WP 2: Validation of methods for plastics analysis in environmental samples

Results of the EUROqCHARM/QUASIMEME/NORMAN Interlaboratory Study on the Analysis of Microplastics in Environmental Matrices

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This project has received funding from the European Union's Horizon 2020 coordination and support action under grant agreement No. 101000805 (EUROqCHARM).
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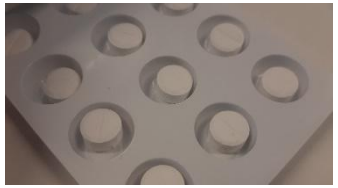
Test materials

Tablets

QMP010SW	µg added to tablet	particle size (µm)
Polyethylene (PE)	36	50 – 299
Polyethylene terephthalate (PET)	32	50 – 299
Polystyrene (PS)	75	50 – 299
QMP011SW	**µg added to tablet**	**particle size (µm)**
Polypropylene (PP)	41	50 - 299
Polycarbonate (PC)	72	50 - 299
Polyvinylchloride (PVC)	72	50 - 299
QMP012SW	**µg added to tablet**	**particle size (µm)**



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Participants

Participating laboratories
n =98

(ILS 1: n=30, ILS 2: n= 35)

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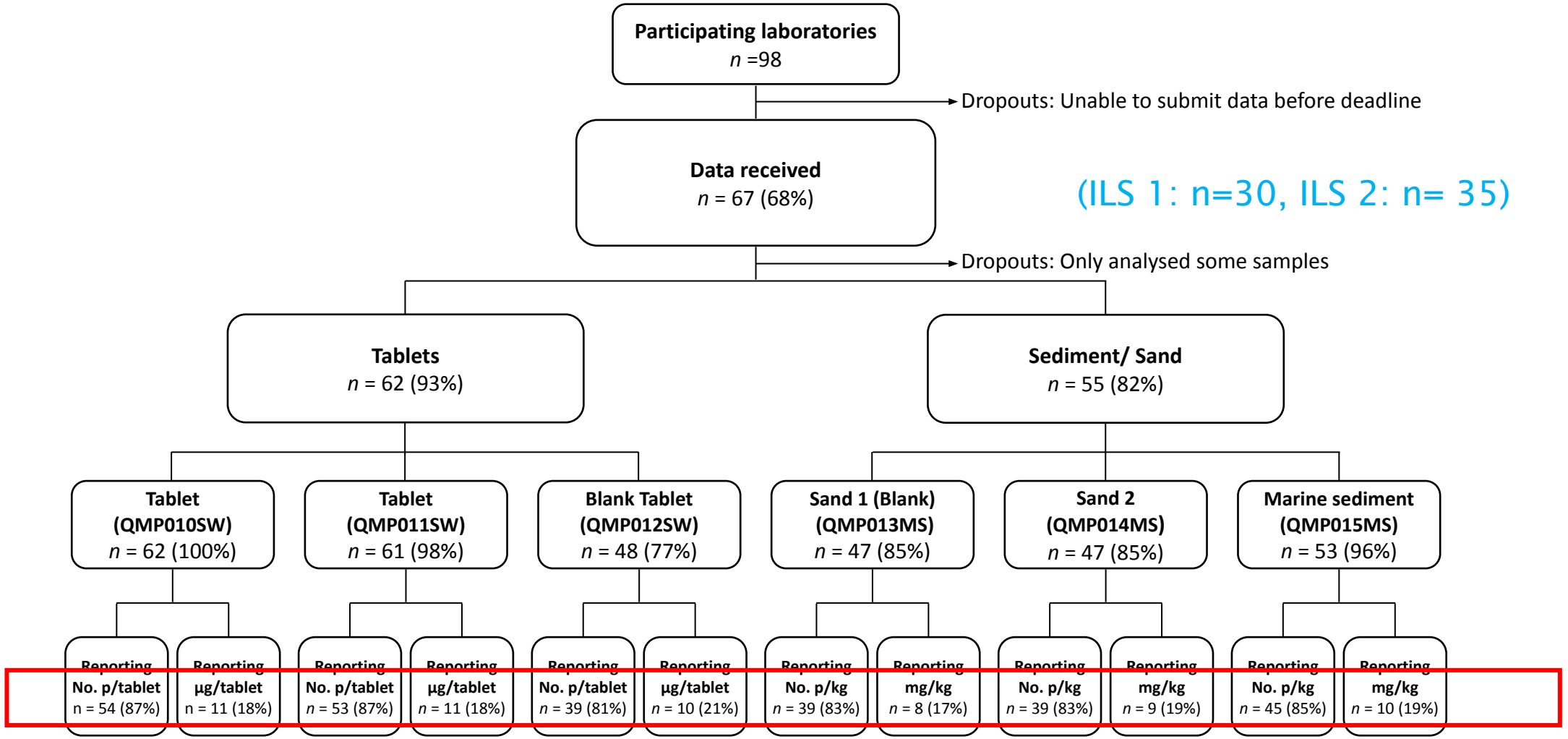
Participants



