ANALYSIS OF PARTICLE FORMATION IN FRAGMENTATION TESTS

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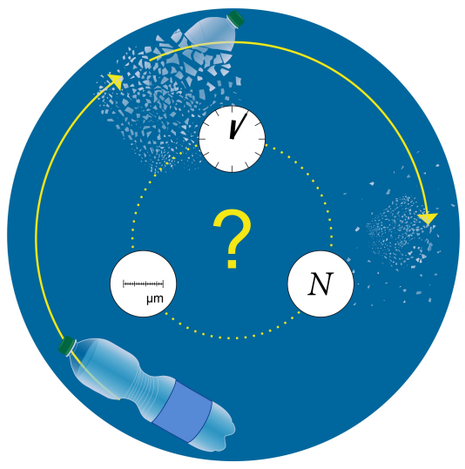
IDEA

Experiments with a fragmentation test setup with abrasive → Extraction of generated polymer fragments by density separation → Particle analysis with dynamic image analysis

MOTIVATION

- 10.5 Mt of macroplastics enter the sea annually [1] → could potentially fragment into microplastics and further into nanoplastics
- One of the hotspots of fragmentation are beaches, where sand acts as an abrasive [2]
- How fast and in how many particles a piece of plastic fragments is important for estimating the potential effects of plastics in the sea
- Studies investigating the fragmentation of plastics
  - experiments with an abrasive
    - the weight or volume of the remaining macroscopic material is compared with the initial state [3]
      - determination of the amount of material removed by fragmentation
    - no information about the generated particles
  - experiments without abrasive with additional energy input
    - generated particles can be easily analyzed due to the lack of other particles interfering with the measurement
    - Disadvantage: energy input, e.g. in the form of ultrasound [4] or through a mechanical impeller [4,5], does not suit the real conditions
  - I. Chubarenko [6] and I. Efimova [7]
    - gravel was used as abrasive
    - Generated particles could be analyzed (separation by size)
    - Only possible if abrasive is coarser than polymer fragments
- **Aim**: Access the generated particles even when a fine abrasive, like sand, is used in the fragmentation test



FRAGMENTATION TEST SETUP

- Roller mixer (figure 1)
- Partially filled glass bottles
- 30 g artificial sand (size between 210 µm and 297 µm)
- 50 ml artificial seawater
- 30 polymer pellets (e.g. polystyrene (PS))
- Rotation for 24 hours
- 70 rounds per minute (roller mixer) = 50 rounds per minute (bottles)
- Pellets are separated with a 1 mm filter
- Sand and polymer fragments are separated from water with a 15 µm filter
- Thoroughly rising, drying, weighing
- Next step: density separation

Figure 1: Mixer with glass bottles filled with sand, water and polymer pellets to test the fragmentation.

DENSITY SEPARATION SETUP

Materials

- Glas funnel with plug and valve made of glas (figure 2)
- Faces of the funnel at least 45° inclined
- No lubrication of the valve, only wetting with water before use
- High-density liquid (HDL):
  - Potassium carbonate (K_2CO_3) solution with a density of 1.52 g/cm³
  - Filtered with 5 µm filter before use

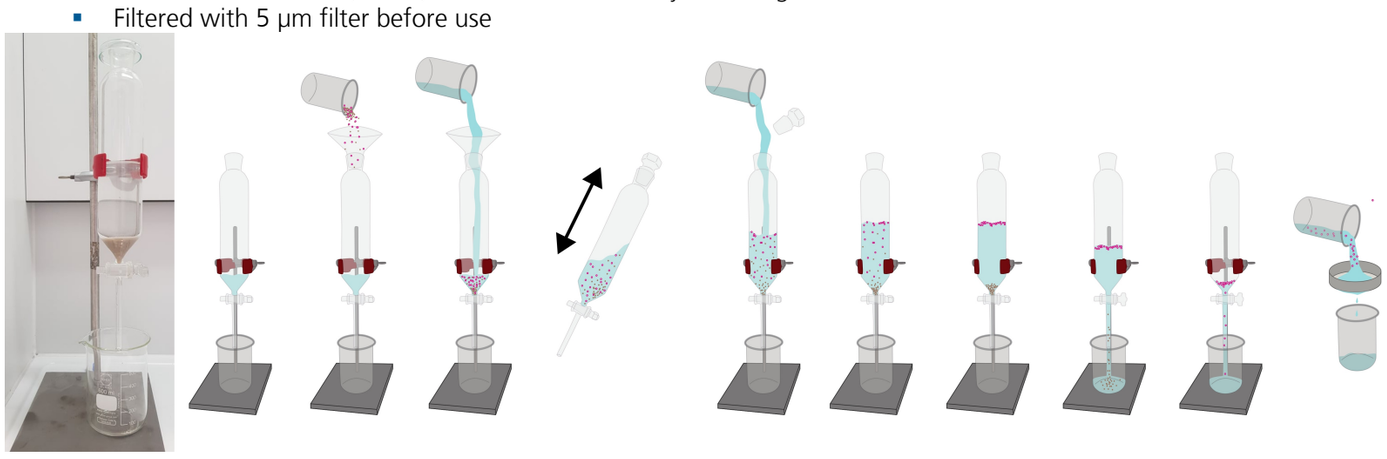


Figure 2: Density separation procedure.

