Supplementary information

Microplastic extraction from sediments established? – A critical evaluation from a trace recovery experiment with a custom-made density separator

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Supplement content: 6 pages (cover page included), 8 tables, 3 figure

Figure S1 Modified glass beaker (a) and displacement cone (b) with respective dimensions.

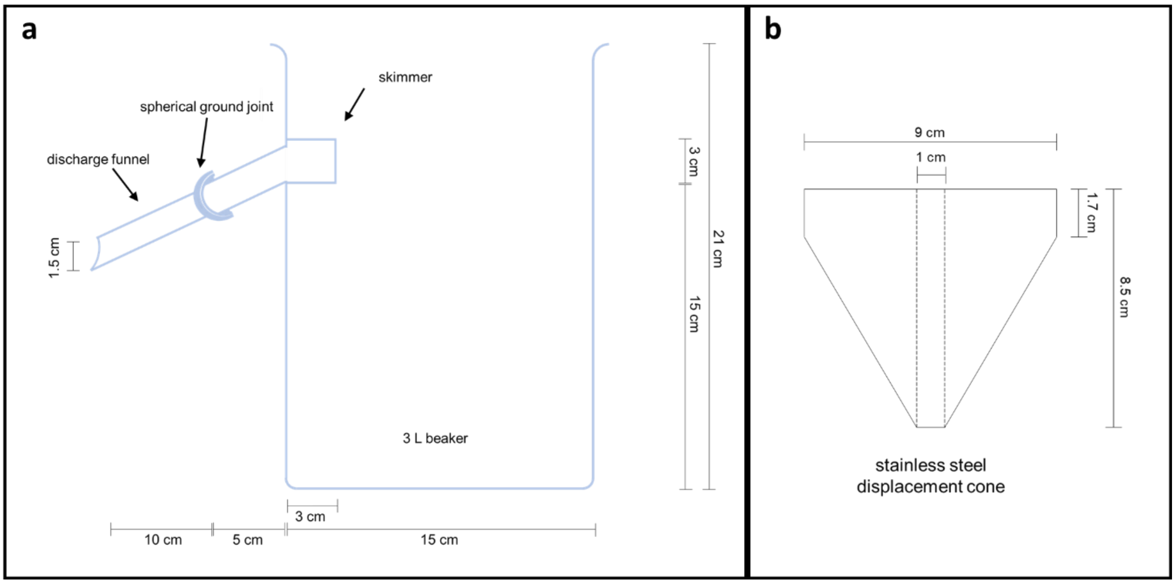


Table S1 Grain size distribution of quartz sand

	< 2 µm	**2 - 6,3 µm**	**6,3 - 20 µm**	**20 - 63 µm**	**63 - 200 µm**	**200 - 630 µm**	**630 - 2000 µm**
Fine quarz sand	0,0	1,0	1,6	1,2	87,5	8,7	0,0

Table S2 Spiked polymer masses in µg.

Polymer	QS1	**QS2**	**QS3**	**QS4**	**QS-A1**	**QS-A2**	**QS-A3**	**QS-A4**
PE	48.1	48.1	43.1	40.7	40.4	40.7	41.5	40.4
PP	46.7	43.6	44.8	42.5		43.7	40.1	40.3
PUR	42.4	40.2	47.5	48.5	47.8	45.8	41.5	44.7
PA6	49.3	42.1	48.2	40.8	45.3	42.0	45.0	48.9
PS	18.9	20.8	19.9	21.2	20	20.1	20.3	21
PMMA	21.6	20.6	20.6	20.0	22.2	19.3	20.3	19.8
PC	4.9	4.9	4.9	5.7	4.8	4.9	4.9	5.2
PVC	43.9	44.6	43.1	40.7	46.3	42.4	40.6	40.4
PET	14.1	14.7	14.4	15.8	15.5	15.3	14.9	15.6

Table S3 Spiked number of polymer particles.

Polymer	**QS1**	**QS2**	**QS3**	**QS4**	**QS-A1**	**QS-A2**	**QS-A3**	**QS-A4**
PE	8	7	7	15	7	8	7	15
PP	8	7	9	8	8	7	8	8
PUR	6	9	10	8	10	7	7	9
PA6	7	7	8	8	6	9	10	7
PS	6	6	6	10	5	9	9	8
PMMA	5	6	5	6	5	7	5	8
PC	6	6	9	5	7	7	5	5
PVC	18	18	18	20	15	20	20	20
PET	6	6	8	8	9	8	8	5

Figure S2 Schematic density separation process.