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NDA rel. St.dev%

	Number				Mass			
	tablet QMP010SW	tablet QMP011SW	sand QMP014MS	sediment QMP015MS	tablet QMP010SW	tablet QMP011SW	sand QMP014MS	sediment QMP015MS
PE (50-299 µm)	96		122		NAV [2]		NAV [3]	
PE (300-5000 µm)				72				25
PET (50-299 µm)	92		102		NAV [3]		NAV [3]	
PS (50-299 µm)	76		108		12		NAV [3]	
PP (50-299 µm)		81		125		NAV [3]		NAV [3]
PC (50-299 µm)		90		66		93		NAV [3]
PVC (50-299 µm)		81		79		NAV [3]		77

= No assigned value

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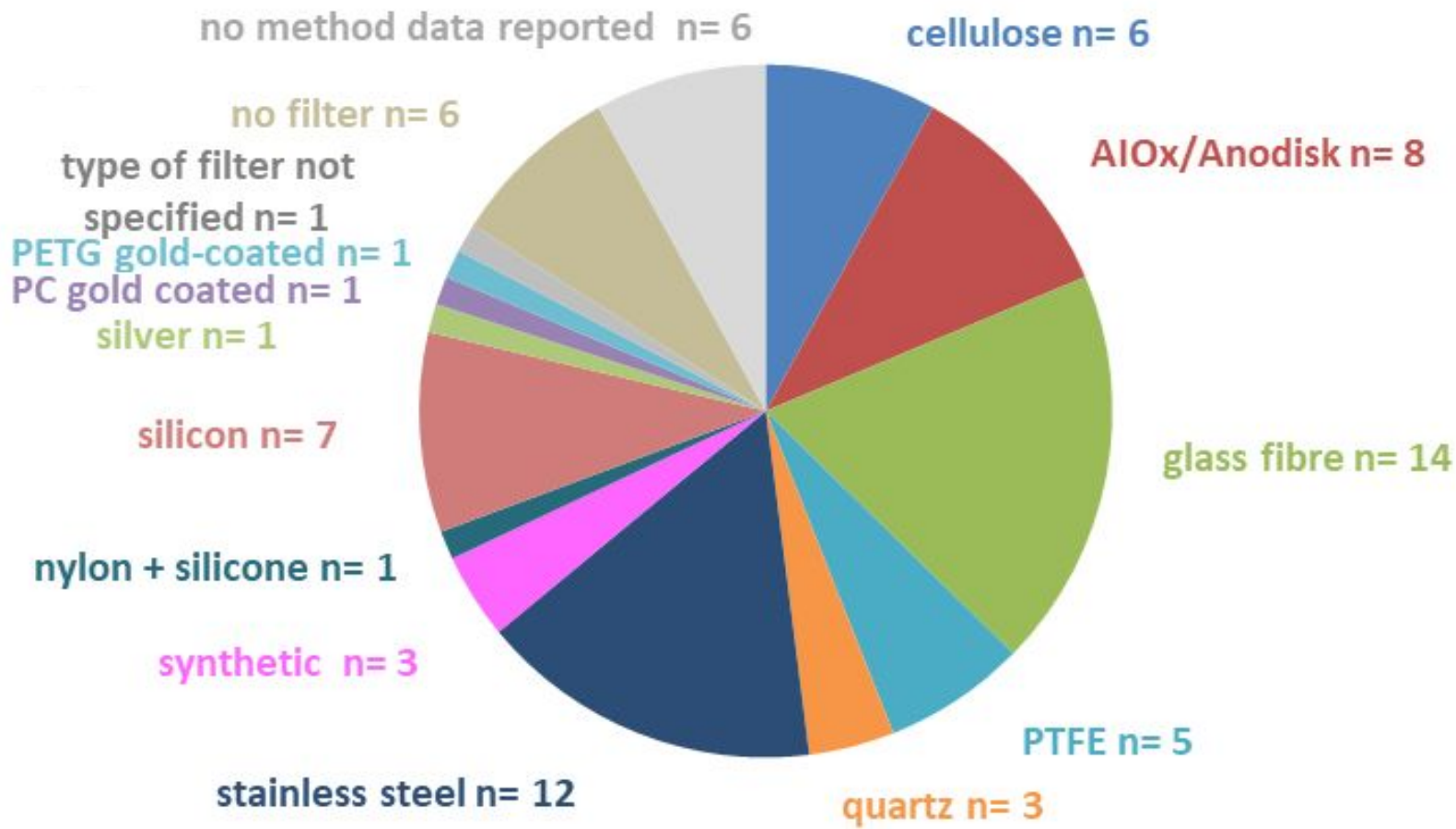
Filter type

