Electronic supplementary information (ESI)

Using castor oil to separate microplastics from four different environmental matrices

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Content Summary: fourteen pages, seven tables, thirteen figures

MATERIALS AND INSTRUMENTS

Table S1 Details of materials and instruments used in the protocol, as mentioned in section *2. Materials and Methods* in their respective order of occurrence.

Material / Instrument	**Details**
Neuston net mesh	0.3 mm mesh cods, attached to a metal and bamboo construction.
Geological sieves	1.0, 0.5, 0.3 mm; Retsch, Haan, Germany
Incubator for drying samples at 40 °C	BINDER BD 115, Tuttlingen, Germany
Scales for weighing samples to nearest mg	Mettler Toledo XS 105 DualRange, Columbo OH, USA
Heated laboratory stirrer	Schott laboratory stirrer SLR, Mainz, Germany
Grinder for polymer fragmentation	Merlin 123 household grinder, WS-Teleshop, Neudorf, Austria
Stereomicroscope	Olympus SZ61, Tokyo, Japan
Fourier-Transform Infrared Spectrometer (FT-IR)	Bruker ALPHA, platinum Diamond-ATR QuickSnap Sampling Module, Bruker, Billerica, MA, USA
Software Opus 7.5	Bruker, Billerica, Massachusetts, USA; B-KIMW ATR-IR Polymers, Plastics and Additives, 898 entries, library is available upon request
Castor oil	Cold pressed, organic, lot-no.: 1703A0138, Armonia GmbH, Azmoos, Switzerland https://www.armonia-shop.ch/online-shop/basis%C3%B6le-1/
Separation funnel	Lenz, Sqibb-F., grad., 2000 mL, PTFE-cock NS 18,8; ground joint NS 29/32
Filter paper	Hahnemühle DP 1505 110, pore size: 25 µm, diameter: 11 cm, Dassel, Germany
Labware detergent	Mucasol universal detergent, Schülke, Norderstedt, Germany
Hydrogen peroxide, H_2O_2 (30%)	Sigma-Aldrich, St. Louis, MO, USA

19 ENVIRONMENTAL MATRICES SEPARATED WITH THE OIL SEPARATION

20 PROTOCOL

21 **Table S2** Basic Characteristics of the Four Environmental Matrices

Name	**Sample description**	**sampling location**	**date**	**figure**
MBS Marine Beach Sediment	Clastic sediment, consisting mainly of quartz; low organic content (< 1%)	36°00'42.8"N, 5°36'31.3"W, Atlantic, Tarifa, Andalusia, Spain	09/2017	S1 (A)
AS Agricultural Soil	Agricultural soil	47°31'57.6"N, 7°36'53.5"E, Münchenstein, canton of Basel Country, Switzerland	10/2017	S1 (B)
MSS Marine Suspended Surface Solids	Suspended surface solids; mainly algae and fine sand	36°00'40.9"N, 5°35'41.7"W, Mediterranean, Tarifa, Andalusia, Spain	09/2017	S1 (C)
FSS Fluvial Suspended Surface Solids	Suspended surface solids; 90% organic biogenic content	47°32'26.3"N, 7°36'59.3"E, Saint-Albanteich, Basel, canton of Basel-City, Switzerland	01/2018	S1 (D)
FSS (n = 5) for calibration of MOSeS with environmental microplastics	Suspended surface solids; 90% organic biogenic content	1. 47°30'16.8"N 8°14'14.0"E, 86.3 m^3, Limmat River, Untersiggenthal, canton of Aargau, Switzerland	08/2016	No fotos

		2. 47°33'19.6"N, 7°35'54.8"E, 94.4 m^3, Rhine River, Basel, canton of Basel-City, Switzerland 3. 47°33'25.9"N, 7°36'27.8"E, 85.6 m^3, Rhine River, Basel, canton of Basel-City, Switzerland 4. 50°37'47.0"N, 7°12'47.1"E, 70.3 m^3, Rhine River, Bad Honnef, North Rhine-Westphalia, Germany 5. 51°45'22.6"N, 6°24'06.7"E, 84.7 m^3, Rhine River, Rees, North Rhine-Westphalia, Germany	09/2016 09/2016 09/2016 09/2016	