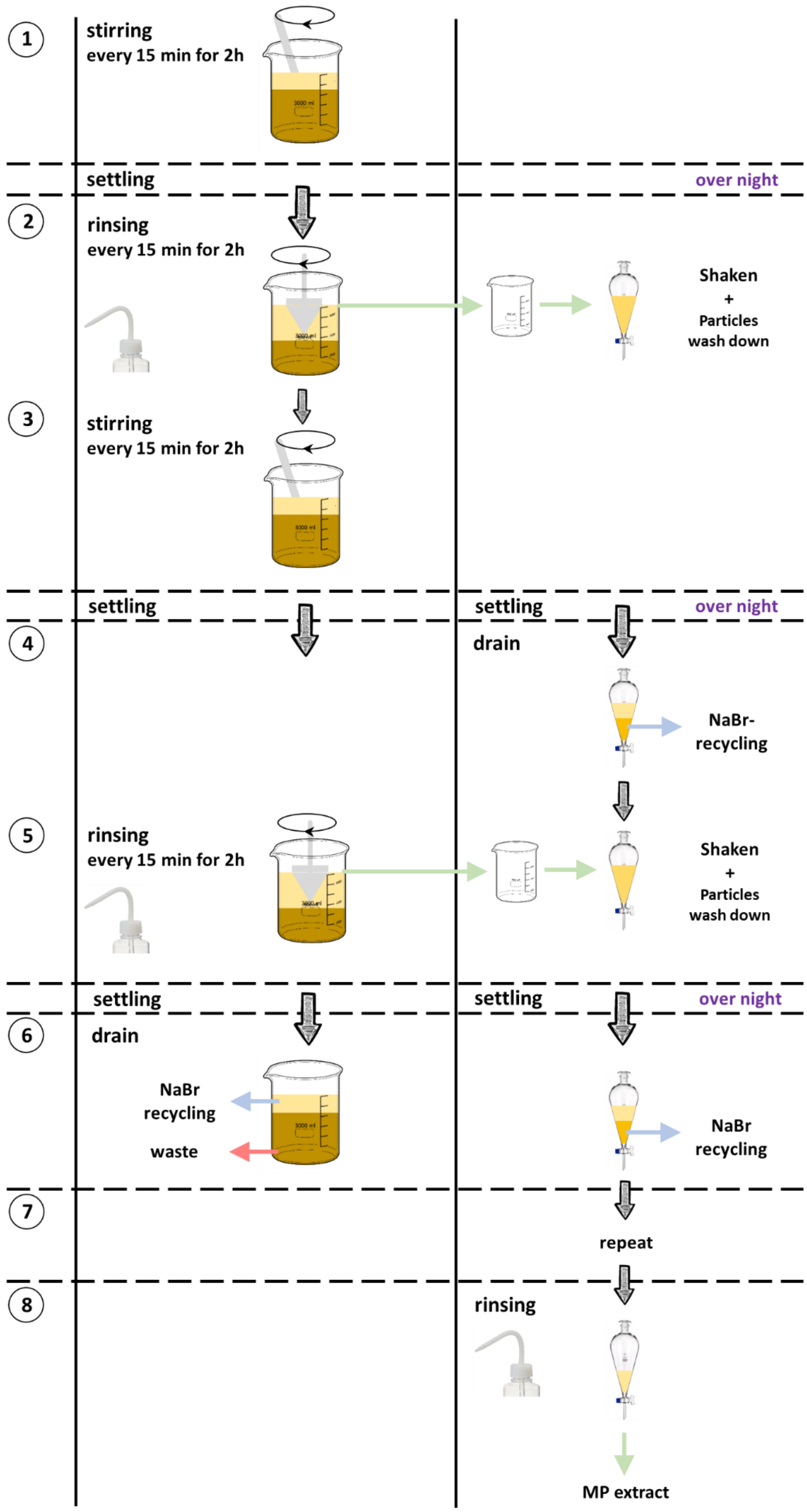


Table S4 Polymer specifications.

abbreviation	polymer	company	product name
PP	Polypropylene	Borealis AG, Wien, Austria	HL508FB
PE	Polyethylene	Borealis AG, Austria	MG7547S
PS	Polystyrene	Total Refining & Chemical Polymers, Total Research & Technology Feluy, Belgium	Impact 7240
PA6	Polyamide-6	Ter Hell GmbH, Germany	Akulon® K222-D
PMMA	Polymethyl-methacrylate	Evonik Performance Materials GmbH, Germany	Plexiglas® 7N
PC	Polycarbonate	Teijin Kasei America, US	Panlite® L-1250Y
MDI-PUR	MDI-Polyurethane	GEBA GmbH	Desmovit® DP LFC 3379
PET	Polyethylene-terephthalate	Ter Hell GmbH, Germany	K896
PVC	Polyvinylchloride	Granulat GmbH, Germany	Troilit® VB 537-HE

Table S5 Injection Standards.

