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Supporting Information

SI.1 Water chemistry characterisation

Table SI.1: Water (river water, surface porewater and subsurface porewater) chemistry characterisation. [1]Concentrations of nitrite (and nitrate below IC detection limit (42 µM)) have been obtained from segmented-flow analysis and not from IC analysis.

	S1			S2			S3			S4		
	River	**Surface**	**Subsurface**	**River**	**Surface**	**Subsurface**	**River**	**Surface**	**Subsurface**	**River**	**Surface**	**Subsurface**
Salinity	0.4	0.3	0.2	3.5	3.1	1.8	21.6	17.0	17.7	26.1	28.0	32.1
pH	7.87	-	-	7.52	-	-	7.90	-	-	8.02	-	-
Conductivity (mS/cm)	0.7383	-	-	5.731	-	-	30.48	-	-	36.42	-	-
NO_3^- (µM)	266	36	37	250	17	26	248	66	17[1]	24[1]	78	7[1]
NO_2^- (µM)[1]	1.6	0.2	0.4	1.6	0.1	0.3	0.4	0.9	<LDL	0.7	1.0	<LDL
NH_4^+ (µM)	7	12	67	7	25	73	12	73	994	23	166	126
SO_4^{2-} (mM)	1	2±0	2±0	3	6±1	3±1	16	33±4	33±2	22	32±4	40±2
Cl^- (mM)	2	4	3	38	49	28	306	265	276	443	347	501
Al (µM)	0.26	0.53	0.29	0.32	0.43	0.37	47.44	47.81	47.44	49.30	41.88	48.93
Fe (µM)	0.1	0.4	4.9	0.1	0.1	0.3	1.2	1.6	3.6	1.8	0.9	3.3
Mn (µM)	1.4	3.4	82.3	1.0	5.1	49	0.6	60	0	23	15	62
Zn (µM)	0.15	0.12	0.01	0.16	0.06	0.04	8.49	3.87	3.52	8.03	3.53	3.53
Cu (µM)	0.05	0.08	0.10	0.05	0.06	0.05	3.70	8.26	8.32	3.76	8.94	8.06

SI.2 Quality Control information for the IC and Autoanalyser analyses

Detection limits of nutrients were calculated as three times the standard deviation of analyses of 5 Milli-Q (Millipore) ultra-pure water samples. For ammonium, nitrate, sulphate and chloride limits of detection are reported in Table SI.2. After some tests with the column-switch method, nitrite was only detected in a set of standards spiked with a high-concentration nitrite solution (Nitrite Nitrogen Standard for IC, Fluka Analytical). Nitrite was not detected in natural samples, so it was not included finally in the study.

The accuracy of the analyses in the IC was tracked by analysis of certified reference material (SPS-NUTR WW2 Batch 115 and MOOS-3 Seawater Certified Reference Material for Nutrients). The SPS CRM was used in the runs for sulphate and chloride analysis since seawater samples were diluted 20 fold.

Table SI. 2: Summary of analytical quality control data: LDL, relative standard deviation (%RSD) and CRM.

	LDL		%RSD		CRM certified values		CRM measured values	
Salinity range of samples	0-4	≥ 4	0-4	≥ 4	0-4	≥ 4	0-4	≥ 4
NH_4^+ (µM)	0.34	0.18	3.1	1.0	na		na	
NO_3^- (µM)	17	42	7.8	8.0	5.00±0.05 mg/L (SPS)	1.43±0.00 mg/L (MOOS-3)	9.1±0.3 mg/L	2.8±2.4 mg/L
SO_4^{2-} (mg/L)	0.9	75	<2		100±1 mg/L (SPS)		104.1±4.5mg/L	
Cl^- (mg/L)	5.4		<2		50.0±0.5 mg/L (SPS)		50.2±0.7 mg/L	

SI.3 Sediment characterisation whats

Energy disperse XRF analysis was carried out on an Olympus Innovex X-5000 spectrometer. The instrument was run in two separate modes using powdered samples. Major elements were determined using a calibration dependant method (using a range of certified natural sediment and shale rock standards, CCRMP). Minor and trace elements were determined using the manufacturers precalibration with Compton normalisation was also applied to reduce problems with matrix effects. Several relevant reference materials (standardized stream sediments, CCRMP) were run for quality check. Analytical uncertainty (measured values versus certified values) was < ±30% for Mg, < ±20% for S, Cl and K and, < ±10% for all other elements.

A printable version of the reference certificate for the CCRMP can be found in the following website: http://www.nrcan.gc.ca/mining-materials/certified-reference-materials/cert ificate-price-list/8023. The

collection and preparation of the four stream sediment reference materials are described in Lynch (1990).

The elemental composition of the estuarine sediments is summarised in Table SI.3. Table SI.4 shows the measured concentrations and certified values of standard reference sediments that were run for quality check.

