated our approach with recovery experiments using different density separation solutions (NaCl, NaI, K(HCOO)) and spiked soil samples (PE, PS in a 1 : 1 ratio). We achieved a:

- ❖ reduction of the organic content of 23%
- ❖ reduction of the mineral content between 29% and 43%
- ❖ a mass loss between 375°C and 510 °C of 82% - 89% (compared to 6%-10% of the blank)
- ❖ plastics recovery rates of 77% to 82%
- ❖ overall plastics distribution: 46% PS, 54% PE

References:

[1]: R. Becker et al. 2020 – Quantification of microplastics in a freshwater suspended organic matter using different thermoanalytical methods – outcome of an interlaboratory comparison

[2]: Dümichen et al. 2016 – Fast identification of microplastics in complex enviromental samples by a thermal degradation method

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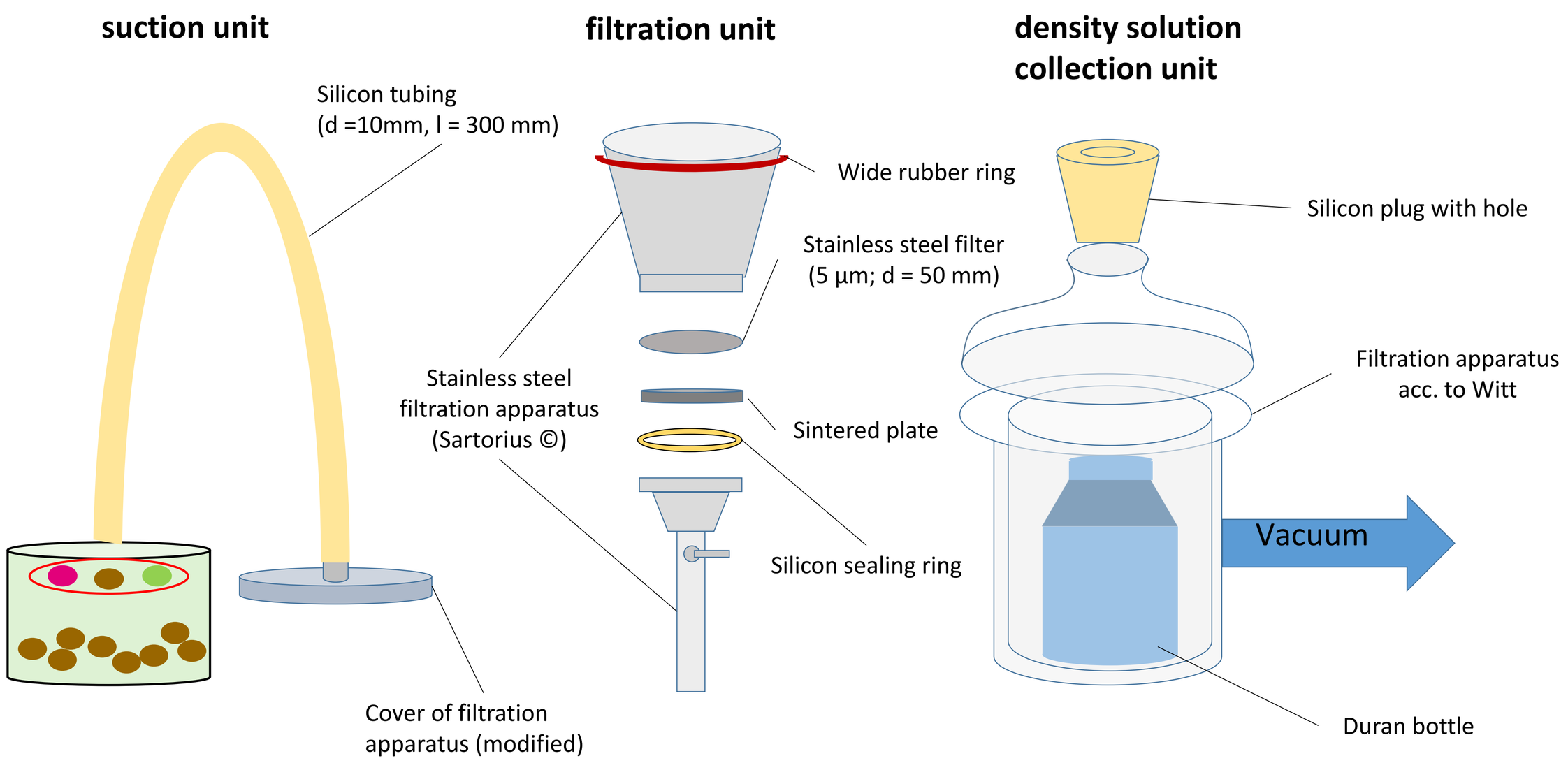
Appendix:
The novel suction apparatus used



collection of flotate with aspirate apparatus:

- integrated 5 µm stainless steel filter
- clean up sample in same apparatus
- recovery of density solution