$ISTD_{PY}$	Injection (µg)
9-tetradecyl-1,2,3,4,5,6,7,8-octahydro anthracene	0.5
cholanic acid	0.5
anthracene (d_{10})	1.0
polystyrene (d_8)	1.0

Table S6 Conditions for Pyrolysis-GCMS/Thermochemolysis measurements.

Micro furnace pyrolyzer	**EGA/PY-3030D (FrontierLabs)**
carrier gas	Helium
curie temperature	590°C
pyrolysis time	1 min
transfer line temperature	320°C
Gas chromatograph	**7890B (Agilent)**
injector	split/split less
mode	split 15:1
temperature	300°C
pre-column	Trajan P/N 064062; 10 m x 250 µm/ 363 µm VSPD Tubing
column	DB5 (J&W); 30 m x 0.25 mm ID, film thickness 0.25 µm
flow (const.)	1.2 ml/min
temperature program	35°C (2 min) → 310 °C (30 min) at 3°C/min
transfer line temperature	280°C
Mass spectrometer	**MSD 5977A (Agilent)**
ionization energy	70 eV
scan rate	2.48 scans/s
scan range	50-650 amu
EI-Source temperature	230 °C
quadrupole temperature	150 °C

Table S7 Overview of different measurements sequences and accompanied calibrations.

	PE	PP	PET	PS	PVC	PC	PMMA	PA6	PUR
QS-A									
$ISTD_{py}$	d-PS	TOHA	none	none	none	TOHA	d-PS	TOHA	TOHA
b	7.88E-05	1.25E-02	-4.06E+06	3.02E+05	3.25E+06	-1.22E+00	2.50E-01	2.51E-01	2.66E-02
slope	8.15E-03	2.58E-02	2.77E+06	1.33E+06	7.02E+05	2.70E+00	3.19E-01	1.70E-01	6.85E-03
r^2	0.93	0.99	0.97	0.98	0.9	0.95	0.95	0.96	0.98
s_{x0} [µg]	5.2	1.46	0.94	1	5	0.4	1.7	3.7	2.8
n	10	8	6	7	12	6	15	8	5
QS									
$ISTD_{py}$	TOHA	Anthracene-d10	Anthracene-d10	Anthracene-d10	none	Anthracene-d10	Anthracene-d10	d-PS	Anthracene-d10
b	-4.32E-03	3.22E-02	5.40E-02	1.54E-04	-8.45E+05	3.17E-01	-7.10E-01	2.51E-01	-6.27E-03
slope	7.41E-03	1.92E-02	1.82E-01	7.39E-02	1.08E+06	2.49E-01	2.58E-01	3.86E-01	7.54E-03
r^2	0.96	0.98	0.8	0.97	0.99	0.99	0.91	0.92	0.99
s_{x0} [µg]	2.2	2.3	3.1	1.4	1.9	0.2	2.1	3.4	1.6
n	5	5	6	5	5	4	6	8	4

r^2 = coefficient of determination. s_{x0} = process standard deviation. TOHA = 9-tetradecyl-1.2.3.4.5.6.7.8-octahydro anthracene

Table S8 List of polymers and their respective specific indicator ions.