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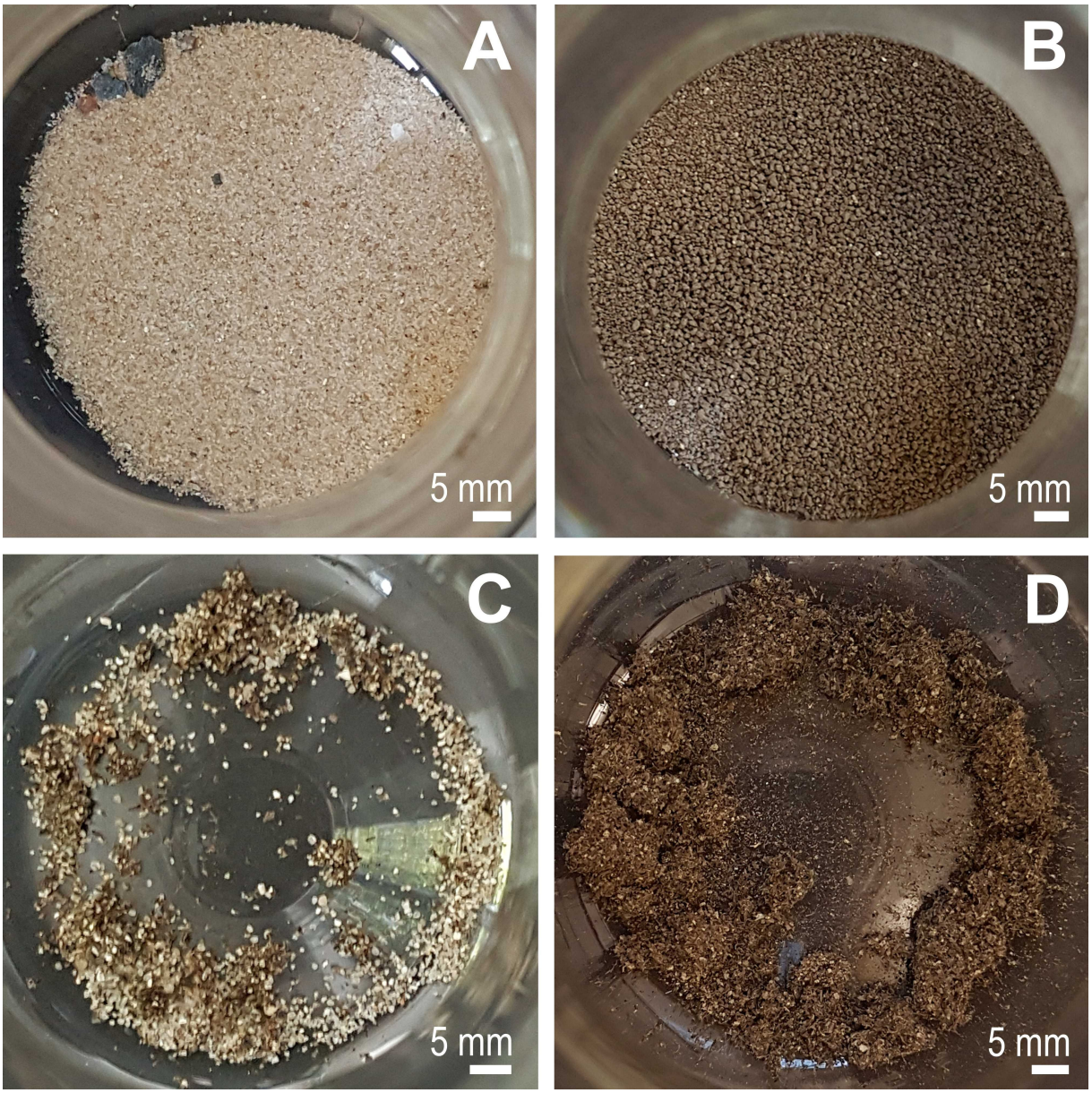

Fig S1 Environmental matrix samples before performing the oil separation protocol. (A) Marine beach sediment (MBS), (B) agricultural soil (AS, 10 g each) and (C) marine suspended surface solids (MSS), (D) fluvial suspended surface solids (FSS, 1 g each).

