Supporting Information for

Microplastics in surficial sediments from UK rivers and canals: seasonal and spatial variation and relationships with concentrations of organophosphate esters

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Laboratory procedure for analysis of sediment samples for OPEs.

Samples were extracted in accordance with the method of (Brommer et al., 2012) with slight modifications. One (1) g of sediment was mixed in a clean dry test tube with 1 g of copper powder and spiked with 10 ng of internal (surrogate) standard mixture (d_{12}-TCEP, d_{12}-TDCPP, and d_{15}-TPhP). The samples were then extracted by vortexing for 1 minute with 5 mL of hexane: acetone (1:1 v/v), before ultrasonicating for 10 minutes at 30 °C. Samples were centrifuged at 3500 rpm for 3 min and the supernatant collected in a clean dry test tube. The steps from extraction to collection of supernatants were repeated twice and the combined extracts evaporated under a gentle stream of nitrogen to ~1 mL. The crude extracts were loaded onto pre-conditioned Florisil cartridges (conditioned with 2 x 3 mL of hexane) and the extract washed with 10 mL of hexane before elution of OPEs with 8 mL of ethyl acetate. The eluate was then collected in a clean dry test tube and evaporated under a gentle stream of nitrogen until incipient dryness. The concentrate was then reconstituted in 100 µL of toluene containing 250 pg/uL of PCB 62 as recovery determination (syringe) standard before transferring into an inserted vial and stored in a freezer ready for GC-MS analysis.

Analysis of OPEs was conducted on an Agilent 5975C GC coupled to an Agilent 5975C MSD fitted with a 30 m DB-5 MS column (0.25 mm ID, 0.25 µm film thickness) and operated in electron ionisation mode (EI) (Restek, USA). The carrier gas was helium at a constant flow rate of 1.0 mL/min. The injector temperature was set at 290 °C in split-less mode and the MS operated with a solvent delay of 5 minutes. Temperatures of the ion source, quadrupole and interface were set at: 230 °C, 150 °C and 300 °C respectively. The GC temperature programme was 65 °C, hold for 0.75 min, ramp 20 °C/min to 250 °C, hold for 1 min, ramp 5 °C/min to 260 °C, hold for 0 min, ramp 30 °C/min to 305 °C, and hold for 1 min. TnBP, TCEP, and TCIPP were quantified against d_{27}-TnBP, TDCIPP, TPHP, EHDPP, and TMPP against d_{12}-TPHP, while TDCIPP was quantified against d_{12}-TDCIPP. The dwell time for each ion was 30 milliseconds.

To ensure accuracy and precision of the analytical data generated during this study, the following measures were taken. A full five-point calibration comprising concentrations of each individual native OPE of 50, 100, 250, 500, and 1,000 pg/µL was conducted (with relative standard deviation (RSD) values for the relative response factors < 6.5% for all target OPEs. Concentrations of the internal standards in each calibration standard were 30 ng/µL and as an indication of the high efficiency of the extraction method, good recoveries (>77%) of the internal standards (d_{12}-TCEP, d_{15}-TPHP, and d_{12}-TDCIPP) were obtained in all samples. Two procedural blanks (comprising 1 g Na_2SO_4 treated as a sediment sample), and one standard reference material (SRM 1944) were analysed for each batch of 20 sediment samples. Low concentrations (5-20% of those found in samples from the same batch) of TCEP and TBOEP were detected in the procedural blanks and the average concentration detected in the blanks were subtracted from those in all samples from that batch. While to our knowledge no other data exist on OPEs in SRM1944 against which we can compare the accuracy of our method, our good internal standard recoveries and satisfactory blank levels provide reassurance of the quality of our data. The limit of detection (LOD) and the limit of quantification (LOQ) were calculated as the concentrations of analyte corresponding to signal to noise ratios of 3 and 10, respectively, except for TCEP and TBOEP where LOD and LOQ were calculated as 3 and 10 times the standard deviation of the blank levels.

Quality assurance and Quality control (QA/QC) for Analysis of Microplastics

Apart from ensuring the use of non-plastic materials during sampling and previous validation studies, other measures were taken to avoid contamination and ensure data accuracy (Onoja et al., 2022). These include a strict use of 100% cotton lab coats and rinsing all equipment before use under fast flowing tap water and then 3 to 4 times with deionised (DI) water (Wen et al., 2018, Yuan et al., 2019, Nel et al., 2020). Equipment was also covered with aluminium foil throughout the process and the workspace was always cleaned with 70% alcohol before use (Wen et al., 2018). A procedural blank which involved extracting an additional set-up of only DI water without sediment was run along each batch to account for background contamination. The number of particles (fibres) found in the blanks ranged between 0 and 1 and was therefore not used in the final count.

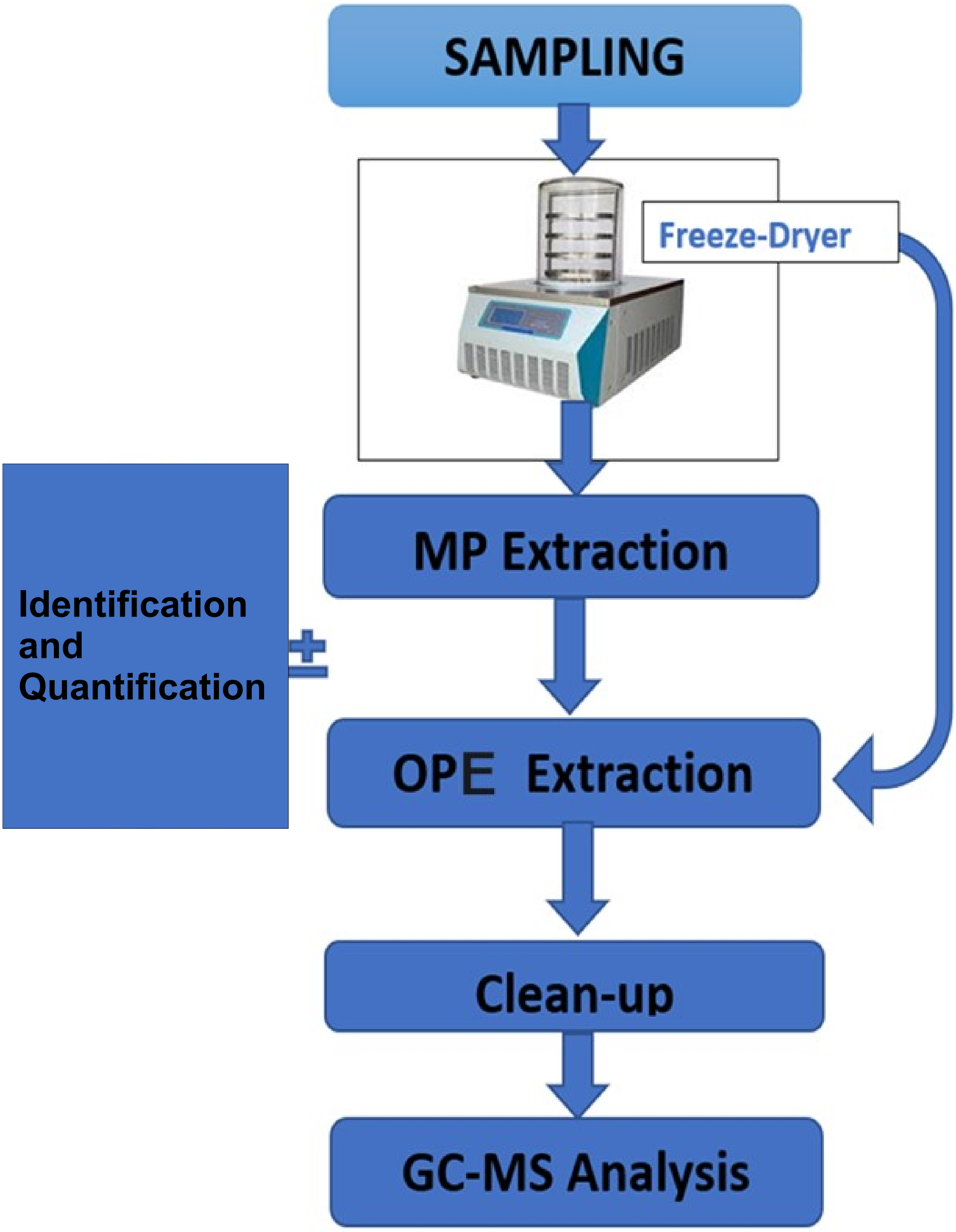

Figure S 1. Flow chart of the procedures for analysis of sediment samples for MPs and OPEs

