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Raman-Assisted Assessment of Microplastic Extraction Utilizing Natural Deep Eutectic Solvents

MOTIVATION

- One challenge in reliable measurement of microplastics is the low concentration of particles in the environment [1]
- The extractive enrichment of microplastics from aqueous samples could be a way of increasing the microplastic concentration to enable subsequent analysis
- In order to measure the extractive enrichment of polymer mixtures, suitable analytical methods must first be established

AIMS

- Raman spectroscopy as an analytical method for the quantification of different polymers in aqueous suspension
- Development of an indirect hard model to analyze the spectroscopic data [2]
- Calibration and validation of the model
- Natural deep eutectic solvents (NADES) for the extractive enrichment of microplastics
- *In-situ* measurement of the extraction kinetics

INDIRECT HARD MODEL FOR MICROPLASTIC ANALYSIS

Production of microplastics → **Indirect hard model** → **Calibration & validation**

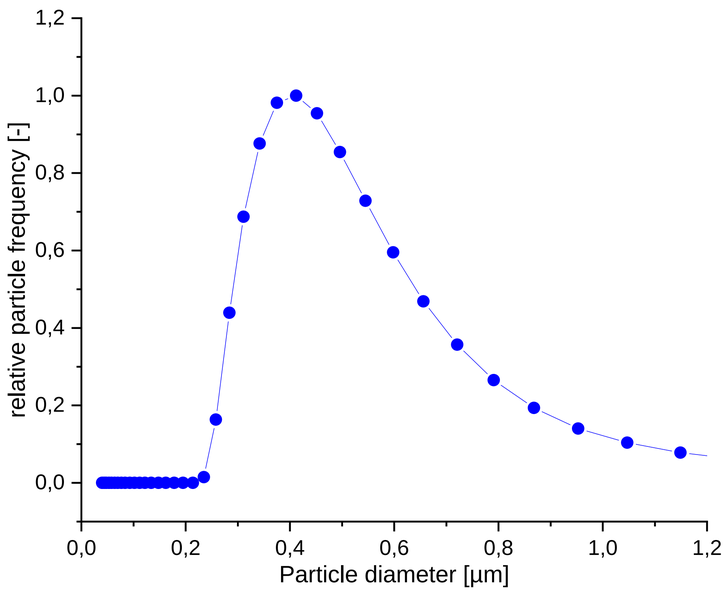

Fig. 1: Polystyrene was dissolved in tetrahydrofuran and precipitated in water, particle size distribution was recorded with Beckman Coulter LS230

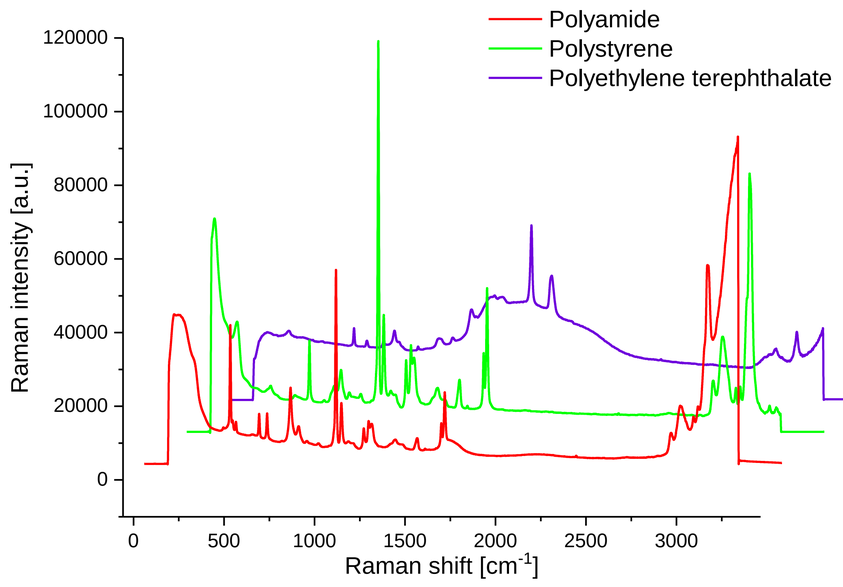

Fig. 2: Pure substance Raman spectra of polyamide, polystyrene and polyethylene terephthalate

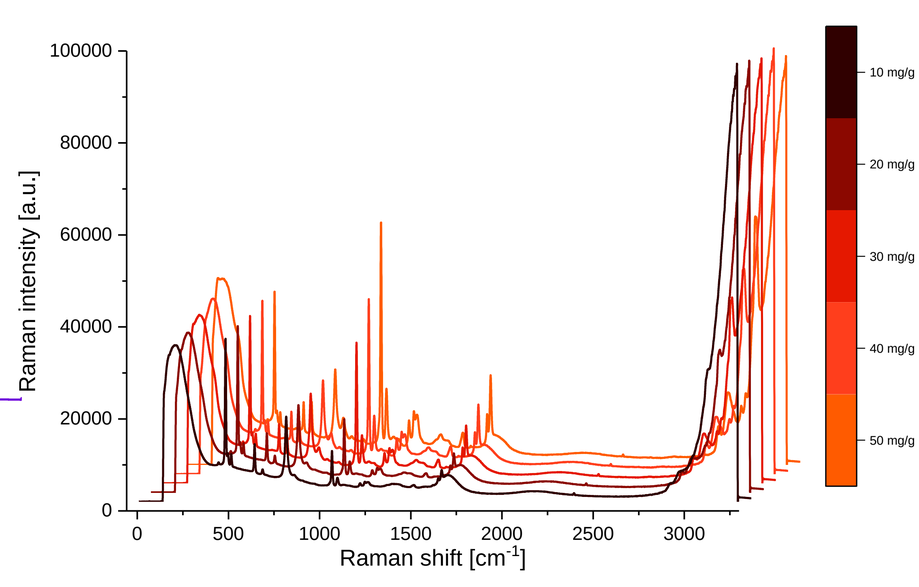

Fig. 3: Raman spectra of polystyrene with different mass fractions stabilized in water with 1 g/L sodium lauryl sulphate (SDS)