SCHEMATIC DIAGRAM FOR THE OIL SEPARATION PROTOCOL

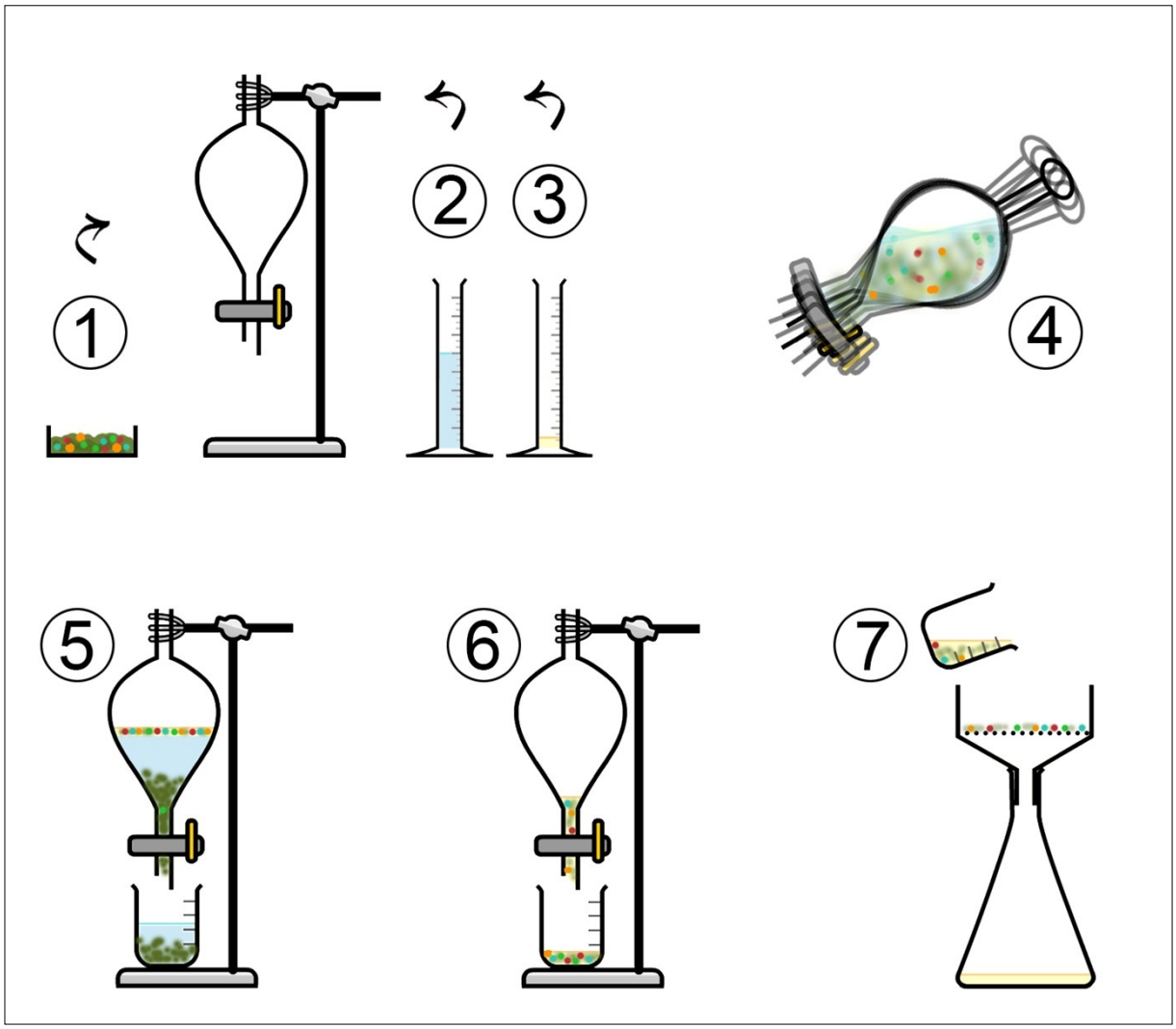

Fig S2 Castor oil separation protocol depicted in seven steps: add environmental sample (1) and 100 mL of aq. dest. water (2) to the separation funnel and make sure to wet the entire sample. Next, add 10 mL of Castor oil (3) to the separation funnel. Seal and shake the separation funnel for 1 min (4). Let the mixture separate and release lower, aqueous and solid phase (5). Release oil phase to different container (6) and filter oil phase for the recovery of microplastics (7). Diagram was constructed using the CHEMIX School software v2.8.1 (Arne Standnes, Bergen, Norway).

PLASTIC SPIKE RECOVERY AND ENVIRONMENTAL MATRIX REDUCTION

Table S3 Petrochemical Polymer Spike Recovery Rates and Environmental Matrix Reduction Rates for All Experiment Replicates (n = 4 for each Matrix)

Environmental matrix	replicate	Recovery rate for reference polymer particles								Environmental matrix dw reduction
		Small particles (0.3–0.5 mm)				Large particles (0.5–1 mm)				
		PP (n = 15)	PS (n = 15)	PMMA (n = 15)	PET-G (n = 15)	PP (n = 10)	PS (n = 10)	PMMA (n = 10)	PET-G (n = 10)	
MSS	1	100%	100%	100%	100%	100%	100%	100%	100%	94%
	2	100%	100%	100%	100%	100%	100%	100%	100%	95%
	3	100%	93%	100%	100%	100%	100%	100%	100%	93%
	4	100%	100%	100%	93%	100%	100%	100%	100%	93%
FSS	1	100%	100%	100%	100%	100%	100%	100%	100%	92%
	2	100%	100%	100%	93%	100%	100%	100%	100%	93%
	3	100%	93%	93%	87%	100%	100%	100%	100%	92%
	4	100%	100%	100%	93%	100%	100%	100%	100%	85%
MBS	1	100%	93%	87%	87%	100%	100%	100%	100%	96%
	2	100%	100%	100%	100%	100%	100%	100%	100%	98%
	3	100%	93%	100%	100%	90%	100%	100%	90%	96%
	4	93%	93%	93%	87%	100%	100%	100%	90%	96%
AS	1	100%	100%	100%	100%	100%	100%	100%	100%	97%
	2	100%	100%	100%	100%	100%	100%	100%	100%	99%
	3	100%	100%	100%	100%	100%	100%	100%	100%	98%
	4	87%	100%	87%	100%	100%	100%	100%	100%	98%