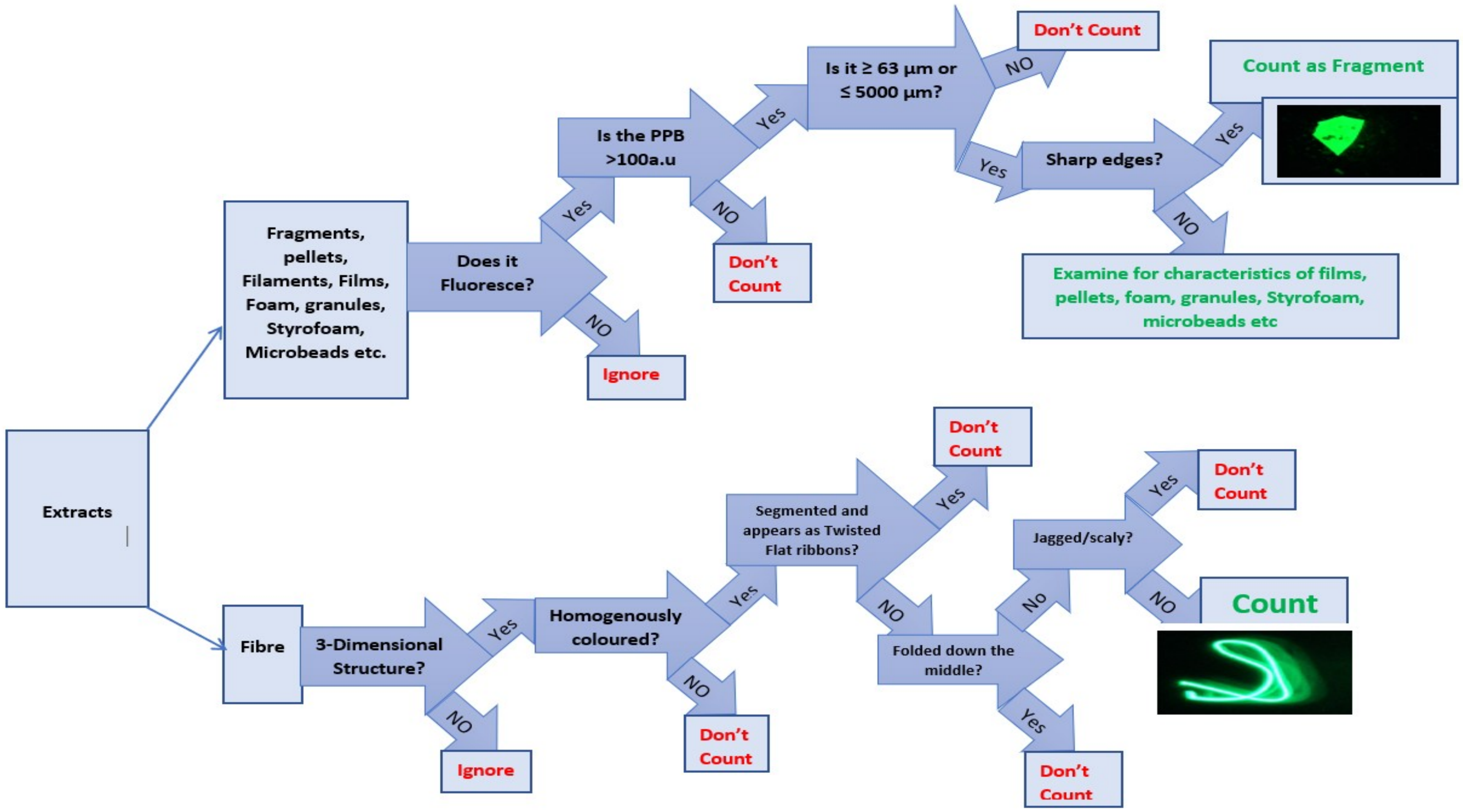

Figure S2: Fragment and Fibre identification tree (Nel et al., 2021, Hidalgo-Ruz et al., 2012, Catarino et al., 2018, Eerkes-Medrano et al., 2015, Nor and Obbard, 2014).

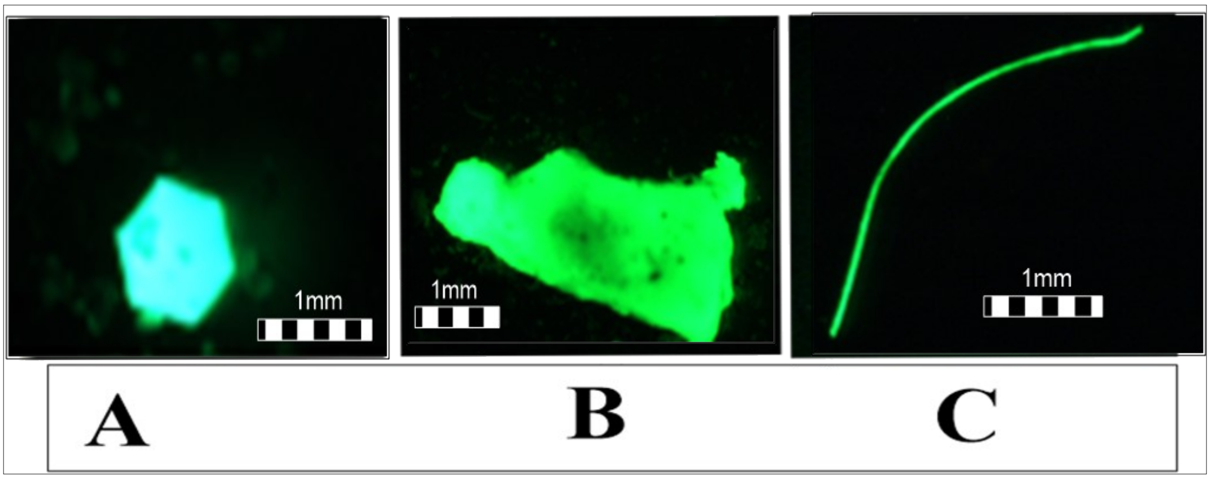

Figure S3: Pellet (A), Fragment (B), and Fibre (C) as identified by a Nikon SMZ-1000 stereo microscope.

Table S1: Calculation of Limit of Detection (LOD) and Limit of Quantification (LOQ) based on the average number of particles (fibres only) isolated during blank analysis (Horton et al., 2021).

Polymer	**mean particles/50 Kg dw⁻¹**	**SD**	**LOD (mean + 3 SD)**	**LOQ (mean + 10 SD)**
PVC	0.00	0.00	0.00	0.00
PP	5.00	0.31	5.93	8.10
PET	4.70	0.35	5.75	8.20
PE	3.80	0.43	5.09	8.10
PS	0.00	0.00	0.00	0.00

Table S2. MP abundance in River Sowe, River Tame, River Severn and Worcester-Birmingham canal.

Sampling Year/Month	Sampling Point	Mean MPs/30g (dw)	Mean MPs/kg (dw)	Median Particle/kg	Average length (µm)	Average Area (µm²)
RIVER SOWE						
2019-12	Downstream	4	133	83	390	67518
2019-12	Upstream	3	100	67	195	35877
2020-01	Downstream	5	167	100	291	45449
2020-01	Upstream	3	100	2	240	46463
2020-02	Downstream	7	233	74	342	52702
2020-02	Upstream	3	100	2	206	32228
2020-03	Downstream	8	267	167	445	91915
2020-03	Upstream	3	100	67	285	57049
2020-07	Downstream	1	33	33	394	59954
2020-07	Upstream	3	100	67	173	17992
2020-08	Downstream	9	300	167	395	174170
2020-08	Upstream	5	167	133	302	50094
2020-09	Downstream	5	167	100	634	31087
2020-09	Upstream	2	67	33	202	21152
2020-10	Downstream	2	67	33	270	31410
2020-10	Upstream	2	67	33	940	198025
2020-11	Downstream	3	100	67	591	66806
2020-11	Upstream	3	100	67	276	72275
2021-04	Downstream	3	100	67	596	55395
2021-04	Upstream	2	67	2	1196	6483
2021-05	Downstream	2	67	33	180	19821
2021-05	Upstream	4	133	67	482	54254
2021-06	Downstream	2	67	50	114	634
2021-06	Upstream	2	67	33	58	2278
Min		1	33	2	58	634
Max		9	300	167	1196	198025
Average		4	119	64	383	53793
Median		3	100	67	296	48279

River Tame						
2019-12	Downstream	9	300	167	102	8989
2019-12	Upstream	4	133	83	392	126719
2020-01	Downstream	11	367	200	281	122656
2020-01	Upstream	6	200	117	327	148300
2020-02	Downstream	9	300	200	368	113823
2020-02	Upstream	3	100	67	264	42173
2020-03	Downstream	9	300	167	433	118277
2020-03	Upstream	8	267	133	406	172851
2020-07	Downstream	7	233	133	438	303730

2020-07	Upstream	6	200	100	303	24131
2020-08	Downstream	8	267	4	307	53046
2020-08	Upstream	8	267	133	442	229627
2020-09	Downstream	5	167	100	336	60709
2020-09	Upstream	5	167	100	679	274954
2020-10	Downstream	3	100	67	888	6618607
2020-10	Upstream	5	167	100	346	303314
2020-11	Downstream	5	167	100	1382	2499860
2020-11	Upstream	7	233	4	1223	1175559
2021-04	Downstream	5	167	100	829	212205
2021-04	Upstream	4	133	83	314	63537
2021-05	Downstream	5	167	100	89	1242
2021-05	Upstream	3	100	67	39	729
2021-06	Downstream	5	167	100	118	2128
2021-06	Upstream	3	100	67	105	2008
Min		3	100	4	39	729
Max		11	367	200	1382	6618607
Average		6	199	104	434	528299
Median		5	167	100	341	120466

River Severn						
2019-12	Downstream	11	367	200	498	274579
2019-12	Upstream	8	267	133	624	390182
2020-01	Downstream	13	433	233	457	249138
2020-01	Upstream	6	200	133	654	278850
2020-02	Downstream	7	233	4	665	135997
2020-02	Upstream	4	133		793	270302
2020-03	Downstream	8	267	167	669	234610
2020-03	Upstream	7	233	167	525	194153
2020-07	Downstream	2	67	33	76	2755
2020-07	Upstream	6	200	100	570	126521
2020-08	Downstream	7	233	133	774	159720
2020-08	Upstream	5	167	100	1169	122243
2020-09	Downstream	7	233	133	343	65765
2020-09	Upstream	6	200		916	162562
2020-10	Downstream	6	200	100	413	120429
2020-10	Upstream	6	200	100	555	179088
2020-11	Downstream	4	133	67	488	111640
2020-11	Upstream	3	100	67	662	202881
2021-04	Downstream	2	67	33	373	62205
2021-04	Upstream	3	100	67	561	161829
2021-05	Downstream	4	133	67	61	2084
2021-05	Upstream	4	133	83	74	1165
2021-06	Downstream	2	67	33	49	1772
2021-06	Upstream	2	67	33	48	2229
Min		2	67	4	48	1165

Max		13	433	233	1169	390182
Average		6	185	99	501	146362
Median		6	200	100	540	147858

Birmingham and Worcester Canal						
2019-12	Worcester & Birm Canal	4	133	67	251	19404
2020-01	Worcester & Birm Canal	8	267	133	144	14414
2020-02	Worcester & Birm Canal	8	267	133	166	10911
2020-03	Worcester & Birm Canal	2	67	33	392	105097
2020-07	Worcester & Birm Canal	2	67	33	1615	29928
2020-08	Worcester & Birm Canal	2	67	2	629	208748
2020-09	Worcester & Birm Canal	4	133	100	589	301239
2020-10	Worcester & Birm Canal	3	100	67	378	66034
2020-11	Worcester & Birm Canal	2	67	33	1161	269882
2021-04	Worcester & Birm Canal	3	100	67	639	88388
2021-05	Worcester & Birm Canal	2	67	33	42	2862
2021-06	Worcester & Birm Canal	2	67	50	45	926
Min		2	67	2	42	926
Max		8	267	133	1615	301239
Average		4	117	63	504	93153
Median		3	83	58	385	47981