Methods for tablets, sediment and sand samples



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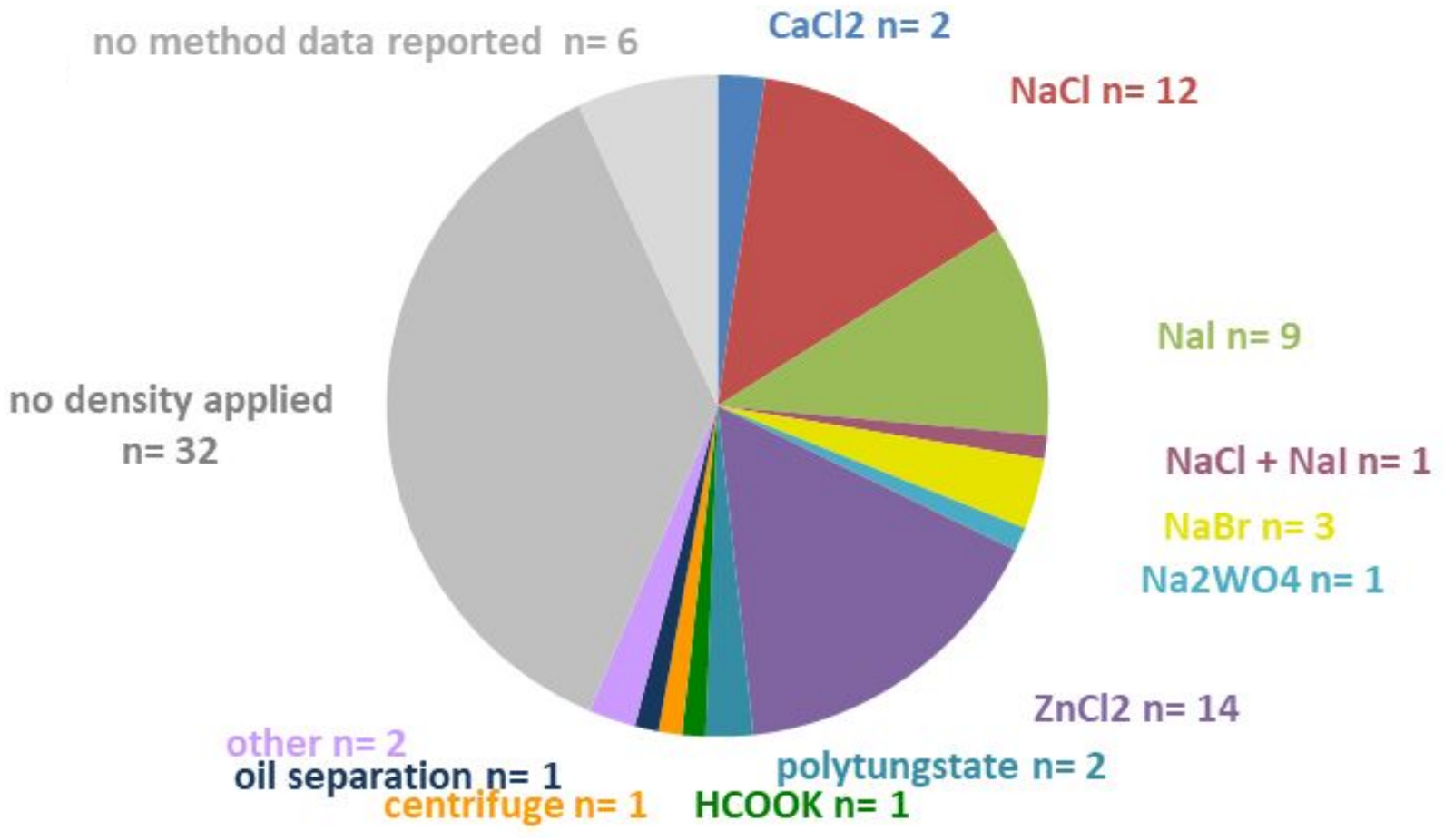
Density separation