Table SI.3: Elemental composition of the solid phase (XRF analysis)

	Sediment type	Al (%)	Fe (%)	Mn (ppm)	As (ppm)	Cd (ppm)	Cr (ppm)	Cu (ppm)	V (ppm)	Zn (ppm)	Ni (ppm)
S1	surface	3.08±0.95	2.77±0.76	656±8	23±4	<2	69±4	31±4	61±1	132±3	57±22
	subsurface	3.89±0.15	3.30±0.74	785±8	20±2	<2	82±3	33±3	71±2	149±4	66±1
S2	surface	3.75±0.17	3.05±0.63	681.±20	18±3	<2	76±13	31.5±2.1	62±2	139±4	51±4
	subsurface	3.90±0.16	2.89±0.52	654±5	18±2	<2	77±13	27±2	62±4	129±4	71±4
S3	surface	5.15.0.33±	3.75±0.74	847±6	19±2	<2	107±1	39±2	80±3	161±3	73±8
	subsurface	5.00±0.35	4.10±0.85	969±7	37±4	<2	118±3	31±3	98±3	199±13	84±9
S4	surface	5.61±0.37	4.48±0.99	758±14	30±1	<2	116±3	33±2	93±2	174±4	68±8
	subsurface	5.48±0.44	4.28±0.89	732±11	25±2	<2	113±4	37±11	99±8	167±6	71±5

Table SI.4: Comparison of certified values and measured values of CRM for major and minor elements expressed in % and ppm respectively (averaged values, n=2).

Constituent	STSD-1 CRM	Measured STSD-1	STSD-2 CRM	Measured STSD-2	STSD-3 CRM	Measured STSD-3	STSD-4 CRM	Measured STSD-4
Al (%)	4.7	3.4	8.5	7.1±0.1	5.7	4.8±0.1	6.4	4.7
Fe (%)	4.5	5.4	5.2	6.2±0.1	4.3	5.3±0.2	4.0	4.4
Mn (ppm)	3740	2759±2394	720	1050±8	2630	2936±18	1200	1553±16
As (ppm)	17	33±12	32	44±2	22	31±1	11	12±1
Cd(ppm)	0.8	ND	0.8	ND	1	ND	0.6	ND
Cr(ppm)	28	89±35	50	114±10	34	82±6	30	76±9
Cu(ppm)	36	32±7	43	36±2	38	31±6	66	55±6
V(ppm)	47	96±2	58	98±3	61	169±6	51	166±8
Zn (ppm)	165	209±36	216	238±7	192	208±4	82	86±7
Ni (ppm)	18	ND	47	26±8	25	ND	23	ND

SI.4 ICP analyses information

SI.4.1 Instrument information

The analyses were performed using a Thermo Scientific iCAPQc Inductively Coupled Plasma Mass Spectrometer (ICP-MS).

Aluminium was analysed in standard mode and all other elements in Kinetic Energy Discrimination (KED) mode using helium as a collision gas to remove polyatomic interferences (Table SI.5).

Table SI. 5: Summary of the elements and modes used.

Element	Mass (m/z)	Instrument Mode
Al	27	Std
V	51	KED
Cr	52	KED
Mn	55	KED
Fe	56	KED
Co	59	KED
Cu	63	KED
Zn	64	KED
As	75	KED
Cd	112	KED

SI.4.2 Sample and standard preparation

For the brackish-seawater analysis, samples were diluted 50 fold in 1 % v/v HNO_3 before analysis (0.2 ml sample + 9.8 ml diluent) to reduce matrix effects during the analysis.

Standard additions type calibration was used for the analysis using 1:50 diluted NASS-6 Seawater Certified Reference Material (CRM) as the standard matrix (http://www.nrc-cnrc.gc.ca/eng/solutions/advisory/crm/certificates/nass_6.html). The CRM used for the spikes was SLRS-5 (http://www.speciation.net/Database/Materials/National-Research-Council-of-Canada-NRC--CNRC/SLRS5-River-water-reference-material-for-trace-metals-;i1230).

Calibrations were performed in the range of 1-100 µg L^{-1}.

Blanks of 3 % w/v NaCl solution were prepared.

As internal standardisation, Rhodium at a concentration of 1 ppb was added to all standards and samples for use as an internal standard.

For the freshwaters, everything was run as above except they were analysed with no dilution.

SI.4.3 Analytical figures of merit

Limits of detection were calculated from repeated measurements of 5 individual blank solutions using the equation below.

$$LOD = 3\sigma_{5\ Blanks}$$

Accuracy of the method was estimated by spiking a sample with a known amount of analyte and measuring the analyte recovery. Sample and Sample + Spike were analysed 5 times.

$$\% \ Recovery = 100 \times \frac{M_{Spike} - M_{Sample}}{C_{spike}}$$

Where M_{spike} = Measured concentration of spiked sample.

   M_{Sample} = Measured concentration of sample.

   C_{Spike} = Actual concentration of spike.