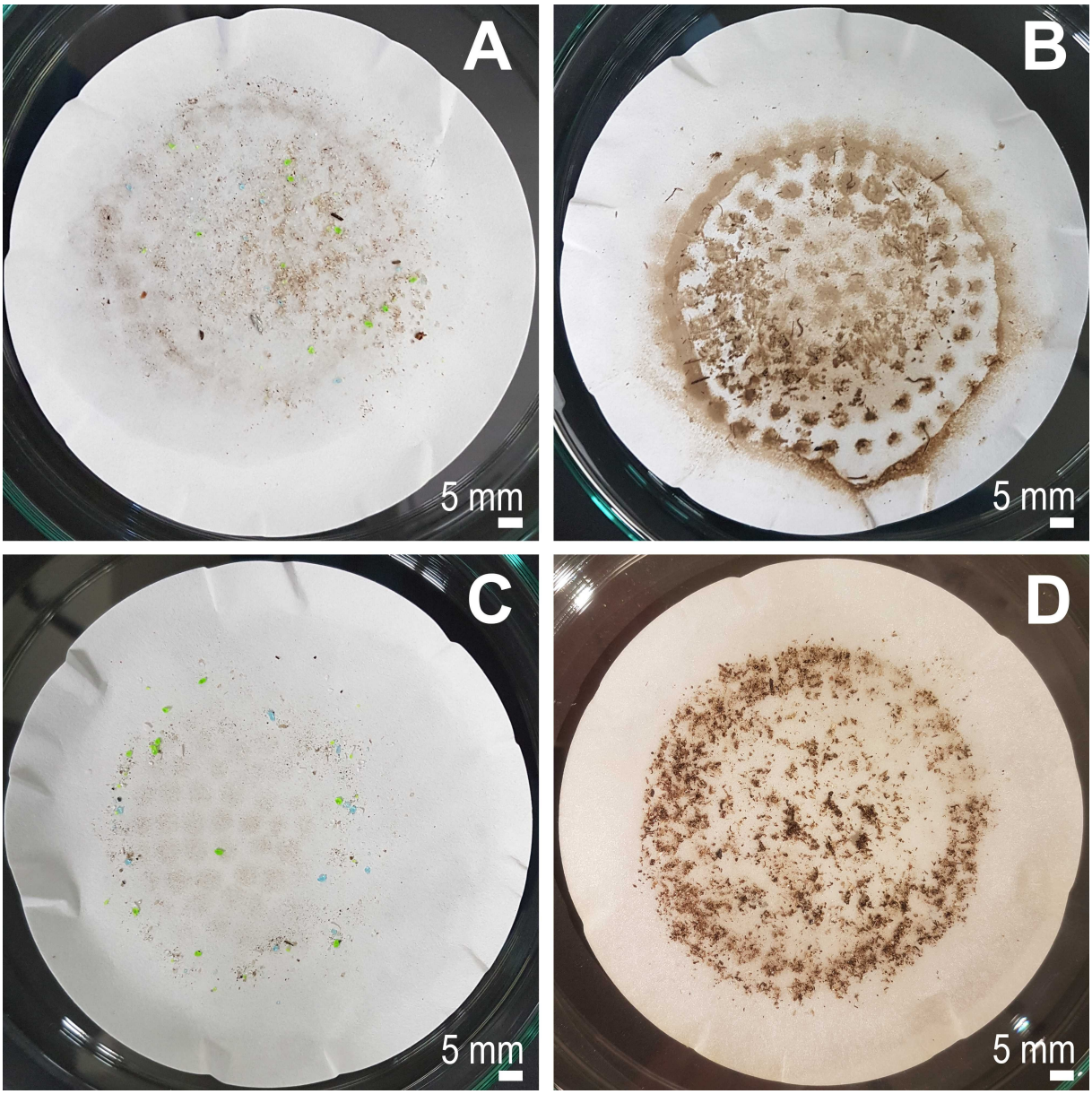

Fig S3 Environmental matrix residues and recovered spiked microplastic particles (blue: PS, green: PET-G; size range: 0.3–1 mm, visible in panels A and C) on cotton/cellulose filter paper (Hahnemuehle DP 1505 110, pore size: 25 µm, diameter: 11 cm, Dassel, Germany) after performing the MOSeS protocol. (A) Marine beach sediment (MBS), (B) agricultural soil (AS), (C) marine suspended surface solids (MSS) and (D) fluvial suspended surface solids (FSS).