Methods for tablets, sediment and sand samples



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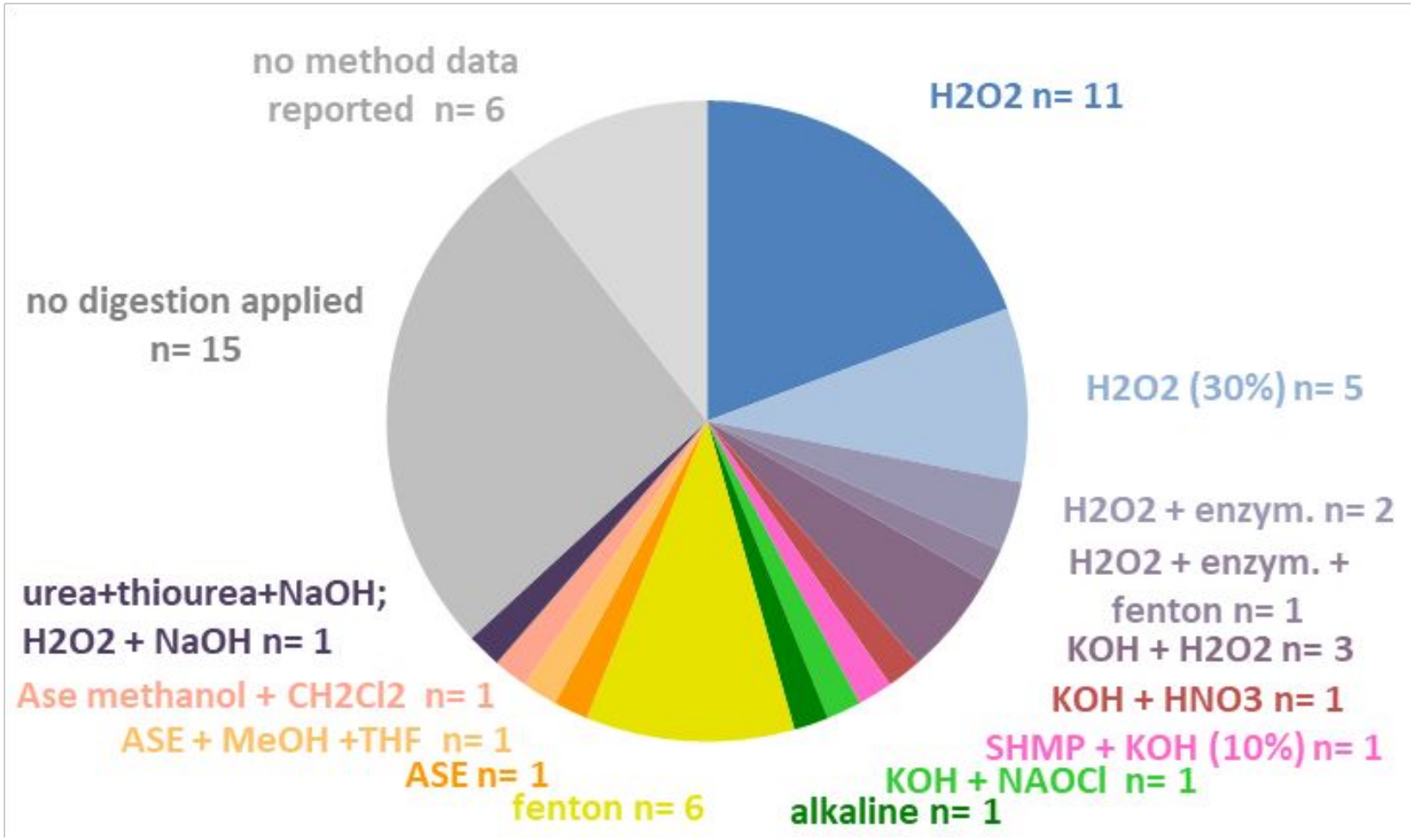
Digestion