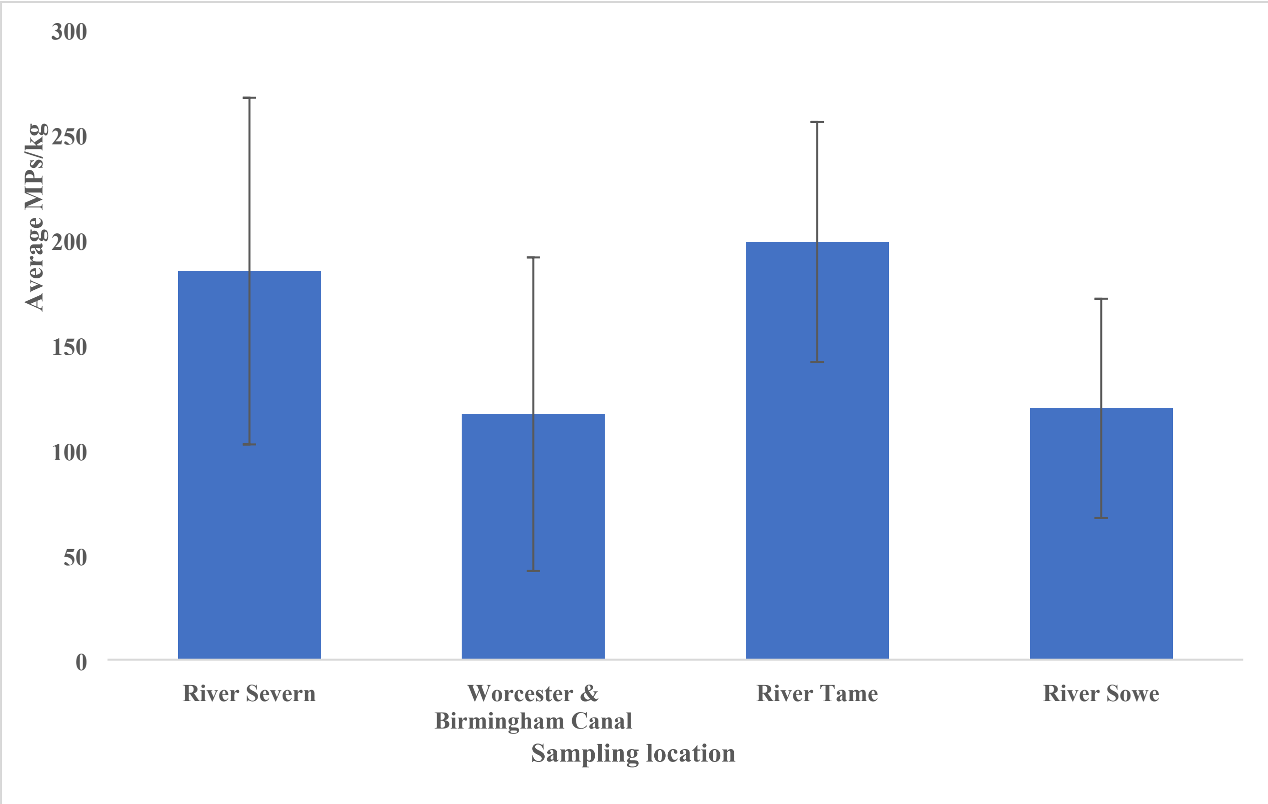

Figure S4. Average MP abundance in River Severn, River Sowe, River Tame and Worcester & Birmingham Canal. (Y error bars = 1 standard deviation).

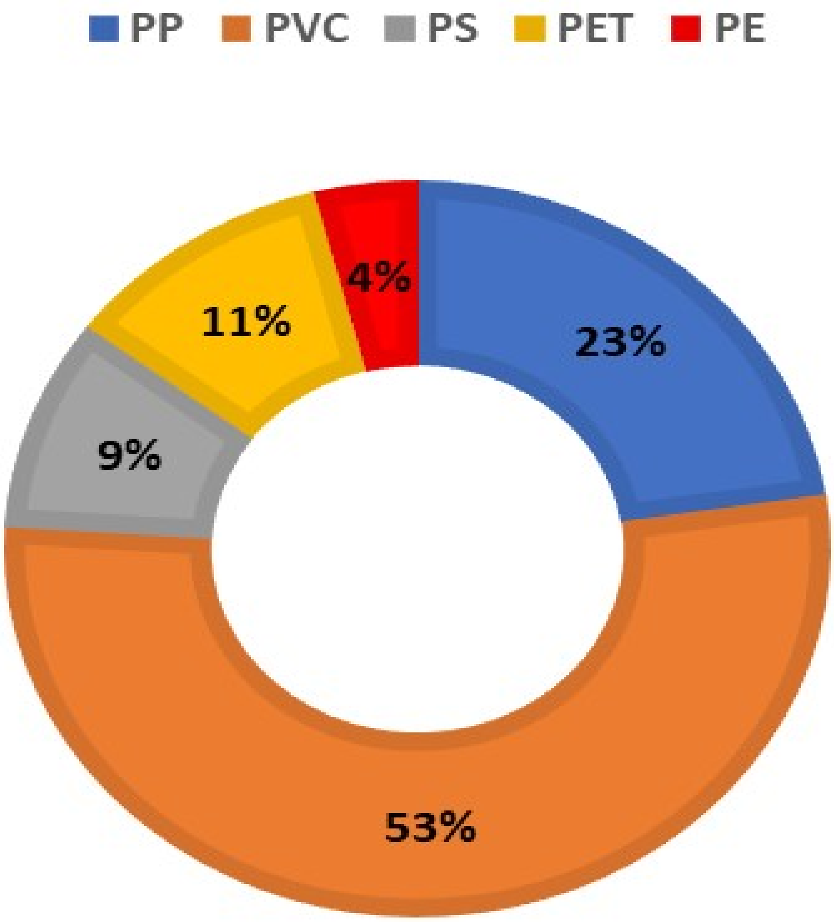

Figure S5: Relative abundance of the five polymer types identified.

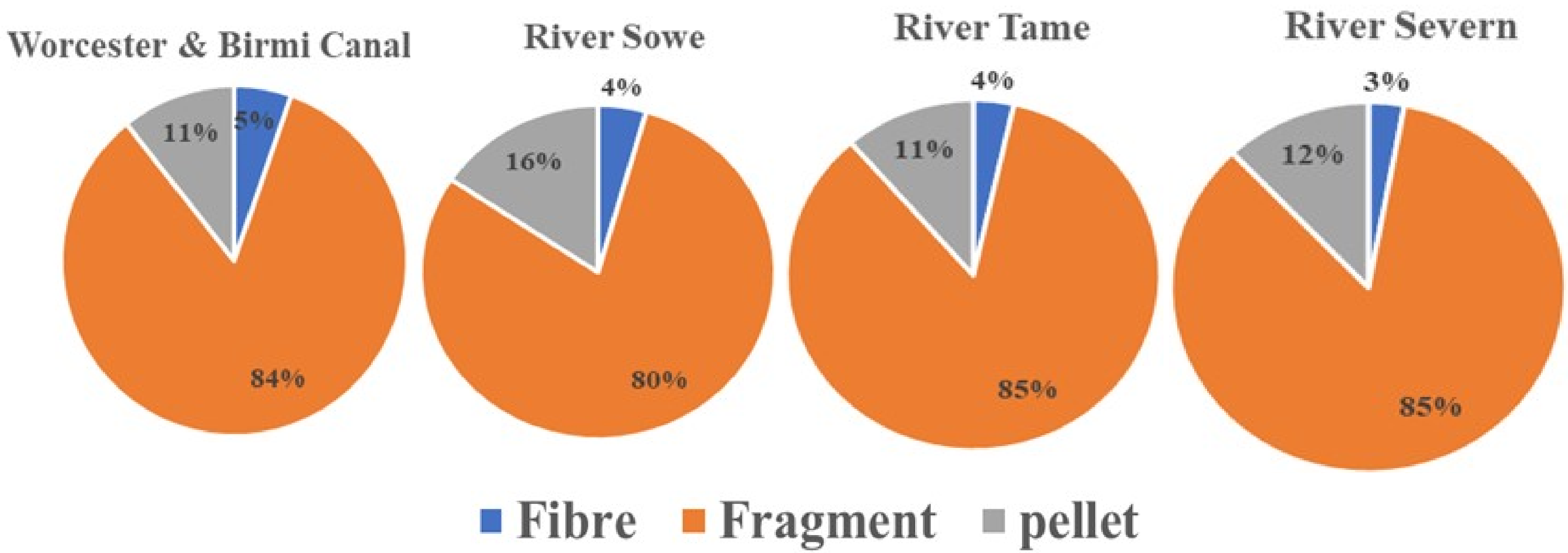

Figure S6a: Morphology of isolated MPs from the Worcester- Birmingham canal, River Tame, River Severn, and River Sowe.

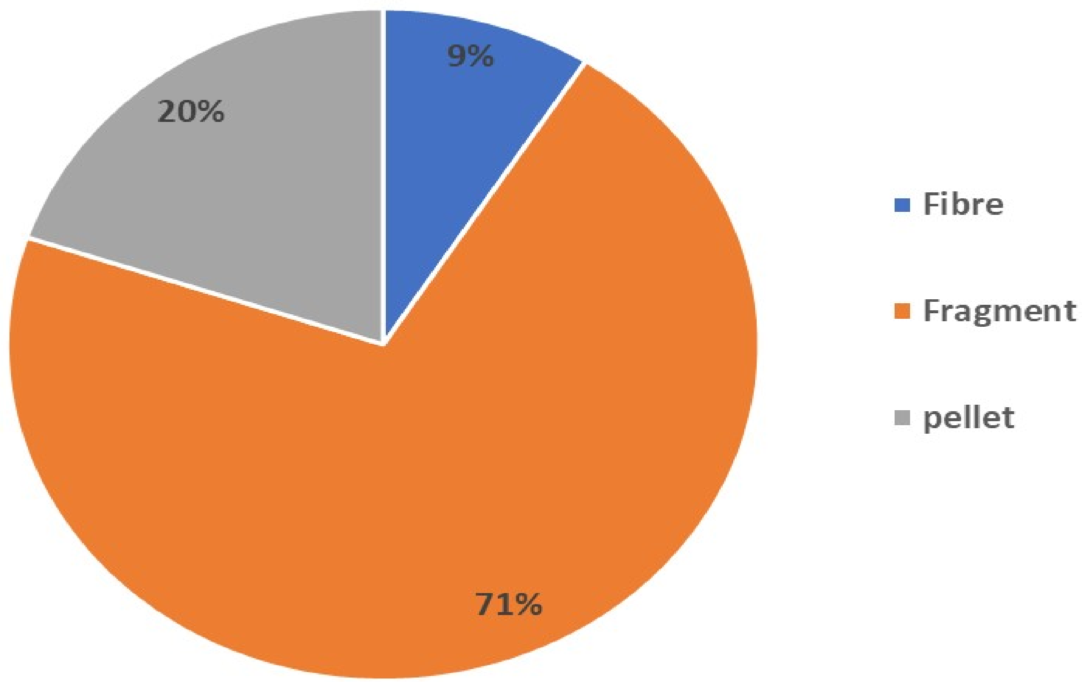

Figure S6b: Average morphology of isolated MPs from all sampling locations over the entire period of sampling.