MICROPLASTICS IN FIVE NON-SPIKED FSS SAMPLES

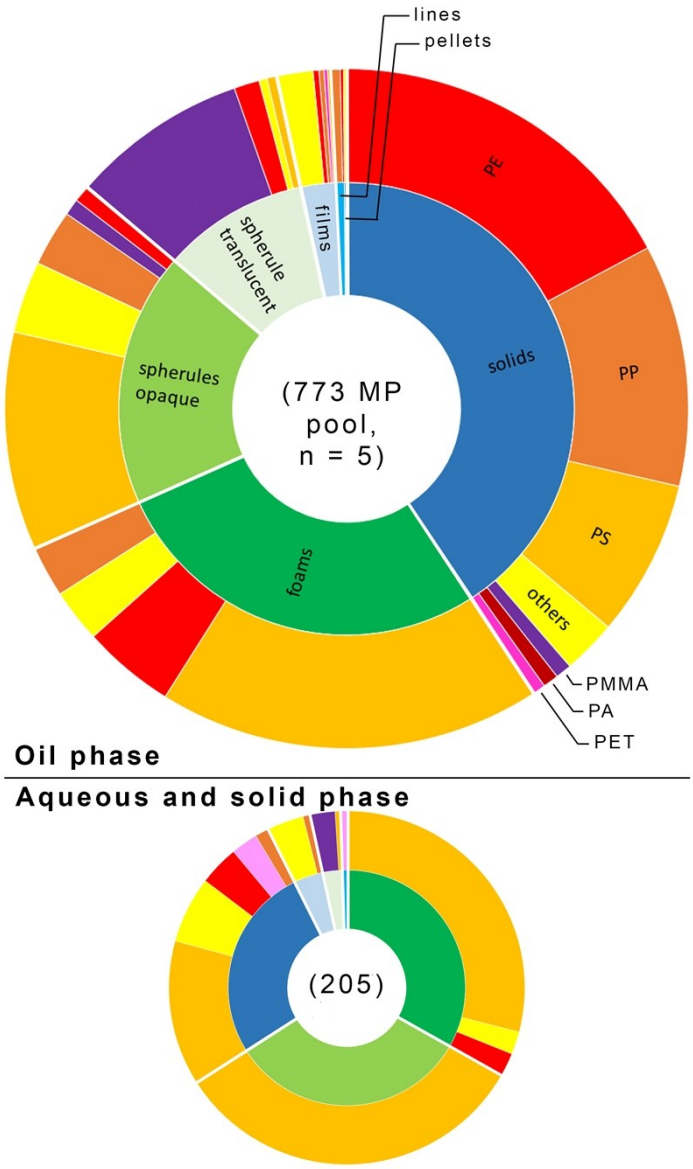

Fig S4 Relative proportions of microplastic (MP) categories (inner ring) and polymer types (outer ring) retrieved from the oil (above) and the aqueous and solid phases (below), respectively. The two diagrams together represent the pooled amount of MP from five Rhine River surface samples. After oil separation the five upper oil phases combined contained 773 (79.1%) and the lower aqueous and solid phases contained 205 (20.9%) of the total 978 (100%) MP particles from all five samples. The two inner rings, representing the relative shares of MP categories, are scaled in percental proportion to each other by surface area according to number of particles.

MICROPLASTIC PARTICLES FOR SPIKING

Table S4 Basic Characteristics of the Polypropylene (PP) used for Spiking

Polymer/Density	Product	Retailer	Fragmentation method (0.3–0.5; 0.5–1.0 mm)
Polypropylene (PP), 0.84 g cm^{-3}	Kitchen strainer RONDO 32.5 cm red https://www.rothoshop.de/Kueche/Sieb-RONDO-32-5-cm.html, accessed on 05.06.18	Rotho Kunststoff AG, Würenlingen, Switzerland	Coffee grinder (Merlin 123 household grinder, WS-Teleshop, Neudorf, Austria), geological sieves (1 mm, 0.5 mm and 0.3 mm, Retsch, Haan, Germany)

Fig S5 Large (left) and small (right) PP particles after recovery

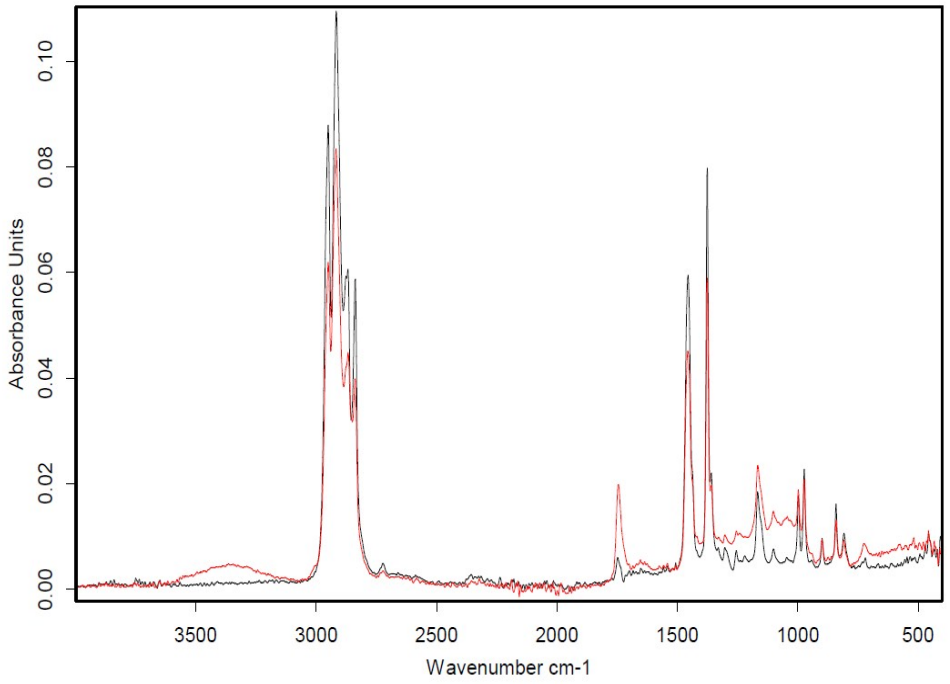

Fig S6 FT-IR Spectrum of PP before (black) and after (red) separation using the MoSeS protocol (Bruker ALPHA, Billerica, MA, USA)

62 **Table S5** Basic Characteristics of the Polystyrene (PS) used for Spiking

Polymer/Density	Product	Retailer	Fragmentation method (0.3–0.5; 0.5–1.0 mm)
Polystyrene (PS), 1.05 g cm^{-3}	Colouraplast melting pellets medium-blue, article-ID: 607199 https://www.aduis.ch/colouraplast-200-g,-mittelblau-art607199.aspx, accessed on 05.06.18	Creartec trend-design, Lindenberg, Germany	Coffee grinder (Merlin 123 household grinder, WS-Teleshop, Neudorf, Austria), geological sieves (1 mm, 0.5 mm and 0.3 mm, Retsch, Haan, Germany)