- Production of microplastic samples
- Nanoprecipitation
- Cryogenic mill

- Establishing an indirect hard model
- Pure substance spectra
- Model is fitted to the spectra

- Calibration of the model using pure and mixed microplastic suspensions
- Model validation with samples of known composition

EXTRACTION WITH NATURAL DEEP EUTECTIC SOLVENTS

- Extraction of microplastics with green solvents
- Affinity to the organic phase due to the hydrophobic character of the particles
- Natural deep eutectic solvents from terpenes and carboxylic acids [3]
- Thymol & octanoic acid

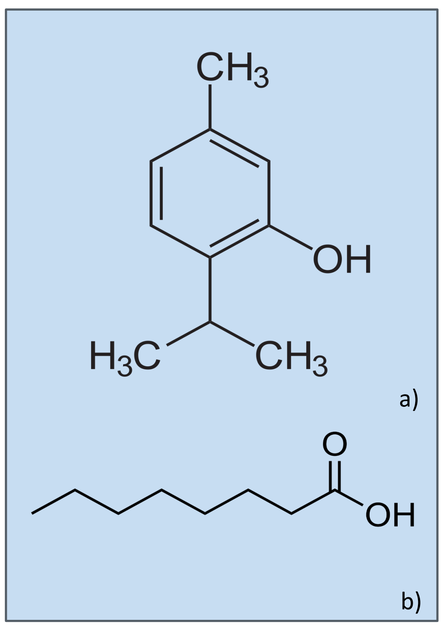

Fig. 4: Structural formulas of a) thymol and b) octanoic acid

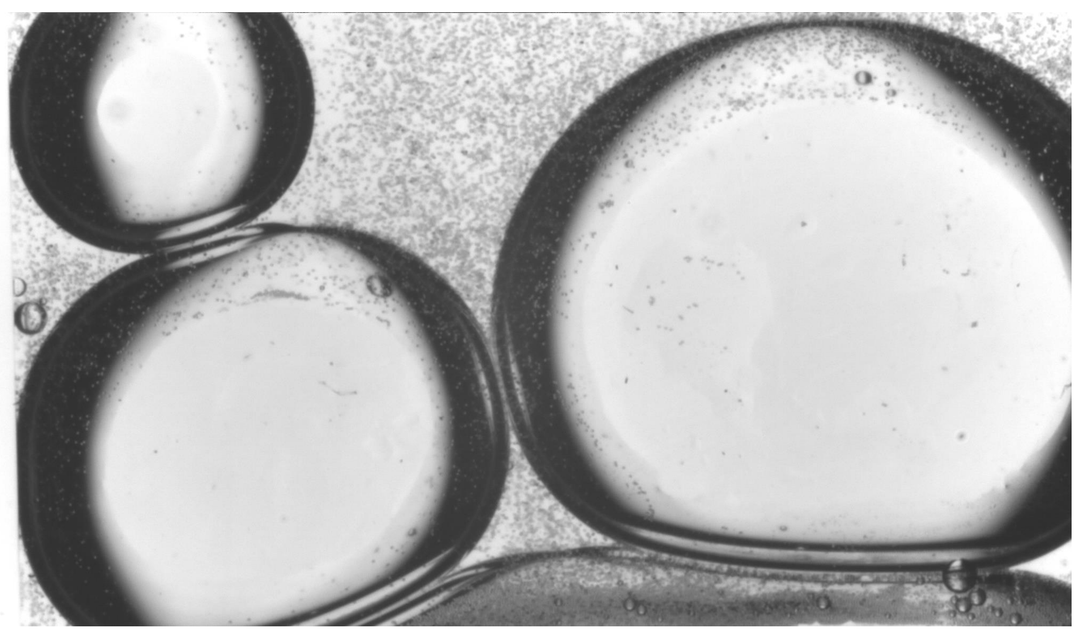

Fig. 5 Extraction of polystyrene microplastics (d=20 µm) with thymol : octanoic acid (1:1) from water

SUMMARY

- Production of microplastic suspensions and quantification using Raman spectroscopy possible

OUTLOOK

- Calibration via polymer mixtures
- Validation of the model using samples of known concentration
- Extraction of polymer mixtures

Funded by
Bundesministerium für Bildung und Forschung
PlastiQuant

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[2] Alsmeyer, F., Koß, H. J., & Marquardt, W. (2004), Applied spectroscopy, 58(8), 975-985.
[3] Van Osch, D. J., Dietz, C. H., Van Spronsen, J., Kroon, M. C., Gallucci, F., van Sint Annaland, M., & Tuinier, R. (2019), ACS Sustainable Chemistry & Engineering, 7(3), 2933-2942.

Annual Meeting DECHEMA-Group Extraction
14. - 15. February 2024,
Technische Universität Dresden, Germany