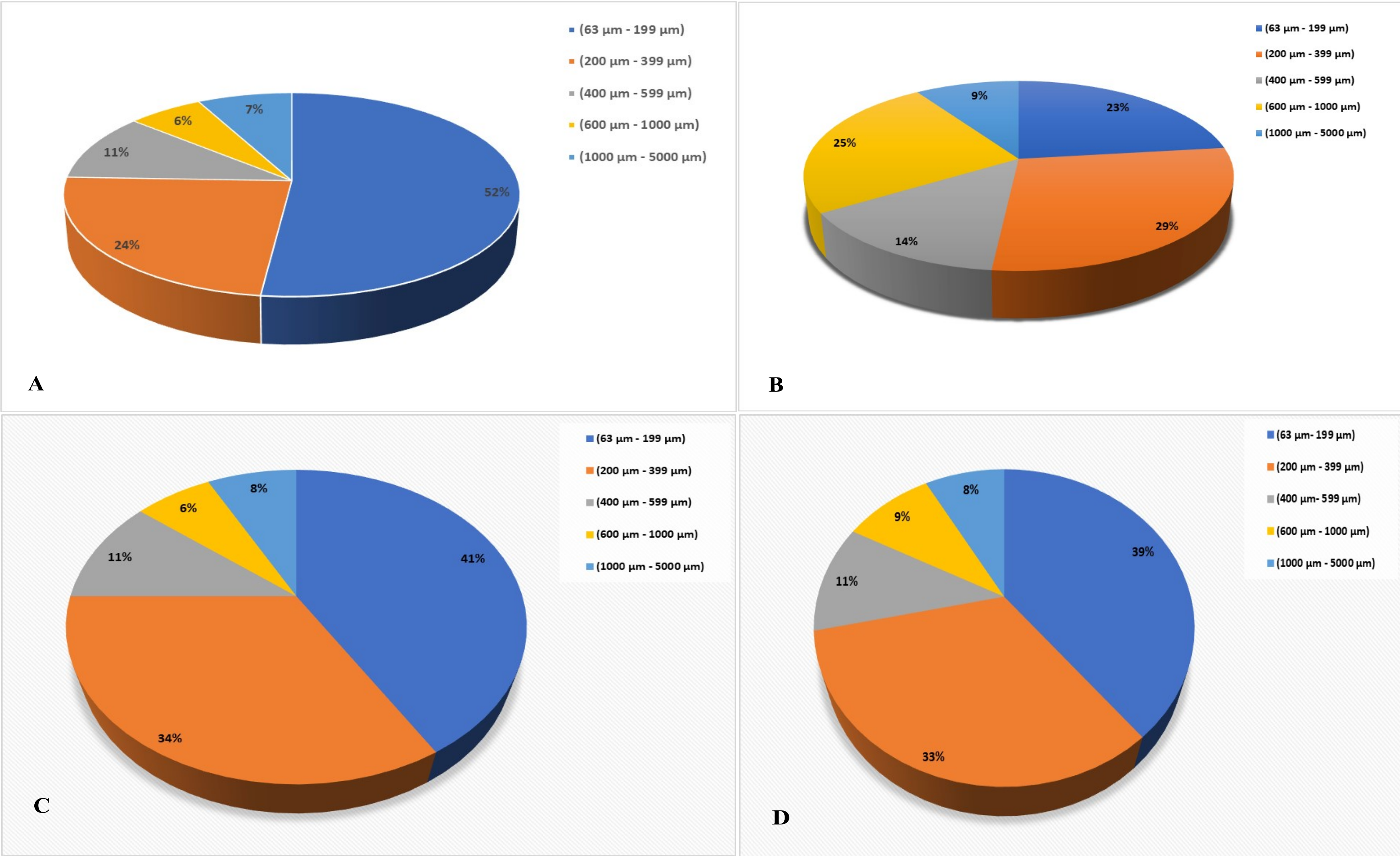

Figure S7: Size range of MPs at Worcester and Birmingham Canal (A), River Severn (B), River Sowe (C), and River Tame (D).

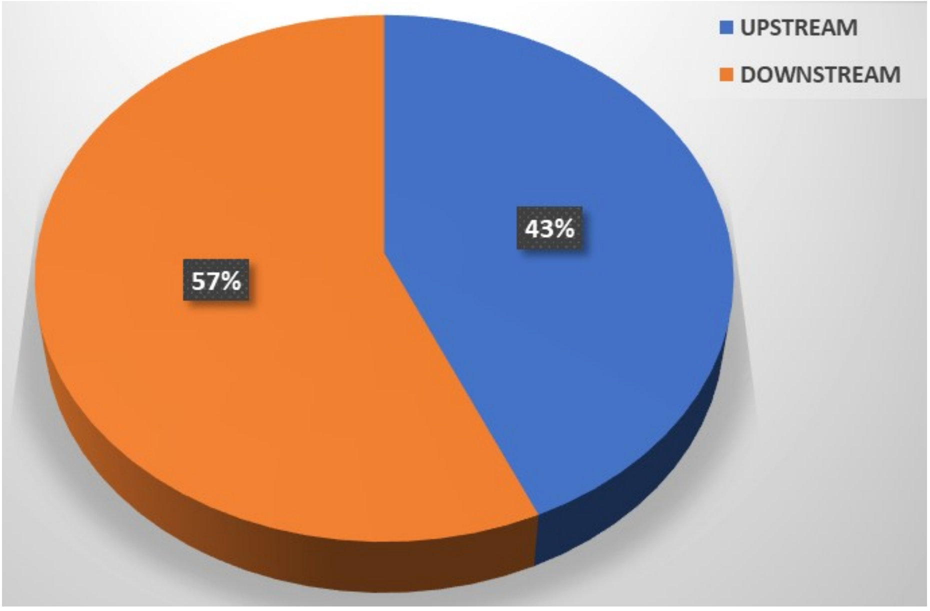

Figure S8: Distribution of Mean MPs upstream and downstream of WWTPs across all three river locations over the 12-month sampling period.

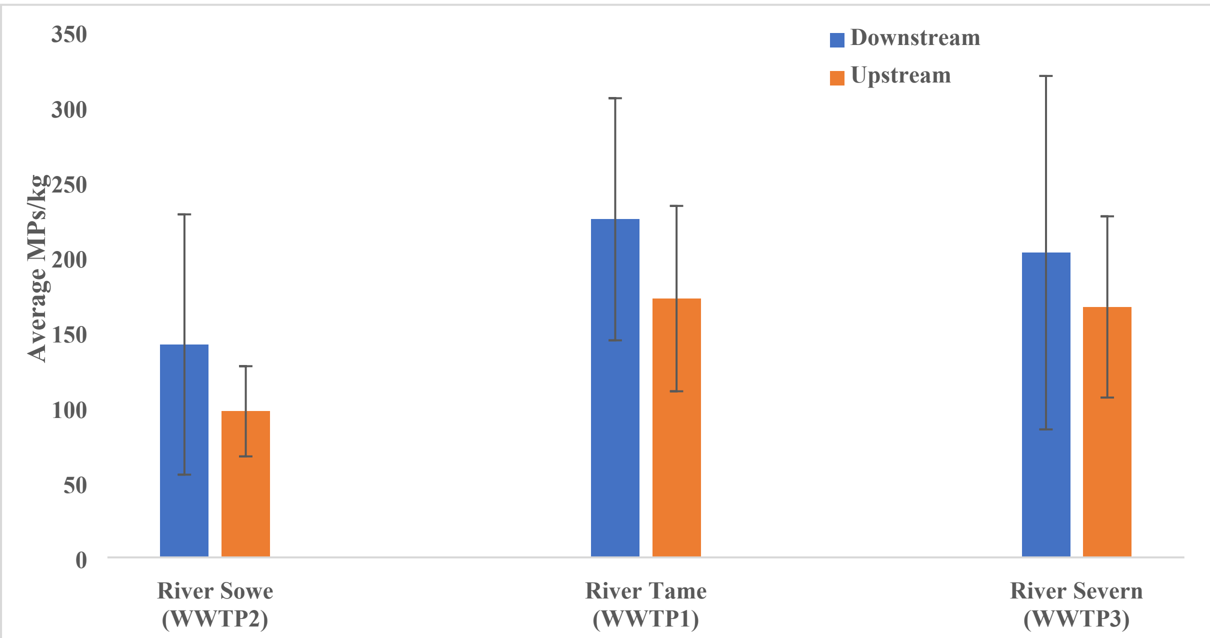

Figure S9. Abundance of MPs Upstream and Downstream of WWTPs. (Y error bars are standard deviation error bars showing variation around the mean)