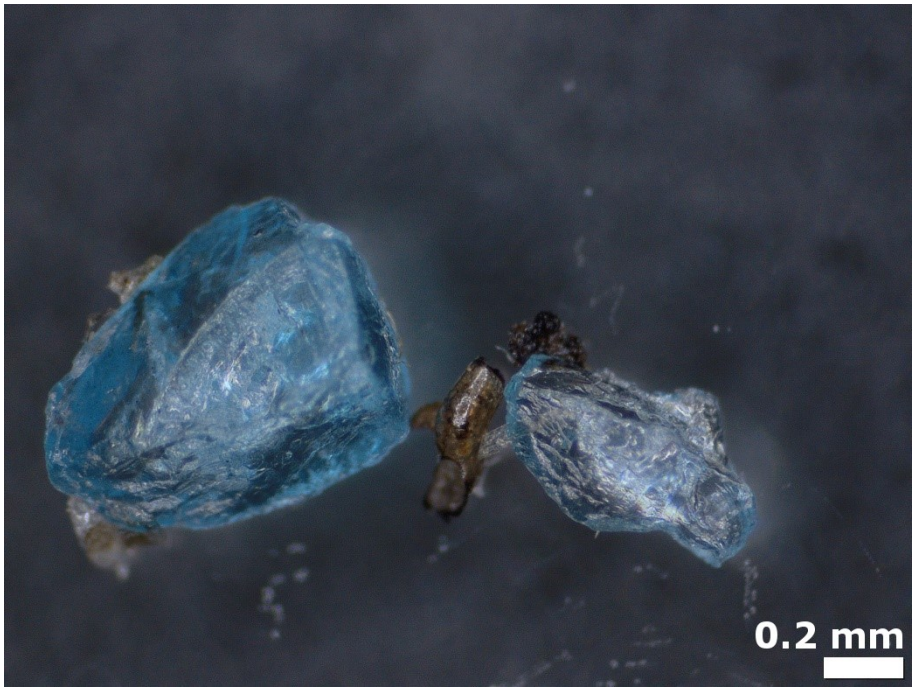

Fig S7 Large (left) and small (right) PS particles after recovery

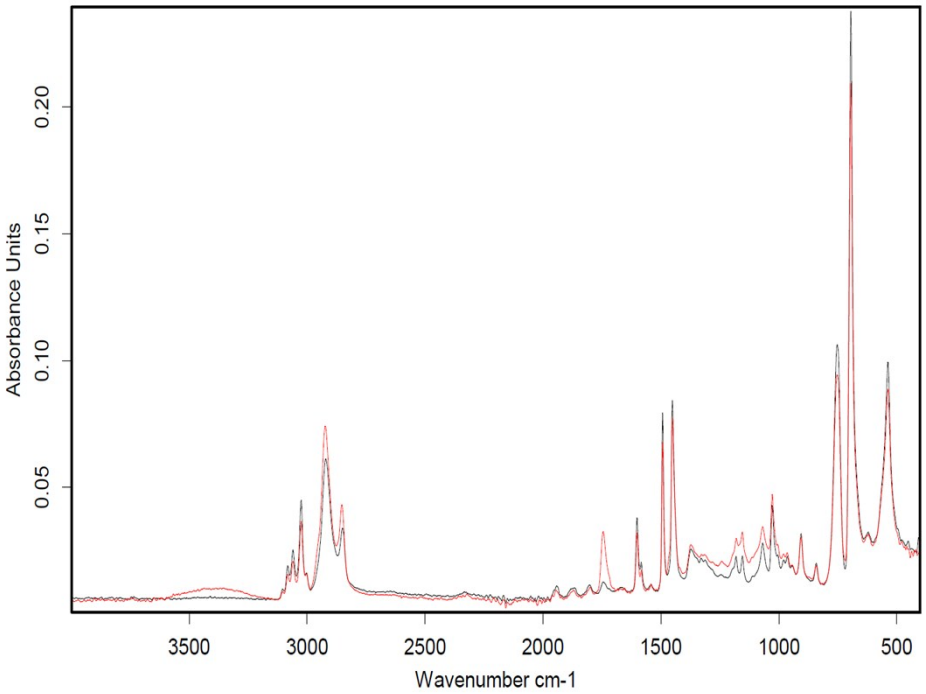

Fig S8 FT-IR Spectrum of PS before (black) and after (red) separation using the MoSeS protocol (Bruker ALPHA, Billerica, MA, USA)

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64 **Table S6** Basic Characteristics of the Polymethyl Methacrylate (PMMA) used for Spiking

Polymer/Density	Product	Retailer	Fragmentation method (0.3–0.5; 0.5–1.0 mm)
Polymethyl methacrylate (PMMA), 1.19 g cm^{-3}	Acrylic glass sheet (3 mm) Evonik, article-ID: 3000813 https://www.modulor.de/plexiglas-gs-farbig-3-mm-3-0-x-120-x-250-mm-orange-transparent-2c04.html, accessed on 05.06.18	Modulor Material Total, Berlin, Germany	coffee grinder (Merlin 123 household grinder, WS-Teleshop, Neudorf, Austria), geological sieves (1 mm, 0.5 mm and 0.3 mm, Retsch, Haan, Germany)