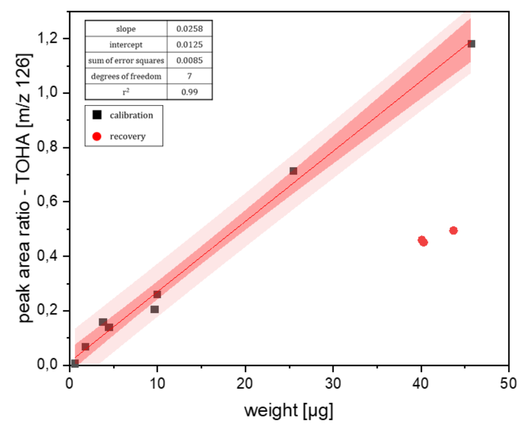
Polymer	Abbre-viation	Characteristic decomposition product(s)	RI [a]	M (m/z)	Indicator ions (m/z)
Polyethylene	PE	Alkanes (e.g. C_{20})	2000	282	85
		α-Alkenes (e.g. C_{20})	1994	280	83
		α,ω-Alkenes (e.g. C_{20}**)**	1987	278	**82**
Polypropylene	PP	**2,4-Dimethylhept-1-ene**	832	126	126, **70**
		2,4,6,8-Tetramethyl-1-undecenes[b]	1306	210	100, 69
		2,4,6,8-Tetramethyl-1-undecenes[c]	1315	210	100, 69
		2,4,6,8-Tetramethyl-1-undecenes[d]	1323	210	100, 69
Polystyrene	PS	Styrene	890	104	104
		2,4-Diphenyl-1-butene	1720	208	91
		2,4,6-Triphenyl-1-hexene	2440	312	**91**
Polyvinyl chloride	PVC	**Benzene**	738	78	**78**
		Naphthalene	1187	128	128,102,64
Poly(methyl methacrylate)	PMMA	Methylacrylate	726	86	55
		Methyl methacrylate	775	100	**100**, 69
Polyamide	PA6	**ε-Caprolactam**	1257	113	**113**
		N-methyl caprolactam[e]	1224	127	**127**
Polyethylene terephthalate	PET	**Dimethyl terephthalate**[e]	1504	194	**163**
Polycarbonate	PC	p-Methoxy-tert-butylbenzene[e]	1240	242	164, 149
		2,2-Bis(4'-methoxy-phenyl) propane[e]	2065	256	256**, 241**
MDI-Polyurethane	MDI-PUR	4,4'-Methylenbis(N-methylaniline)[e]	2330	226	226
		N,N-Dimethyl-4-(4-methylamino)benzylanilin[e]	2341	240	240
		4,4'-Methylenbis(N,N-dimethylaniline)[e]	2354	254	253, **254**

[a]RI = Retention index calculated after Van Den Dool 1963, DB-5 column; M = molecular ion, m/z = mass to charge ratio; [b]Isotactic. [c]Heterotactic. [d]Syndiotactic. [e]Only after TMAH teatment; bold: indicator ions used for calibration

Table S9 Comparable information on recovery replicates and blank.

	sample type	unit	PE	PP	PET	PS	PVC	PC	PMMA	PA6	PUR
QS1	recovery	area	4,423,989	69,749,536	146,911,204	44,489,460	57,876,856	79,473,631	52,603,129	216,781,652	8,299,622
QS2	recovery	area	895,447	43,990,738	179,307,147	41,640,345	27,443,610	86,370,510	76,219,166	99,725,655	5,295,626
QS3	recovery	area	2,365,030	57,724,282	77,197,126	45,917,973	134,302,177	76,988,927	25,326,922	98,979,717	5,444,208
QS4	recovery	area	1,814,458	59,692,763	91,303,143	71,910,368	40,685,610	67,179,622	47,915,105	110,531,649	7,760,737
QS-A1	recovery	area	157,769		16,848,259	7,017,346	43,320,480	21,063,230	16,630,824	11,932,827	820,954
QS-A2	recovery	area	98,896	12,016,237	75,259,770	11,205,206	42,564,412	37,494,823	29,214,028	14,11,615	1,022,456
QS-A3	recovery	area	192,270	10,152,031	52,272,313	14,382,739	24,386,681	31,431,327	17,894,426	15,795,688	862,991
QS-A4	recovery	area	179,347	11,104,362	49,782,414	14,594,378	35,814,383	44,873,513	17,881,931	21,637,572	1,175,387
QS-AB1	blank	area	n.d.	n.d.	1,188,910	184,206	3,090,954	25,619	1,267,267	476,340	n.d.
QS-AB1 quantified	blank	µg	n.d.	n.d.	0.58	-0.09	-0.23	0.46	4.42	-0.10	n.d.

Figure S3 Formal and calculation example (PP) for the process standard deviation and its projection on the polymer recovery.



formula:

$$\frac{\sqrt{\frac{degrees\ of\ freedom}{sum\ of\ error\ squares}}}{slope} = process\ standard\ deviation$$

$$\frac{process\ standard\ deviation}{mass_{spiked}} * 100 = \begin{matrix} process\ standard\ deviation \\ in\ percent\ recovery \end{matrix}$$

example:

$$\frac{\sqrt{\frac{7}{0.0085}}}{0.0258} = 1.46$$

$$\frac{1.46\ \mu g}{46.7\ \mu g} * 100 = 3.12\%$$