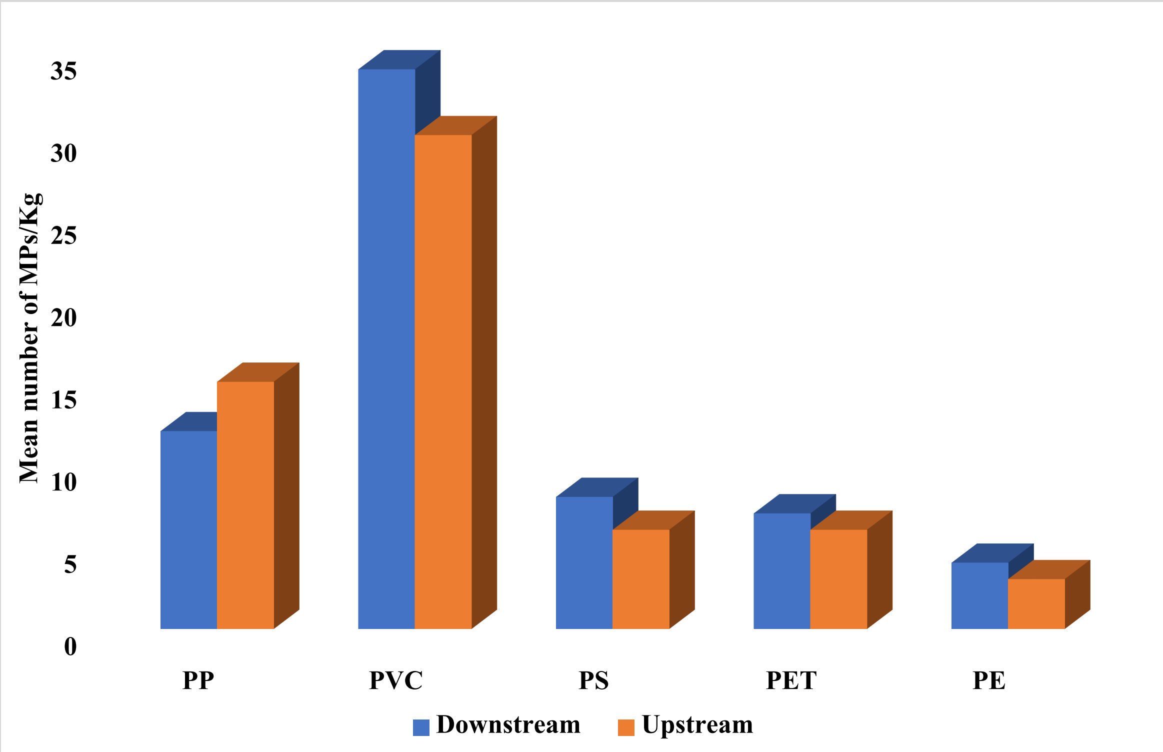

Figure S10: Relative abundance of the five polymer types upstream and downstream of the target WWTPs

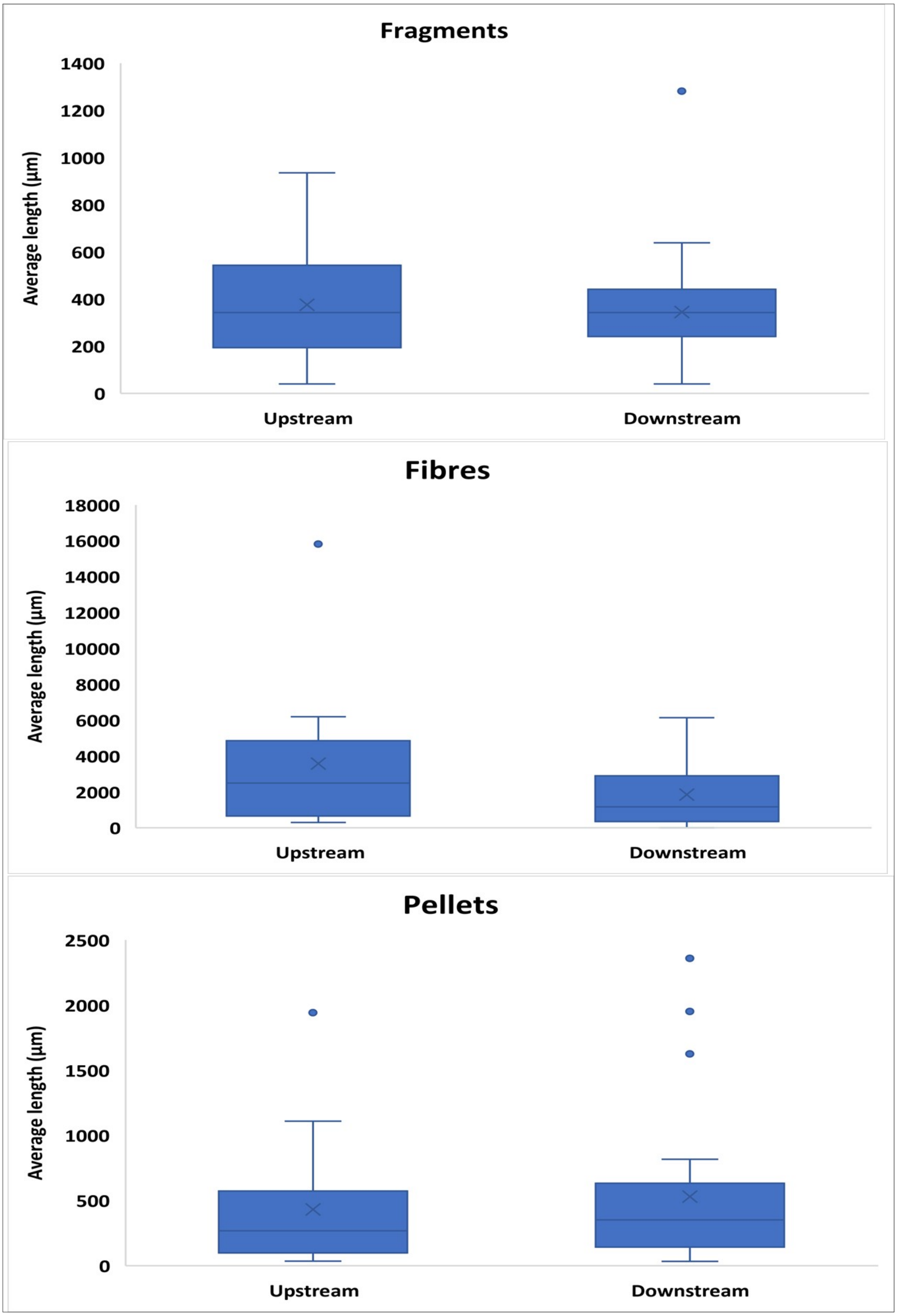

Figure S11: Box plots showing the distribution of fragment, fibre, and pellet length upstream and downstream.

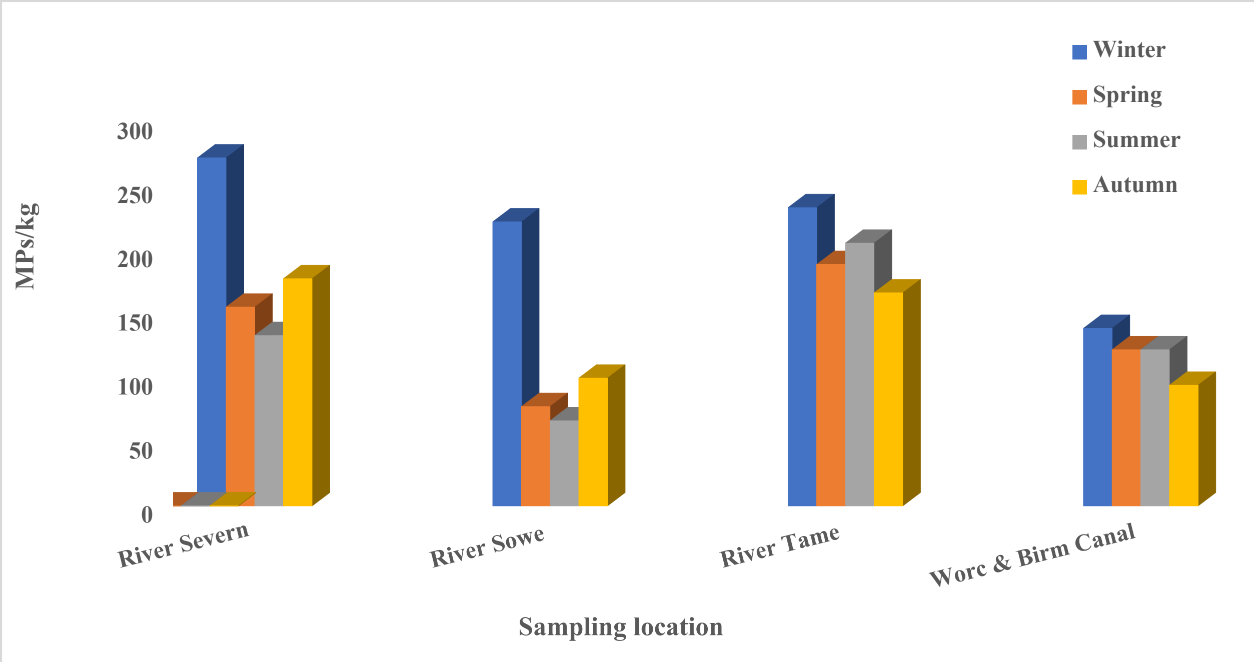

Figure S12. Seasonal variation of MPs abundance across study locations

Table S2: UK seasons, months, notable features, and mean MP abundance at Rivers, Severn, Sowe, Tame and Worcester-Birmingham Canal

Season	Months	Notable features	Mean MPs/kg at River Severn	Mean MPs/kg at River Sowe	Mean MPs/kg at River Tame	Mean MPs/kg at W&B Canal
Summer	June to end of August	• Usually has the hottest temperatures. • Sunniest days • Sometimes driest season • Varying rainfall as with all seasons in the UK	133	67	206	122
Autumn	September to November	• Cooler temperature • Stormier weather • Shorter days.	178	100	167	94
Winter	December to February	• Coldest months • Shortest days • Often wet and windy • Frost and even snow often	272	222	233	139
Spring	March to May	• longer and warmer days • Often calm and dry	156	78	189	122