Fig S9 Large (right) and small (left) PMMA particles after recovery

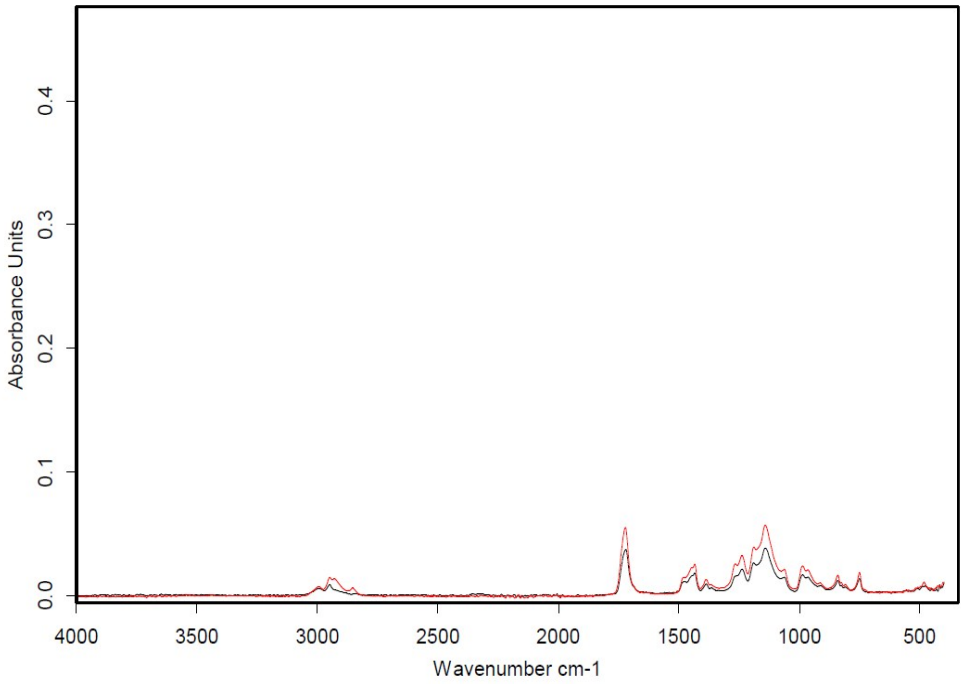

Fig S10 FT-IR Spectrum of PMMA before (black) and after (red) separation using the MoSeS protocol (Bruker ALPHA, Billerica, MA, USA)

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Table S7 Basic Characteristics of the Polyethylene Glycol-Modified (PET-G) used for Spiking

Polymer/Density	Product	Retailer	Fragmentation method (0.3–0.5; 0.5–1.0 mm)
Polyethylene terephthalate glycol-modified (PET-G), 1.27 g cm^{-3}	3D printer filament Minadax, article-ID: 16863 Https://www.amazon.de/Qualitaet-PET-Filament-transparent-3D-Drucker-hergestellt-gr%C3%bcn/dp/B01781ERXE, Accessed on 05.06.18	Minadax online retail services, Dortmund, Germany	Coffee grinder (Merlin 123 household grinder, WS-Teleshop, Neudorf, Austria), geological sieves (1 mm, 0.5 mm and 0.3 mm, Retsch, Haan, Germany)

