Microplastics extraction from a sandy beach: methodology development and challenges

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Worldwide plastic production

China is the largest producer of plastics

World plastic production almost reached 350 M t

Source: PlasticEurope, 2018
Microplastics

- Small plastic pieces smaller than 5 mm in size
- **Majority of items** in the contaminated aquatic ecosystems
- Spherical beads, films, irregular fragments, filaments, foam, granules and fibres
- Poses a threat to aquatic life:
  - sorbent of toxic pollutants like HM or PCBs
  - persistent
  - bioaccumulation

Entering the **food chain** by ingestion by marine species
Microplastics

• The Marine Strategy Framework Directive was amended to highlight that the “composition of micro-particles (in particular microplastics) has to be characterized in marine litter and the marine coastal environment”.

• There are still no harmonised analytical methods for quantifying and determining the occurrence and composition of microplastics in those environments.
Objectives

1 - Development of an extraction technique for separating microplastics (PP, LDPE, PS, PET and PVC) from a sandy beach by using routine laboratory equipment
   - using the principle of flotation and decantation process to promote the separation

2 - Assessment of the recovery efficiency of the proposed method for:
   i) each microplastic material
   ii) microplastics fractions by size
Preparation of microplastics samples

Cutting (≈3 cm size) → Grounding in a mill → Sieving (2, 1, 0.5, 0.2 and 0.05 mm)

PP (polypropylene), LDPE (low-density polyethylene), PS (polystyrene), PET (polyethylene terephthalate), PVC (polyvinyl chloride)
## Preparation of microplastics samples

<table>
<thead>
<tr>
<th>Size</th>
<th>Plastic material used in spiked samples (g)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>PP</td>
</tr>
<tr>
<td>&gt; 2 mm</td>
<td>2.5</td>
</tr>
<tr>
<td>2 – 1 mm</td>
<td>2.5</td>
</tr>
<tr>
<td>1 – 0.5 mm</td>
<td>2.5</td>
</tr>
<tr>
<td>0.5 – 0.2 mm</td>
<td>1.5</td>
</tr>
<tr>
<td>0.2 – 0.05 mm</td>
<td>0.5</td>
</tr>
<tr>
<td>Total (g)</td>
<td>9.5</td>
</tr>
</tbody>
</table>

PP (polypropylene), LDPE (low-density polyethylene), PS (polystyrene), PET (polyethylene terephthalate), PVC (polyvinyl chloride)
Sampling of sandy beach

3 kg of sandy beach
Dried (105°C) and sieved
(2, 1, 0.5, 0.2 and 0.05 mm)

Washing of plastics out of the sand:

i) Stirring: sand + ZnCl₂
ii) Supernatant was discarded (2x)
iii) Rising with 0.001 M HCl

2-1 mm: ≈2%
1-0.5 mm: 63%
0.5-0.2 mm: 35%
0.2-0.05 mm: 35%

Figueira da Foz, Coimbra, Portugal
Extraction method

150 g of sand + microplastics + 250mL of ZnCl₂

Pumping of 600 mL of ZnCl₂, creating an overflow of the top layer

Sieving of supernatant (0.05 mm)

Microplastics in the 0.05mm sieve + Sand
Extraction method

Microplastics in the sieve

Rising
(0.001M HCl)

Drying
(at 60°C, 48h)

Sieving
(2, 1, 0.5, 0.2 and 0.05mm)

Weighing

Recovery efficiencies for the 1st flotation procedure
Extraction method

Sand + 250mL of ZnCl₂

Pumping of 600 mL of ZnCl₂, creating an overflow of the top layer

Sieving of supernatant (0.05 mm)

Microplastics in the 0.05mm sieve + Sand
Extraction method

Microplastics in the sieve

- Rising (0.001M HCl)
- Drying (at 60°C)
- Sieving (2, 1, 0.5, 0.2 and 0.05mm)
- Weighing

Recovery efficiencies for the 2nd flotation procedure
Extraction method

Sand

Washed
(0.001M HCl)

Drying
(at 105°C,
48h)

Stored
Recovery efficiencies of the proposed methodology - calculation

**Overall recovery rate (%)**

\[
\text{Overall recovery rate} = \frac{\text{plastic mass obtained at the end of extraction (g)}}{\text{plastic mass added at the beginning of extraction (g)}} \times 100
\]

**R**

**Recovery rate for microplastics fraction (%)**

\[
\text{Recovery rate for microplastics} = \frac{\text{plastic fraction mass obtained at the end of extraction (g)}}{\text{plastic fraction mass at the beginning of extraction (g)}} \times 100
\]
Overall recovery rates

- Range from 97 - 100%
- PS and PP registered a slightly lower rate than LDPE, PET, and PVC (2-3%)
- > 94% of microplastics were extracted in the 1st time flotation
- 2nd time flotation can be eliminated
Recovery rates for microplastics with different size

- Ranged from $51 \pm 8$ - $133 \pm 1\%$

Microplastics fraction 0.05-0.2mm displayed the lowest recovery rates

PVC fraction 0.5-1mm showed a very high recovery rate ($\approx 133\%$)
Challenges to overcome

• Fibres contamination from the working space

Performing of blanks to quantify the impact of it on the overestimation of recovery rates

• Presence of sand grains and unknown fragments in the extracted microplastics

Cleaning procedure of extracted microplastics:
• Stirring of the mixture of extracted microplastics with ZnCl₂

• Manual sieving of microplastics can cause an over or underestimation of microplastics mass

Automatic sieved to guaranty the same sample size
Conclusions

• The **good recoveries rates** obtained demonstrate the **potential** of the proposed extraction methodology.

• There are a **few problems** that need to be **addressed in further works**.

• This analytical **extraction method** can contribute to **boosting advancements for determining the occurrence of microplastics** in marine sediments.

Further works

• **Application** of the proposed methodology to **beach samples** from Angola.

• Using of **Fourier Transformed Infrared spectroscopy (FT-IR)** for **identification** of microplastics material.
Thank you for your attention.

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V. Oliveira gratefully acknowledges **FCT** – Fundação para a Ciência e a Tecnologia (SFRH/BD/115312/2016) and **CERNAS** for financial support (UID/AMB/01).