- Setup is based on density separation as a step in the extraction of microplastics from environmental samples presented by Enders et al. [8]
- Subsequent analysis with dynamic image analysis (DIA, device: QICPIC, Sympatec GmbH) to determine the particle size distribution and number of particles

EVALUATION OF THE SEPARATION SETUP

First pilot test: Mass-related recovery rate

- Virgin sand and polymer (PET) particles were mixed
- Mass-related recovery rates: 93.99 % to 98.63 %

Second pilot test: Scanning Electron Microscopy (SEM) and energy-dispersive x-ray spectroscopy (EDX)

- Virgin PS pellets were treated in the fragmentation test setup
- Separated sample contains PS and sand particles (figure 3)
- Size of sand particles is smaller than initial size
  - Sand also fragments
- PS fragments have partly an uneven surface (figure 4)
- Tiny sand particles are incorporated into the polymer surface even at low energies (figure 5)
  - Cannot be separated by density

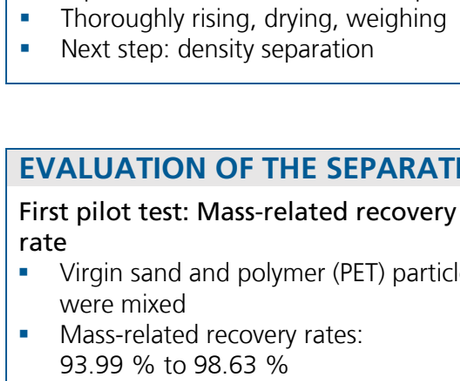


Figure 3: SEM image of PS (dark) and sand (bright) particles.

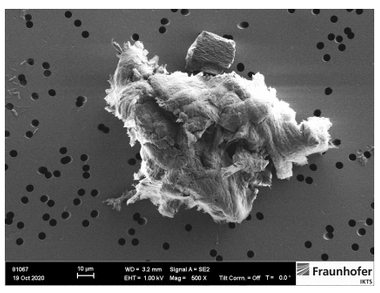


Figure 4: SEM image of PS particle.

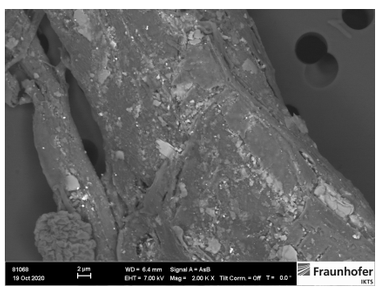


Figure 5: SEM image of sand particles (bright) bonded to a PS particle (dark).

Conclusion

- Absolute number of generated microplastics cannot be determined
- Further experiments will show if the comparison of different samples treated with the setup is possible
- Special attention has to be paid to whether the sand particles always represent a fixed percentage of the fragments

FIRST RESULTS

- Investigation of virgin PS pellets and environmentally aged PS pellets
- Environmental aged pellets were exposed in mesocosms (continuously flown through with pacific water) for 28 days during the voyage of the research vessel SONNE from Vancouver to Singapore

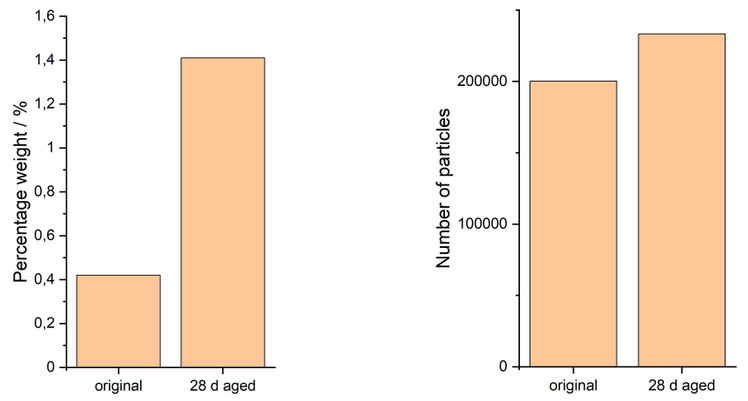


Figure 6: Percentage weight of separated particles from initial weight of pellets.

Figure 7: Number of separated particles.

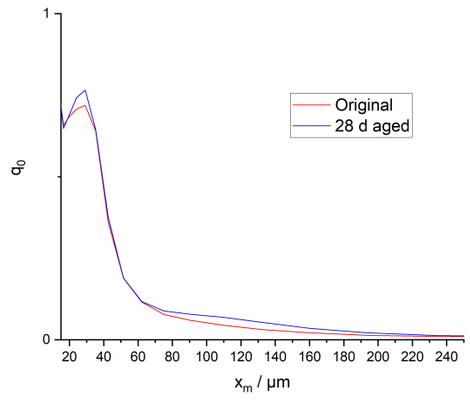


Figure 8: Density distribution of the particle size.

- More fragments were produced during the fragmentation of the aged pellets (figure 6 and 7)
- Maximum number of generated particles for a size of 29,17 µm for virgin and environmental aged sample
- 90 % of the separated particles have a size smaller than 55 µm
- Size of generated fragments is of the same order of magnitude as that measured in the sea [9]
  - Majority of microplastics are < 100 µm, Mean size ≈ 80 µm

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