Methods for sediment and sand samples



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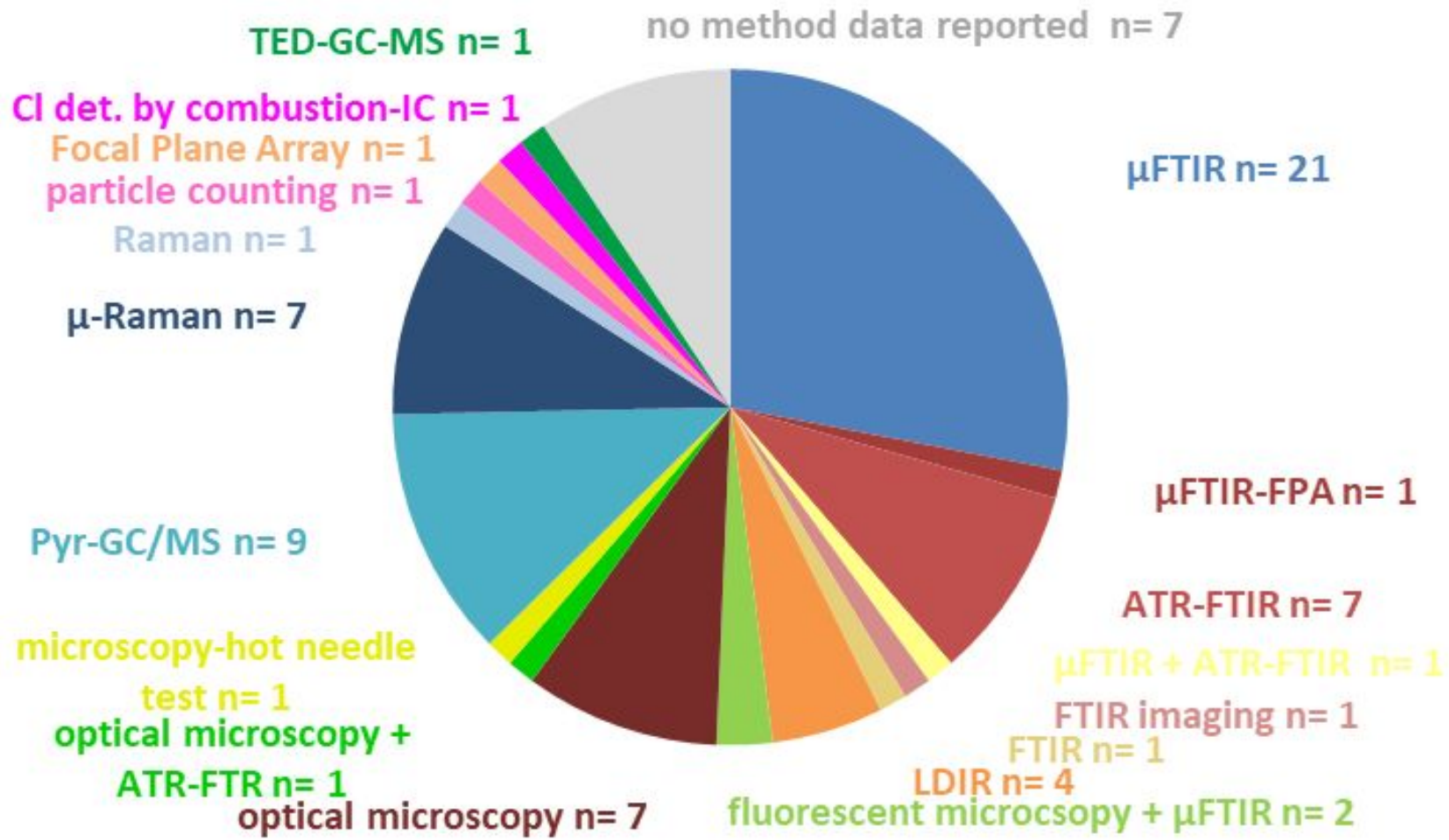
Detection