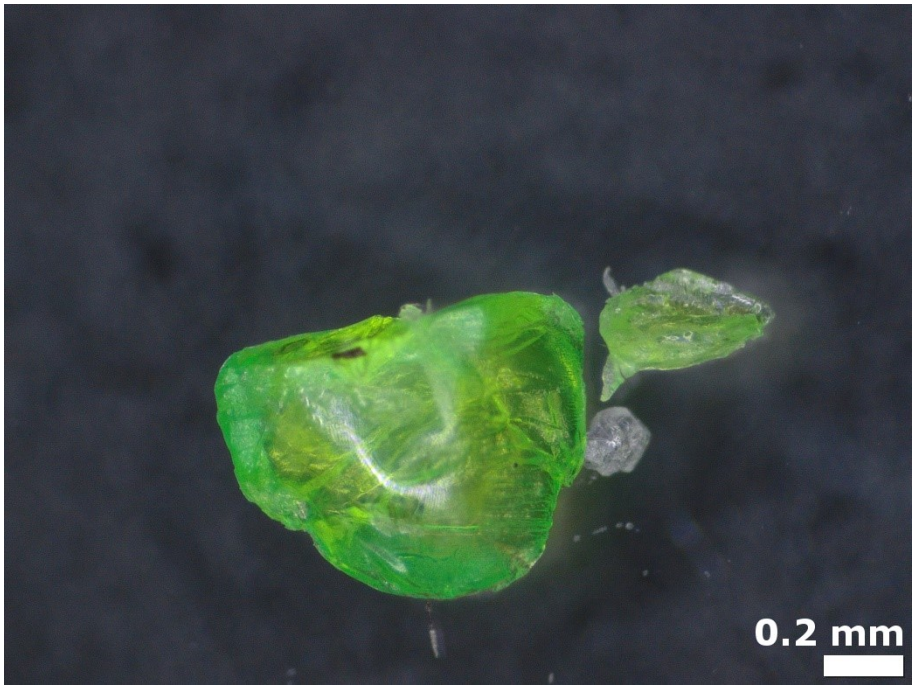

Fig S11 Large (left) and small (right) PET-G particles after recovery

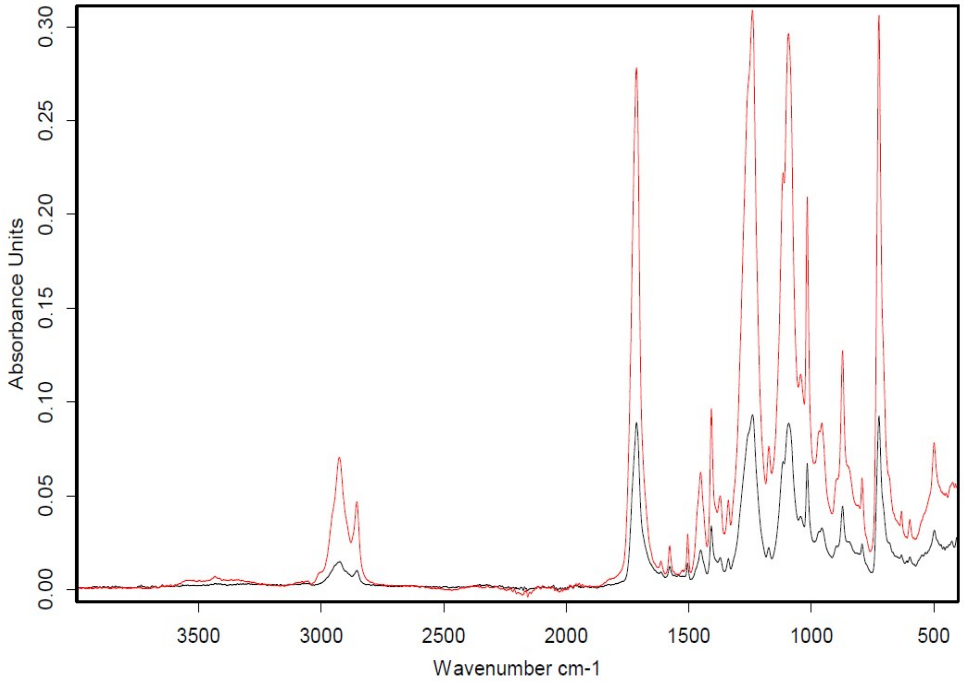

Fig S12 FT-IR Spectrum of PET-G before (black) and after (red) separation using the MoSeS protocol

EFFECT OF ETOH-RINSING ON ATR-FTIR HQI PERFORMANCE

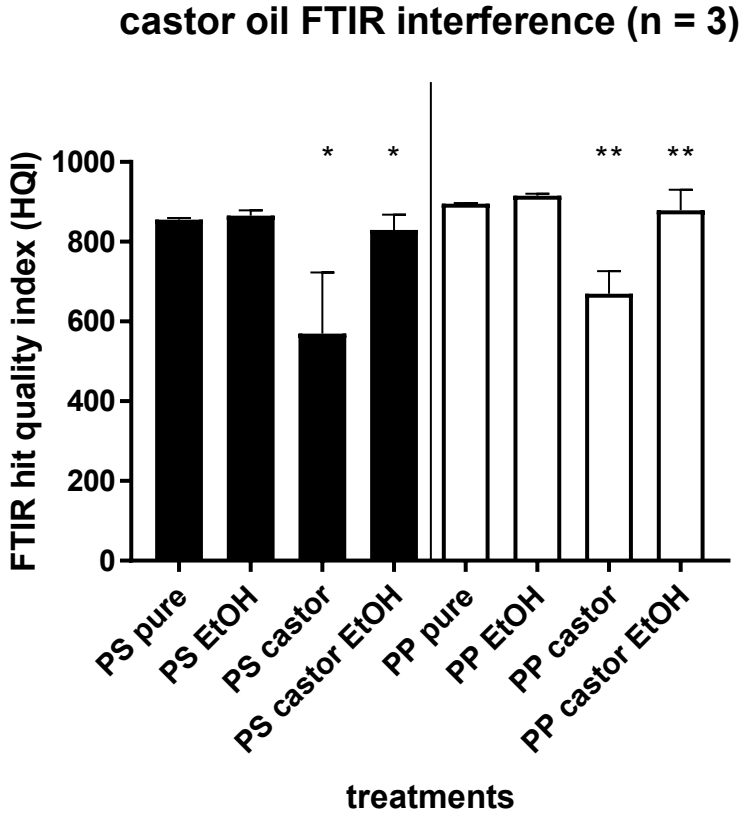

Fig S13 Comparison of ATR-FTIR hit quality indices for four treatments of PS and PP virgin microplastics (0.5–1.2 mm, longest axis) using unpaired t tests. Columns marked with asterisks indicate significant differences between the treatments of interest (castor oil removal using EtOH; * = $p<0.05$, ** = $p<0.01$). HQI>700 is regarded as a reliable level for confirming synthetic poylmers[1 2].

References

1 R. C. Thompson, Y. Olsen, R. P. Mitchell, A. Davis, S. J. Rowland, A. W. G. John, D. McGonigle and A. E. Russell, *Science (New York, N.Y.)*, 2004, **304**, 838.

2 R. W. Obbard, S. Sadri, Y. Q. Wong, A. A. Khitun, I. Baker and R. C. Thompson, *Earth's Future*, 2014, **2**, 315–320.