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Additive Manufacturing of Monodisperse Microplastic Reference Particles Through Micro Extrusion

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Motivation

Need of Reference Materials (RM)

- EU's Green Deal 2050 increasingly requires microplastic (MP) analysis
- RMs are required for calibration, validation and quality control within MP analyses

→ possible solution: new production method using additive manufacturing through micro extrusion of polymer materials [1]

Critical Lack of RMs

- state of the art are two methods: cryo-milled + sieved particles; direct synthesis through emulsion polymerisation of polymer particles
- not monodisperse in particle size, no specific number of particles
- no correlation between mass fraction and particle number to compare mass-based like DSC [2] and particle-counting analysis like RAMAN microscopy [3]

Advantages of additive manufacturing method

Properties/ Requirements	Cryogenic grinding + sieve fractionation	Direct synthesis (emulsions polymerisation)	Additive manufacturing
Particle shape	fragments	spheres	Hemispheres; various shapes possible
Need for additional resources	liquid nitrogen	solvents, filters	-
Particle sizes in fraction	no target size; particle size distribution within sieve limits	small/ monodisperse size distribution	specific, monodisperse size
Specific quantity	no	no	yes
Range of polymer types	almost all	only a few	almost all thermoplastics

- cost-effective due to efficient production, minimal waste and no additional resources
- process of melt extrusion is possible for all thermoplastics
- monodisperse, number-accurate fractions enable correlation between mass and number of particle fractions

Additive manufacturing of microplastic particles

- modified 3D-printing system with high demands on accuracy and precision
- special custom-made nozzle for micro extrusion
- melting polymer filaments (commercially available or self-extruded)
- extruding polymer melt through the nozzle by pushing the filament forward
- depositing small droplets on a surface (glass, metal, foil, water-soluble foil, ...)
- act number of particles produced

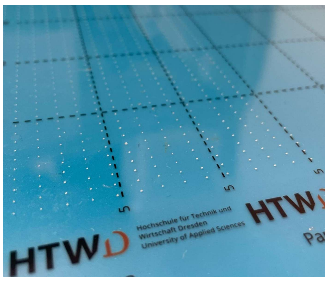

Figure 1: PA6/6.6 particles on a transfer foil to cut a specific amount of particles out

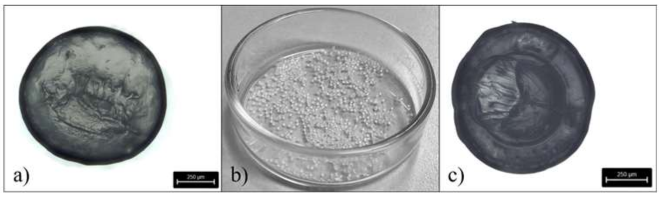

Figure 2: Additively manufactured RM-particles, produced with a 400 µm nozzle a) microscopic image of an LDPE particle; b) exactly 1000 LDPE particles in a petri dish, c) PMMA.

Results of additive RM particles

- 7 Polymers tested (LDPE filament self-extruded)
- 4 nozzle opening diameters
- producing 1000 particles within 1 hour
- hemispherical to drop-shaped particles
- particle sizes from 224 µm to 1349 µm
- adjustable particle size through different nozzle and extrusion parameters
- smallest size produced to date 150 µm (not reproducible)
- average deviation in particle size around 10 % (monodisperse size fraction)
- first tests with smaller nozzles down to 10 µm

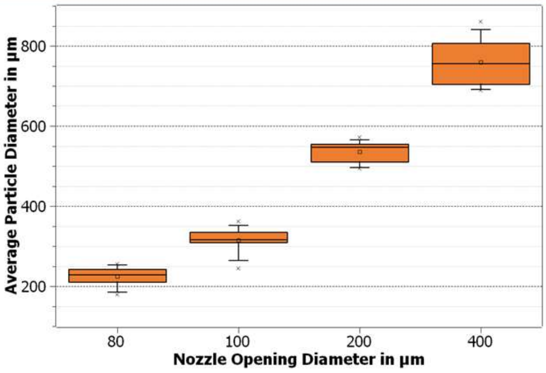

Figure 3: Sizes of PLA particles manufactured with different nozzle opening diameters

Nozzle dia-meter in µm	Polymer with particle sizes in µm (mean value ± standard deviation)						
	LD-PE	**PP**	**PA6/6.6**	**PLA**	**PLA glow**	**PCL**	**PMMA**
400	991 ± 32 (± 3 %)	913 ± 166 (± 18 %)	-	759 ± 59 (± 8 %)	1213 ± 67 (± 5 %)	1349 ± 58 (± 4 %)	-
200	566 ± 62 (± 11 %)	335 ± 13 (± 4 %)	703 ± 155 (± 22 %)	536 ± 27 (± 5 %)	472 ± 59 (± 12 %)	702 ± 105 (± 15 %)	902 ± 43 (± 5 %)
100	308 ± 26 (± 9 %)	-	575 ± 75 (± 13 %)	315 ± 32 (± 10 %)	-	-	-
80	-	-	-	224 ± 25 (± 11 %)	-	-	-

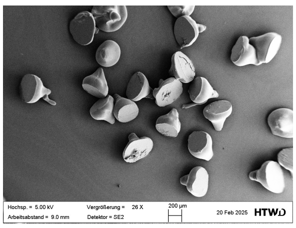

Figure 4: scanning electron microscope image of drop-shaped LDPE particles (26x magnification)

Conclusion

- new and unique reference material for microplastic analysis
- number accurate particle fractions
- particle sizes from approx. 200 µm to 1400 µm
- monodisperse in particle size (± 10 %)
- possible for all thermoplastics
- fast and cost-effective production with minimum waste

Outlook

- expansion of the range of polymers
- smaller particle sizes (< 100 µm)
- automation of the whole process
- inline-process-control

Check out our research:

PrePrint-Paper DOI: 10.21203/rs.3.rs-6411430/v1

References

[1] Harre, K., Himmer, T., Hauffe, M., Vorrichtung und Verfahren zur Herstellung von Polymerpartikeln und Verwendung von Polymerpartikeln als Polymerpartikelstandard, DE 10 2023 118 036, 18.01.2024
[2] Schirrmeister, S., Kurzweg, L., Gjashta, X., Sochere, M., Fery, A., Harre, K.: Regression analysis for the determination of microplastics in sediments using differential scanning calorimetry. 2024. Environmental Science and Pollution Research. 10.1007/s11356-024-33100-8.

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