Methods for tablets, sediment and sand samples



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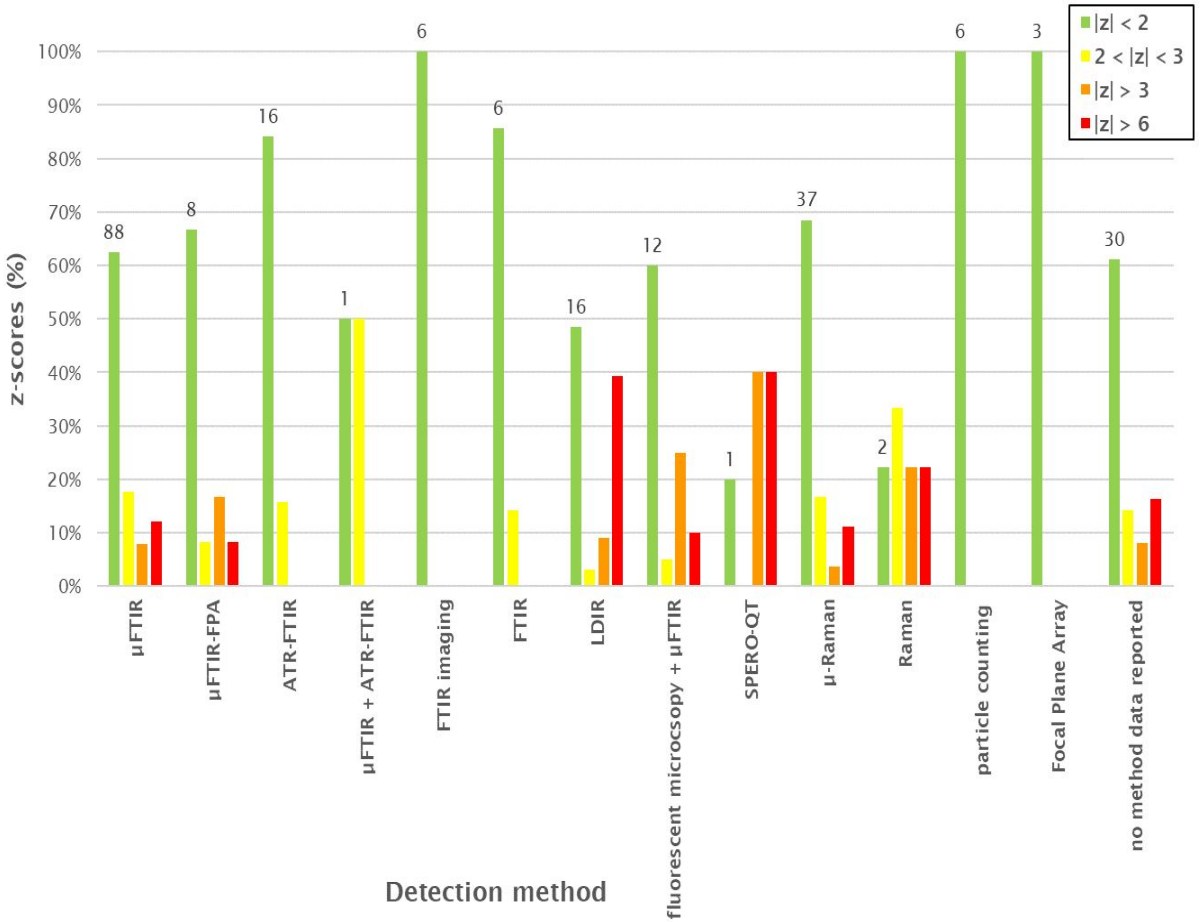
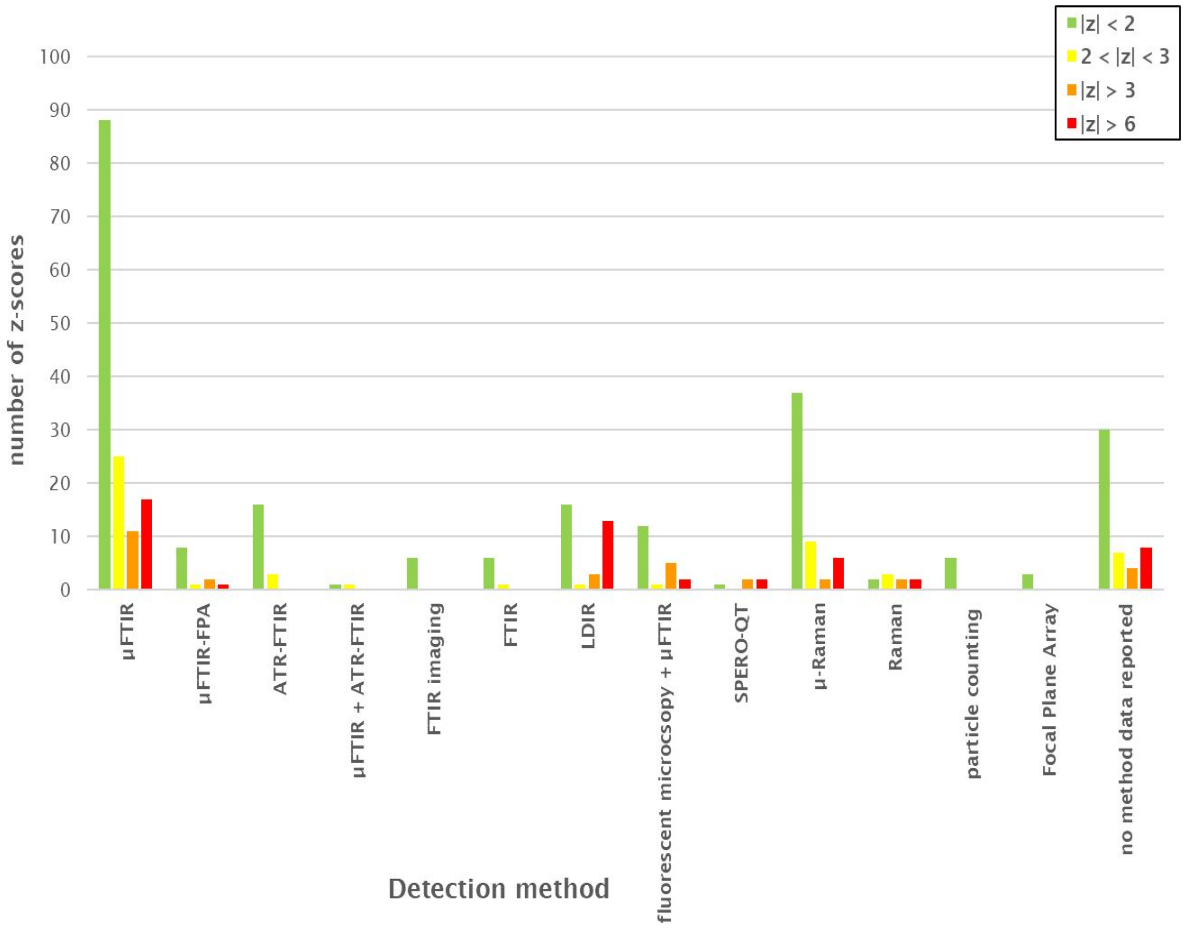
Detection (number)