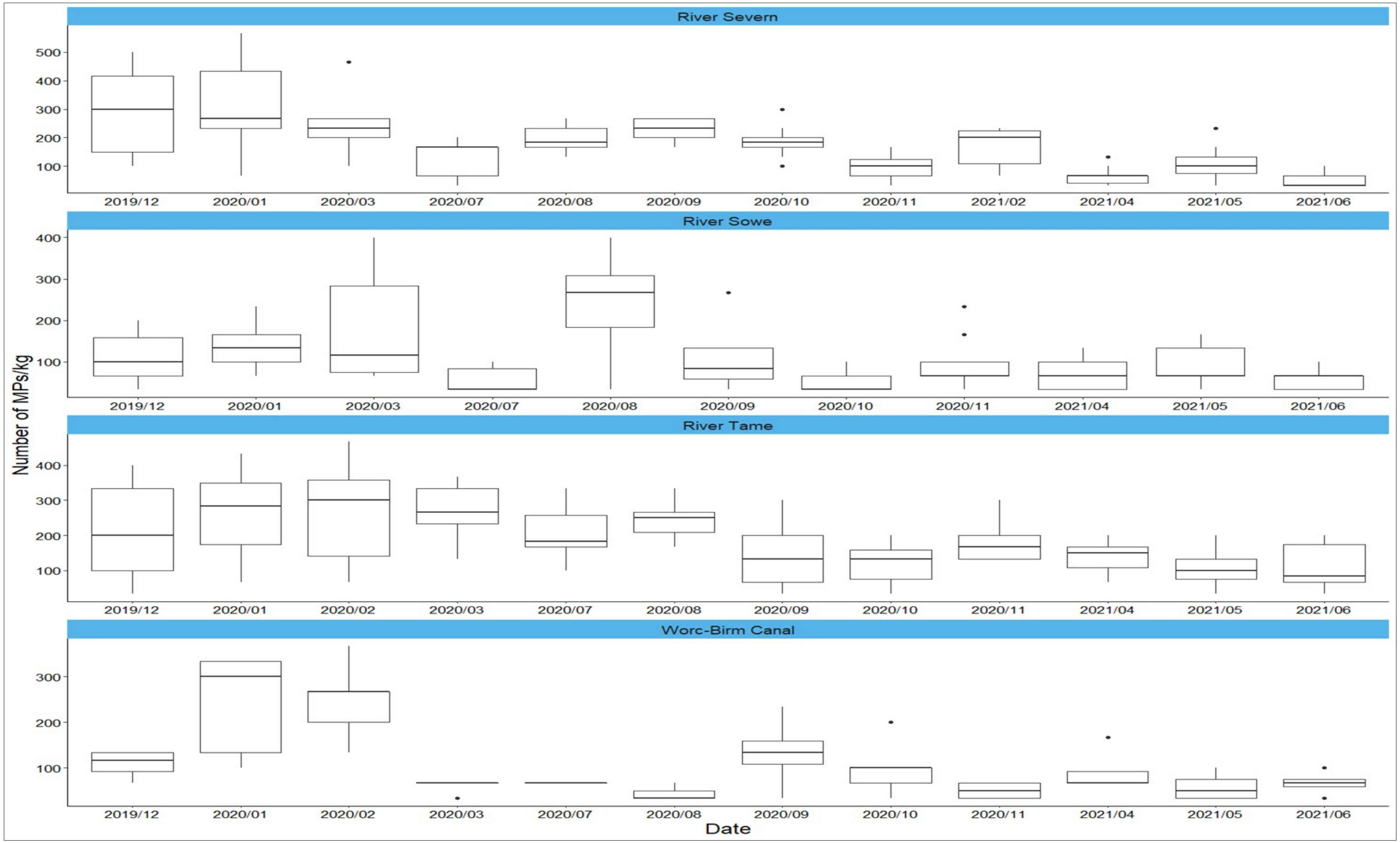
River Severn
500
400
300
200
100
2019/12
2020/01
2020/03
2020/07
2020/08
2020/09
2020/10
2020/11
2021/02
2021/04
2021/05
2021/06
River Sowe
400
300
200
100
2019/12
2020/01
2020/03
2020/07
2020/08
2020/09
2020/10
2020/11
2021/04
2021/05
2021/06
River Tame
400
300
200
100
2019/12
2020/01
2020/02
2020/03
2020/07
2020/08
2020/09
2020/10
2020/11
2021/04
2021/05
2021/06
Worc-Birm Canal
300
200
100
2019/12
2020/01
2020/02
2020/03
2020/07
2020/08
2020/09
2020/10
2020/11
2021/04
2021/05
2021/06
Number of MPs/kg
Date

Figure S 13: Boxplots of the total number of MPs/kg for each month across all four study locations (River Severn, River Sowe, River Tame and Worcester & Birmingham Canal

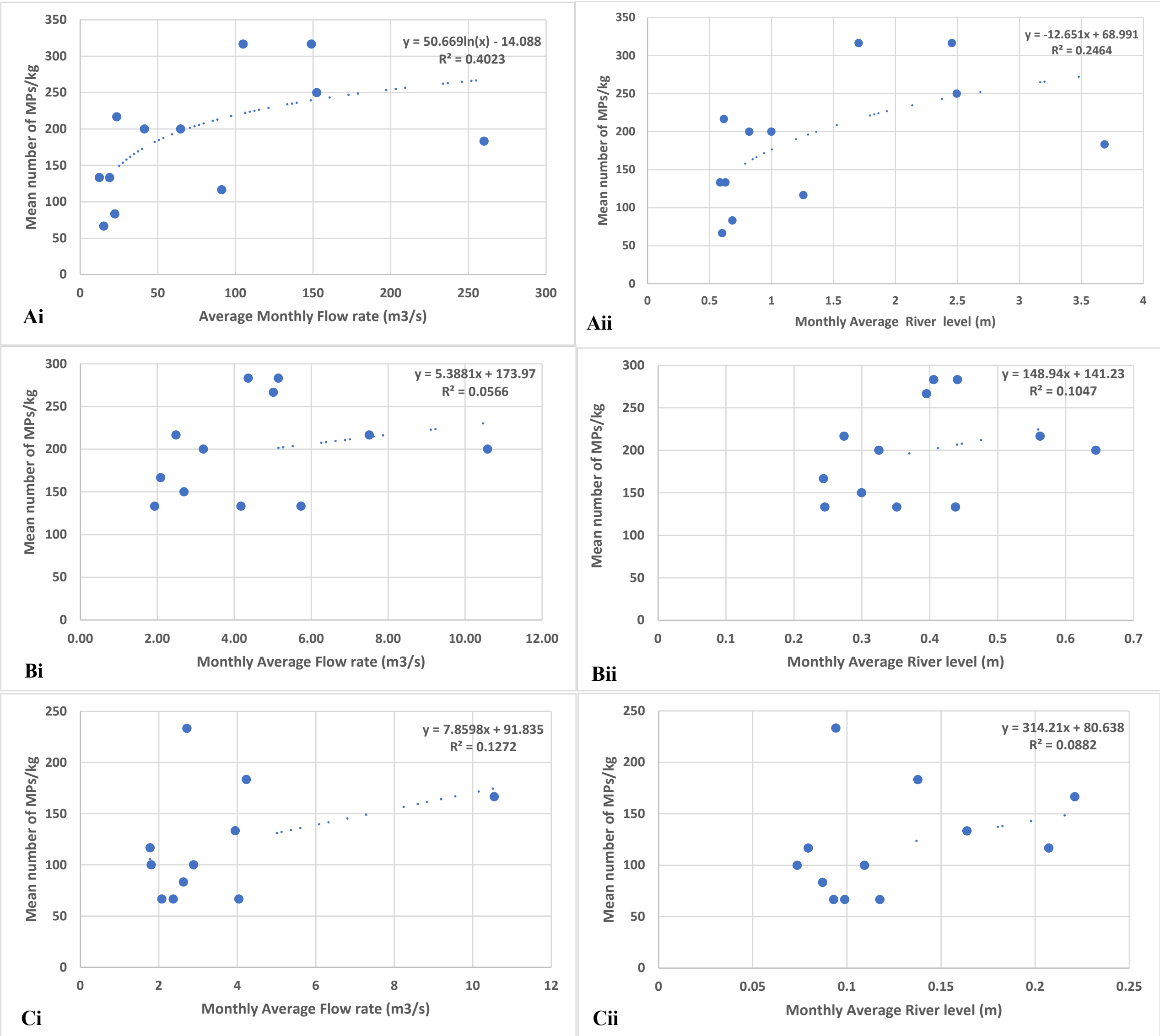

Figure S14. Relationship between MP abundance, River flow rate (i) and River level (ii) in River Severn (A), River Tame (B) and River Sowe.

Table S3: Correlation test between total MPs/kg, River level and Flow rate at River Severn

River Severn				
		Mean MPs//Kg	River Level (m)	Flow Rate (m^3/s)
Mean MPs//Kg	Pearson Correlation	1	.584*	.694*
	Sig.		.046	.026
River Level (m)	Pearson Correlation		1	.982**
	Sig.			<.001
River Level (m)	Pearson Correlation			1
*. Correlation is significant at the 0.05 level				
**. Correlation is significant at the 0.01 level				

Table S4: Correlation test between total MPs/kg, River level and Flow rate at River Tame

River Tame				
		Mean MPs//Kg	River Level (m)	Flow Rate (m^3/s)
Mean MPs//Kg	Pearson Correlation	1	.370	.353
	Sig.		.236	.317
River Level (m)	Pearson Correlation		1	.993**
	Sig.			<.001
River Level (m)	Pearson Correlation			1
**. Correlation is significant at the 0.01 level				

Table S5: Correlation test between total MPs/kg, River level and Flow rate at River Sowe

River Sowe				
		Mean MPs//Kg	River Level (m)	Flow Rate (m^3/s)
Total MPs//Kg	Pearson Correlation	1	.463	.575
	Sig.		.130	.105
River Level (m)	Pearson Correlation		1	.953**
	Sig.			<.001
Mean MPs//Kg	Pearson Correlation			1
**. Correlation is significant at the 0.01 level				

Table S6: Correlation test between total OPE concentration in all locations and the mean particle number, median particle number, mean particle area and median particle area in all study locations.