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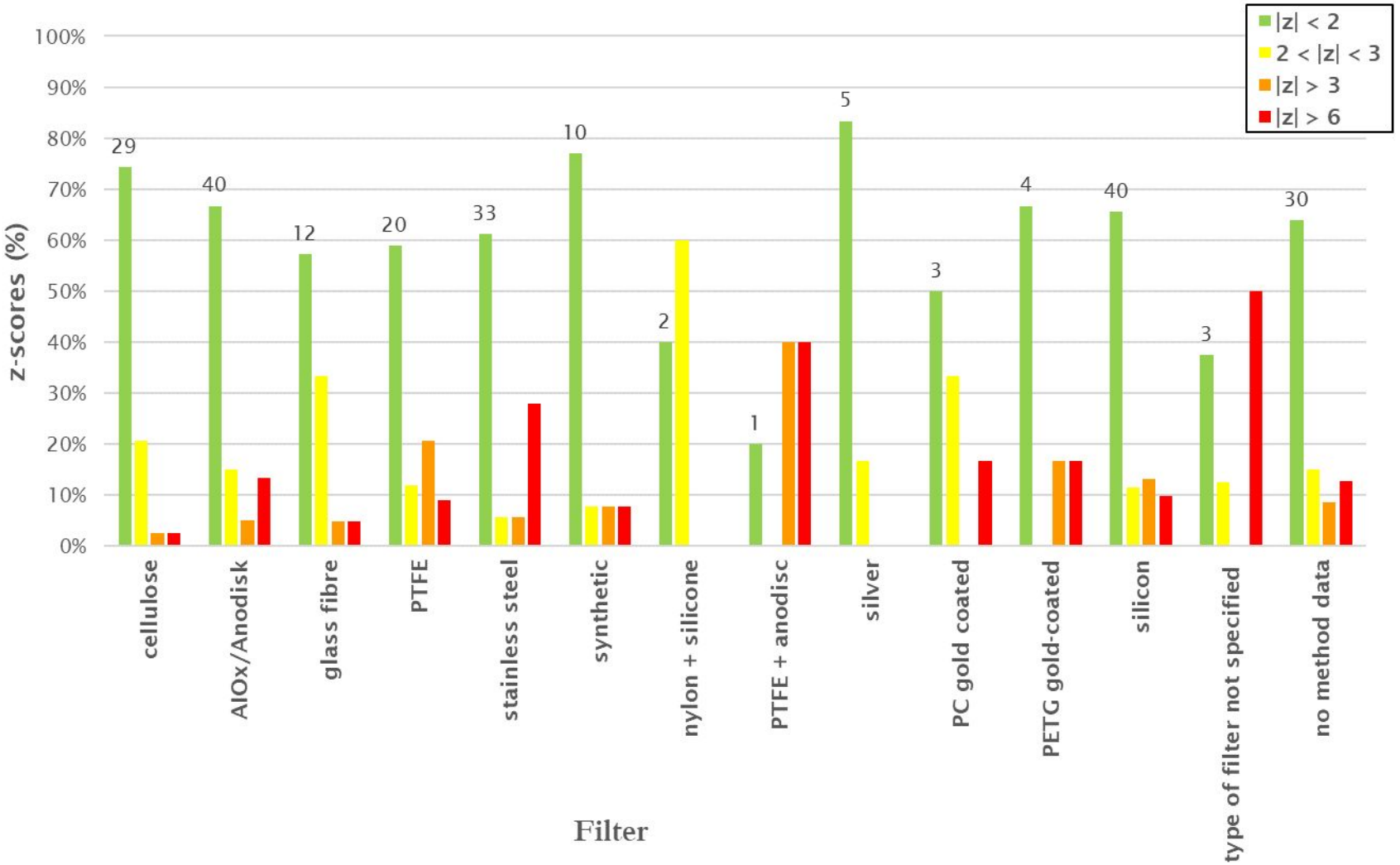
Filter (number)



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Harmonization of methods

Filter:

- Glass fibre most frequently used
  - used by 14 participants (=19%)
  - 57% of the z-scores were satisfactory (number determination)

- Cellulose:
  - used by 6 participants (=8%)
  - 74% of the z-scores were satisfactory (number determination)

≥ 50 % of satisfactory z-scores for all filter types except for PTFE + anodisc (20%) and nylon + silicone (40%)

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Density separation:

ZnCl2 and NaCl most frequently used

- ZnCl2 used by 14 participants (= 29%)
  - 42% of the z-scores were satisfactory (number determination)
  - 44% |z| > 3

- NaCl used by 12 participants (= 24%)
  - 75% of the z-scores were satisfactory (number determination)
  - all |z| < 3

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Harmonization of methods

Digestion:

H_2O_2 and Fenton most frequently used

- H_2O_2 used by 11 participants (= 28%)
  - 53% of the z-scores were satisfactory (number determination)
  - 38% |z| > 3

- Fenton used by 8 participants (= 20%)
  - 75% of the z-scores were satisfactory (number determination)
  - 8% |z| > 3

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Harmonization of methods

Detection:

µFTIR most frequently used

- used by 21 participants (=28%)
- 62% of the z-scores were satisfactory (number determination)

µRAMAN

- used by 7 participants
- 69% of the z-scores were satisfactory (number determination)

Pyr-GC/MS

- used by 9 participants

Large variation in detection methods.

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Conclusions

- Interest in MP analyses increases (ILS 1: n=30, ILS 2: n= 35, this round n=67)

- Majority report on number, only a small number report on mass

- Mass determination: For most polymers too few data to calculate an assigned value

- NDA Rel st. dev. : 76-96 % for tablets (number)
  66-125 % for sediment sample (number)
  > 100 % / No assigned value for sand sample (number)

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Conclusions

- Diversity in filter types, salts, digestion and detection methods

- Filter type: Almost all types ≥ 50 % of satisfactory z-score
  - Glass fibre, Cellulose, Stainless steel and silicon most frequently used

- Density : ZnCl, NaCl and NaI most used

- Digestion: Fenton preferred (based on frequency of use, and % satisfactory z-scores)

- Detection: µFTIR and µRAMAN most used, all > 60% satisfactory z-scores.
  pyr-GC/MS used by 9 participants no assigned value calculated

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Conclusions

Microplastic analyses is still challenging

More work to be done….

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Category 1: n ≥ 7

AV based on model mean when:

- ≥ 50% of values have a z'-score of $|z'| < 2$
- at least 5 values have a $|z'| < 3$.

Category 2: n > 3 and < 7

AV based on model mean when:

- ≥ 70% of values have a z'-score of $|z'| < 3$
- ≥ 4 observations have $|z'| < 2$.

Category 3: n < 4

No assigned value is given