		TnBP	TCEP	TCIPP	TBOEP	EHDPP	TMTP	TPhP	TDCIPP	$\sum_8$ OPEs
Mean MPs number	Pearson Correlation	-.045	-.144	-.214	-.309**	-.012	-.016	-.217*	-.119	-.326**
	Sig.	.685	.192	.051	.004	.911	.888	.047	.282	.002
Median MPs number	Pearson Correlation	-.053	-.143	-.188	-.295**	.030	.002	-.166	-.135	-.299**
	Sig.	.635	.194	.086	.007	.787	.985	.131	.220	.006
Mean MPs Area_µm2	Pearson Correlation	-.154	-.193	-.114	-.139	-.024	-.019	-.207	-.188	-.165
	Sig.	.161	.079	.302	.207	.830	.865	.059	.086	.134
Median MPs Area_µm2	Pearson Correlation	-.209	-.196	-.137	-.262*	-.104	-.114	-.216*	-.113	-.283**
	Sig.	.056	.074	.213	.016	.348	.301	.049	.306	.009

**. Correlation is significant at the 0.01 level

*. Correlation is significant at the 0.05 level

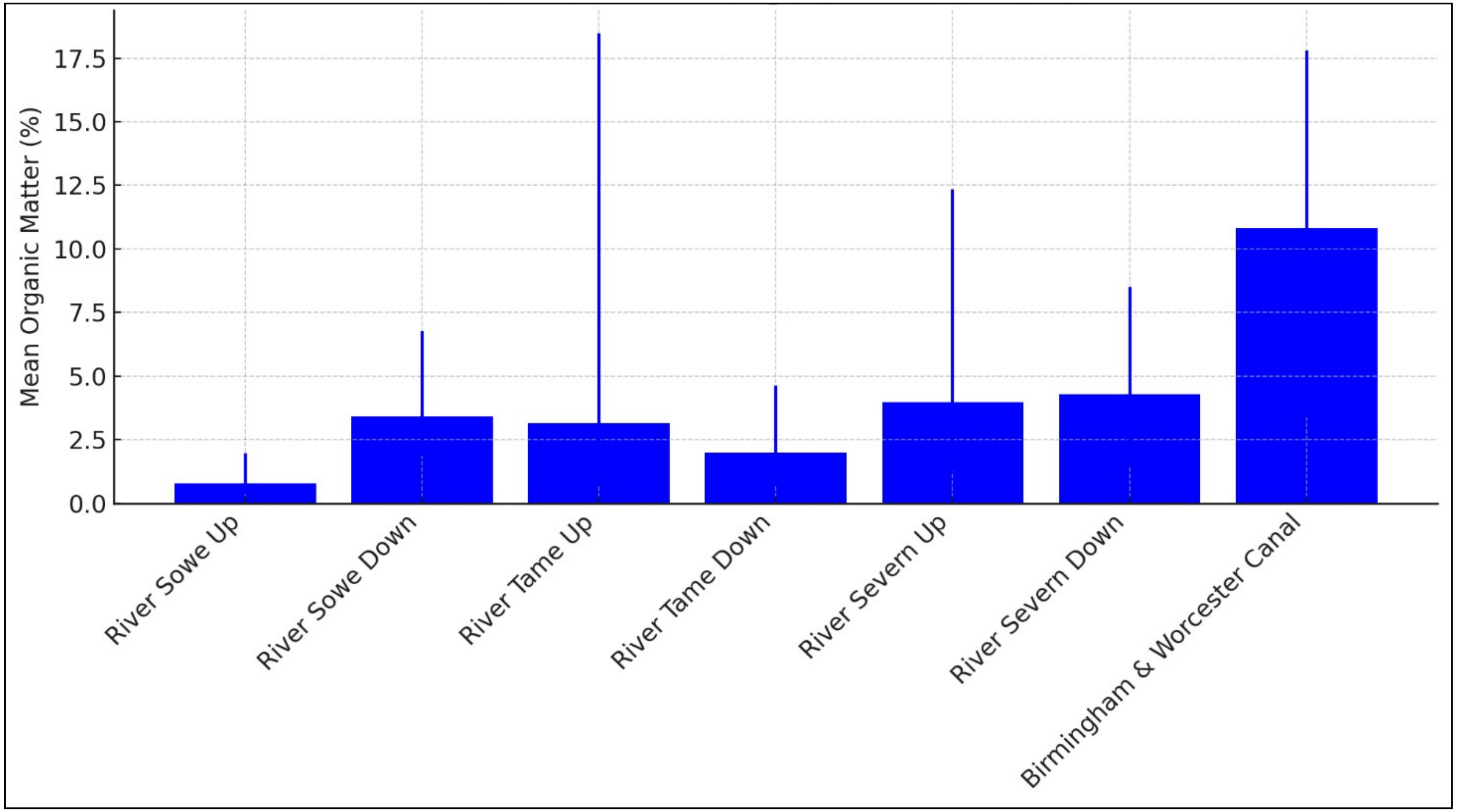

Figure S15: Mean organic matter content (%) upstream and downstream of River Severn, River Sowe, River Tame and Worcester & Birmingham Canal. (Y error bars = range).

Table S7: Names, abbreviations and properties some common OPEs including the ones in the present study (Yin et al., 2022)

Compound	Abb.	CAS No.	Chemical formula	Solubility c	VP b,c	Log Kow	Log Koc
				(mg/L, 25°C)	(Pa, 25 °C)	c	
Tris (2-chloroethyl) phosphate	TCEP	115-96-8	C6H12Cl3O4P	877.9	8.17	1.44	2.48
Tris (1-chloro-2- propyl) phosphate	TCIPP	13674-84-5	C9H18Cl3O4P	51.85	7.53×10-3	2.59	2.71
Tris (1,3-dichloro- 2-propyl)	TDCIPP	13674-87-8	C9H15Cl6O4P	1.5	3.81×10-5	3.65	2.35
phosphate							
Trimethyl phosphate	TMP	512-56-1	C3H9O4P	3.004×105	55.3	-0.65	4.35
Triethyl phosphate	TEP	78-40-0	C6H15O4P	1.115×104	22	0.8	1.68
Tripropyl phosphate	TnPP	513-08-6	C9H21O4P	826.6	3.08	1.87	2.83
Tributyl phosphate	TnBP	126-73-8	C12H27O4P	280	0.151	4	3.28
Triphenyl phosphate	TPhP	115-86-6	C18H15O4P	1.9	1.49×10-3	4.59	3.72
Tris(2-butoxyethyl) phosphate	TBOEP	78-51-3	C18H39O7P	1.963	1.65×10-4	3.75	4.38
Tris(2-ethylhexyl) phosphate	TEHP	78-42-2	C24H51O4P	1.461×10-5	1.10×10-5	9.49	6.87
2-ethylhexyl diphenyl phosphate	EHDPP	1241-94-7	C20H27O4P	0.06659	4.45×10-3	5.37	4.21

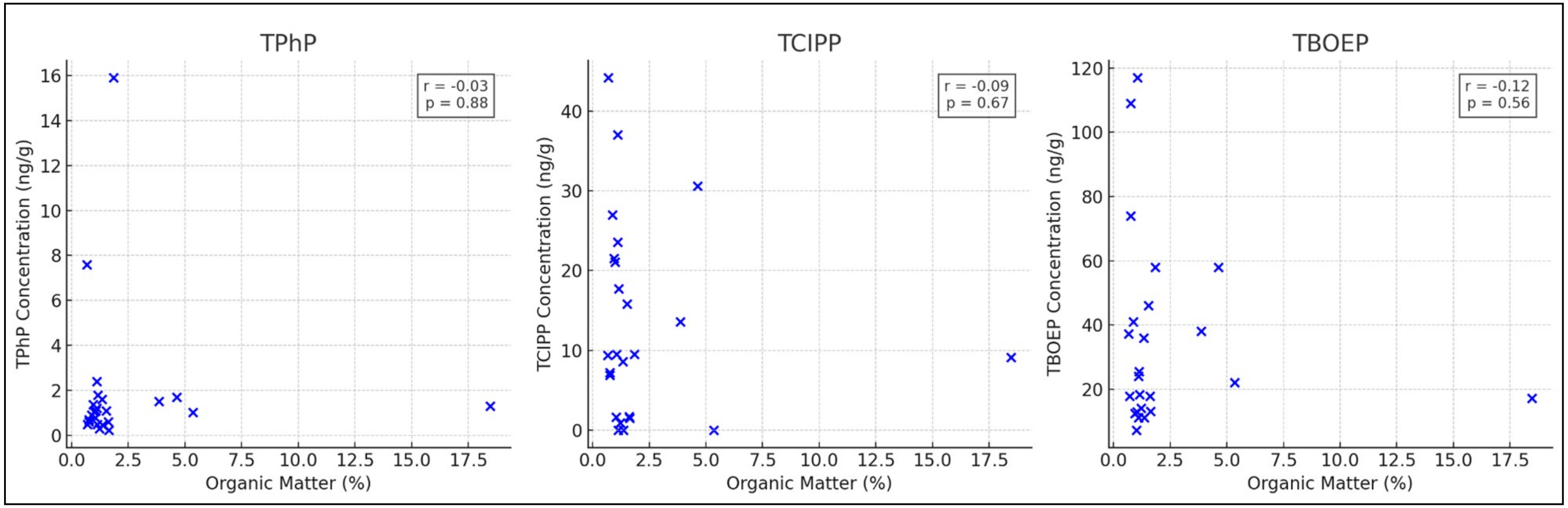

Figure S16: River Tame scatterplots for TPhP, TCIPP and TBOEP against organic matter (%) with the Pearson r and p values.

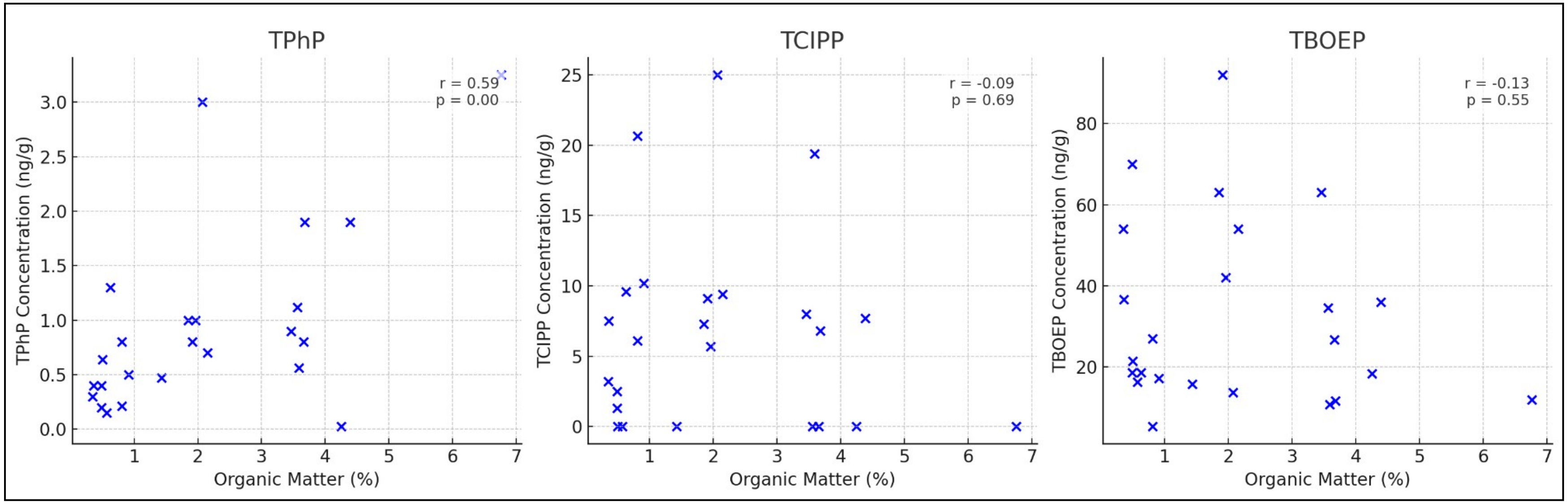

Figure S17: River Sowe scatterplots for TPhP, TCIPP and TBOEP against organic matter (%) with the Pearson r and p values.

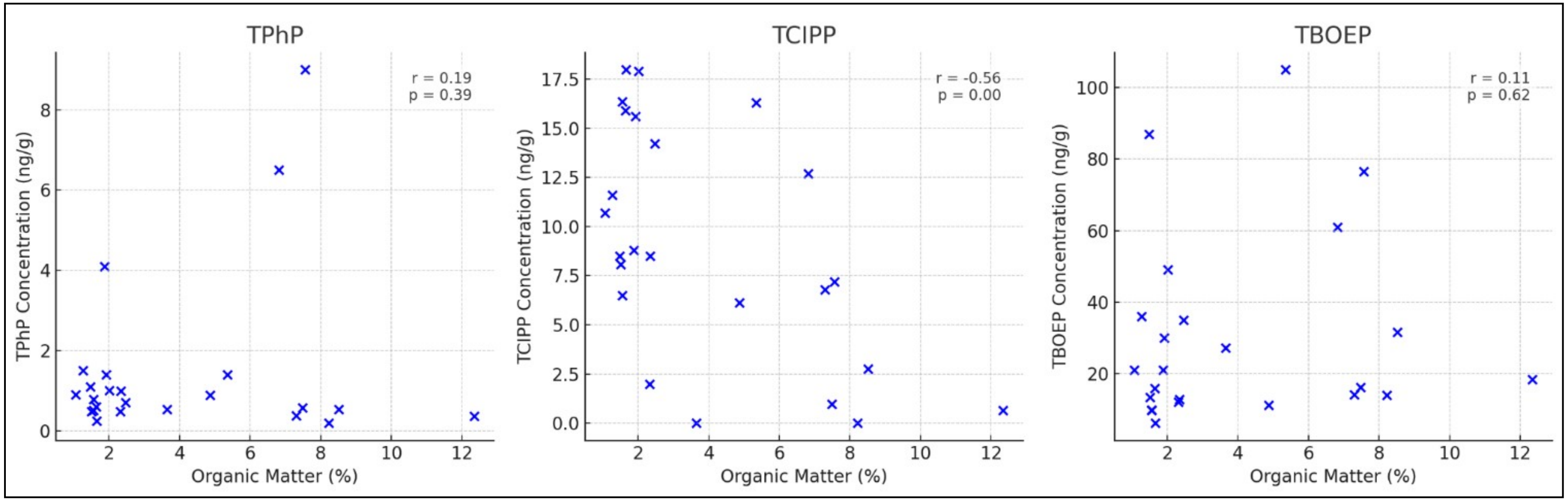

Figure S18: River Severn scatterplots for TPhP, TCIPP and TBOEP against organic matter (%) with the Pearson r and p values.

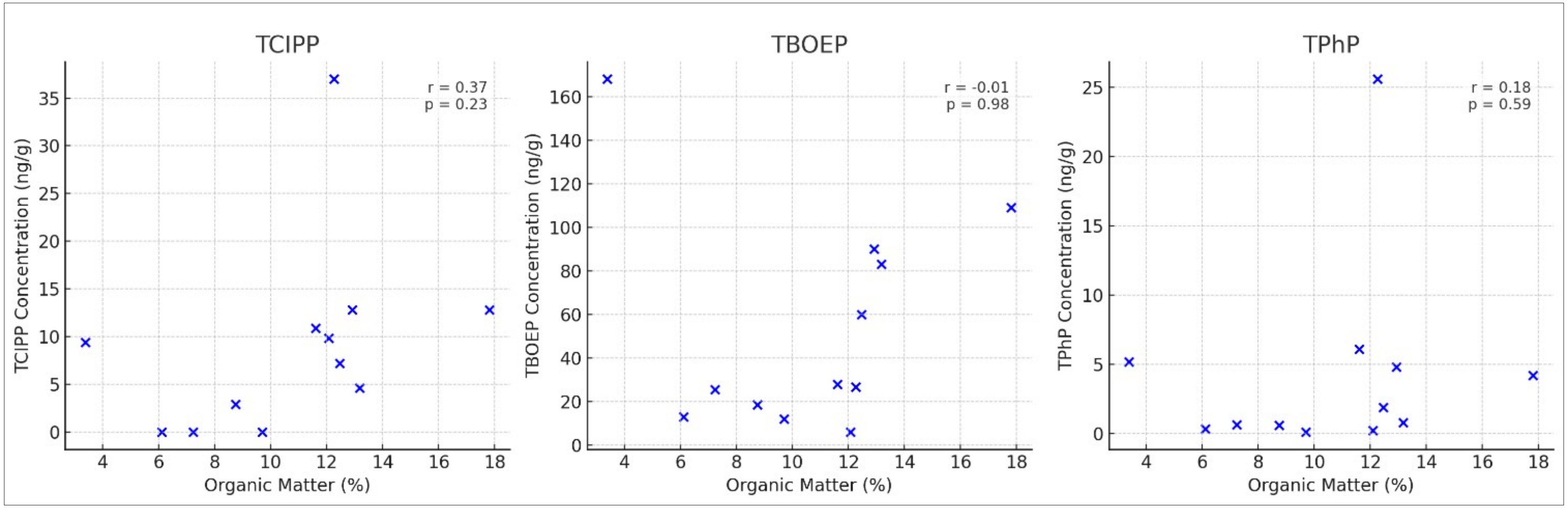

Figure S19: Birmingham and Worcester Canal scatterplots for TPhP, TCIPP and TBOEP against organic matter (%) with the Pearson r and p values.

Table S8 Correlation test between $\sum_8$ OPEs concentration and the mean particle number at the Worcester & Birmingham Canal.

Worcester & Birmingham Canal			
		$\sum_8$ OPEs	Mean particle number
$\sum_8$ OPEs	Pearson Correlation	1	-.588*
	Sig.		.044
Mean particles number	Pearson Correlation	-.588*	1
	Sig.	.044	
*. Correlation is significant at the 0.05 level			

Table S9 Correlation test between mean MPs/kg and mean concentration of individual target OPE at the Worcester & Birmingham Canal.

Worcester & Birmingham Canal									
		TnBP	**TCEP**	**TCIPP**	**TBOEP**	**EHDPP**	**TMTP**	**TPhP**	**TDCIPP**
Mean MPs/kg (dw)	Pearson Correlation	-.297	-.339	-.433	-.390	-.411	-.408	-.352	-.404
	Sig.	.348	.280	.160	.210	.185	.188	.262	.192
*. Correlation is significant at the 0.05 level									

**. Correlation is significant at the 0.01 level

Table S10 Correlation test between $\sum_8$ OPEs concentration, and the mean particle number, median particle number and mean particle area at the River Sowe.

River Sowe		$\sum_8$ OPEs
Mean particles number	Pearson Correlation	-.156
	Sig.	.466
Median Particle number	Pearson Correlation	-.144
	Sig.	.502
Area (μm²)	Pearson Correlation	-.053
	Sig.	.806
**. Correlation is significant at the 0.01 level. *. Correlation is significant at the 0.05 level.		

Table S11 Correlation test between mean MPs/kg and mean concentration of individual target OPEs at the River Sowe.

River Sowe									
		TnBP	TCEP	TCIPP	TBOEP	EHDPP	TMTP	TPhP	TDCIPP
Mean MPs/kg (dw)	Pearson Correlation	.483	.301	-.384	-.476	-.214	-.641*	-.151	.030
	Sig.	.112	.342	.217	.118	.503	.025	.640	.925
*. Correlation is significant at the 0.05 level									
**. Correlation is significant at the 0.01 level									

Table S12 Correlation test between $\sum_8$ OPEs concentration, and the mean particle number, median particle number, median particle area and mean particle area at River Tame

River Tame		$\sum_8$ OPEs
Mean particles number	Pearson Correlation	-.444*
	Sig.	.030
Median Particle number	Pearson Correlation	-.477*
	Sig.	.019
Mean Particle Area_µm2	Pearson Correlation	.049
	Sig.	.821
Median Particle Area_µm2	Pearson Correlation	-.002
	Sig.	.991
**. Correlation is significant at the 0.01 level		
*. Correlation is significant at the 0.05 level		

Table S13 Correlation test between MPs/kg and concentration of individual target OPEs at River Tame

River Tame									
		TBOEP	**TPhP**	**TnBP**	**TCEP**	**TCIPP**	**EHDPP**	**TMTP**	**TDCIPP**
Mean MPs/kg (dw)	Pearson Correlation	-.531	-.198	-.093	-.388	-.273	-.415	-.258	-.102
	Sig.	.076	.538	.774	.213	.390	.180	.418	.752
*. Correlation is significant at the 0.05 level									

**. Correlation is significant at the 0.01 leve

Table S14 Correlation test between $\sum_8$ OPEs concentration, and the mean particle number, median particle number, and mean particle area at River Severn

River Severn		**$\sum_8$ OPEs**
Mean particles number	Pearson Correlation	-.230
	Sig.	.280
Mean particle Area_µm2	Pearson Correlation	-.411*
	Sig.	.046
Median particle Area_µm2	Pearson Correlation	-.447*
	Sig.	.029
**. Correlation is significant at the 0.01 level. *. Correlation is significant at the 0.05 level.		

Table S15 Correlation test between mean concentration of individual target OPEs and mean MPs/kg at River Severn

River Severn									
		TnBP	**TCEP**	**TCIPP**	**TBOEP**	**EHDPP**	**TMTP**	**TPhP**	**TDCIPP**
Mean MPs/kg (dw)	Pearson Correlation	-.628*	-.092	-.554	-.411	-.008	-.088	-.277	-.091
	Sig.	.029	.777	.062	.184	.979	.785	.383	.778
*. Correlation is significant at the 0.05.									
**. Correlation is significant at the 0.01 level.									

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