Precision of the method was assessed from the repeated measurement of one sample 6 times and reported as the 95% confidence interval of these results.

$$\bar{x} \pm \frac{t_{n-1}s}{\sqrt{n}}$$

Where n = number of measurements

   n-1 = degrees of freedom (5)

   t = t value (2.78 for 4 degrees of freedom)

   s = calculated standard deviation of 5 measurements

   x = calculated mean of 5 measurements

Certified Reference Material

The Certified Reference Material (CRM) used was NASS-6 Certified reference material. This is a seawater reference material.

SI.4.4 Quality Control, LDL and error details for ICP-MS analyses

The NASS-6 was not used as a CRM, it was used as a seawater base to test spike recoveries. The seawater samples were diluted by 1:50 to reduce the salinity and matrix effects of the samples on the

analysis. It would therefore be inappropriate to use unaltered NASS-6 as a CRM as the matrix would be completely different from our samples (~3% NaCl for NASS-6 and ~0.1% NaCl for samples).

The point of the procedure outlined above was to test the accuracy of the analysis using a process called **spike recoveries**. This is a standard analytical technique where a sample is taken and analysed, then a known amount of metal is added and the sample re-analysed. In theory one should get 100% recovery of the added metals. In this case, NASS-6 was used as a convenient source of clean seawater as the sample not as a formal CRM. As the samples were diluted 1:50, it was more appropriate to analyse an unaltered freshwater CRM with them. In this case SLRS-5 was used and excellent recoveries were obtained. The quality control data (Table SI.6) show that, with the exception of the aluminium, the concentrations detected from samples containing 1:50 fold dilution of the seawater certified material (zero standard) spiked with a 5 ppb metal concentration solution, were in fact around 5 ppb. From this control test the recovery was >95%, the LDL was 0.027 ppb and the uncertainty 3.72 %.

See also below for details about the summary of SLRS-5 CRM quality data (Table SI.7), and LDL and errors of the different ICP-MS analyses (Tables SI.8).

For the method validation, the protocol followed was:

1) Two blanks are run to start to start with.

2) The calibration is carried out with standards (from 0-10 ppb) made up with 1:50 seawater reference material.

3) Subsequently, blanks (x6) are run.

4) Then, 1:50 seawater spiked with 0.5ppb (metal concentrations) solution is run (x3 times) followed by a 1:50 seawater spiked with 5ppb (metal concentrations) solution (x3 times).

5) Afterwards, two different samples diluted 1:50 (with no spike) (x5 times) are run; followed by the same samples (x5 times) diluted 1:50 but spiked with 10 ppb standard solution.

6) Next, 1:50 seawater spiked with 0.5ppb (metal concentrations) solution (x3 times) and 1:50 seawater spiked with 5ppb (metal concentrations) solution (x3) are run again.

6) To finish, more blanks are run (x6 times).

Table SI.6: Summary of the quality control data.

	27Al (STD) [ppb]	51V (KED) [ppb]	52Cr (KED) [ppb]	55Mn (KED) [ppb]	56Fe (KED) [ppb]	59Co (KED) [ppb]	63Cu (KED) [ppb]	64Zn (KED) [ppb]	75As (KED) [ppb]	112Cd (STD) [ppb]	60Ni (KED) [ppb]
Zero Std	4,539	0,07	0,049	0,04	0,563	0,003	0,136	0,418	0,061	0,008	0,18
Blank	4,662	0,005	0,03	0,04	1,031	0,003	0,202	0,416	0,009	0,005	0,589
1:50 NASS6 + 5 ppb	10,032	4,63	4,892	4,988	5,029	4,957	5,043	5,694	4,887	5,191	5,086
1:50 NASS6 + 5 ppb	11,569	5,106	5,055	4,971	5,485	4,993	5,063	5,115	5,052	4,914	4,801
1:50 NASS6 + 5 ppb	10,278	4,425	4,469	4,395	4,929	4,526	4,674	4,965	4,793	5,14	5,371
1:50 NASS6 + 5 ppb	10,501	4,632	4,648	4,579	5,135	4,63	4,807	5,103	4,965	5,063	4,898
1:50 NASS6 + 5 ppb	10,797	4,696	4,798	4,758	5,293	4,74	4,889	5,164	4,995	5,085	4,896
1:50 NASS6 + 5 ppb	11,512	5,007	5,003	5,04	5,551	4,941	4,981	5,216	5,179	5,005	4,959
1:50 NASS6 + 5 ppb	11,374	5,183	5,147	5,253	5,707	4,974	5,1	5,796	5,132	4,888	5,088
1:50 NASS6 + 5 ppb	11,909	5,164	5,124	5,328	5,814	4,952	5,021	5,219	5,032	4,758	5,199
Mean	10,997	4,855	4,892	4,914	5,368	4,839	4,947	5,284	5,004	5,006	5,037
Adjusted Mean (Mean-Zero Std)	6,458	4,785	4,843	4,874	4,805	4,836	4,811	4,866	4,943	4,998	4,857
Stdev	0,687	0,293	0,241	0,320	0,323	0,181	0,146	0,297	0,125	0,144	0,187
% Recovery	129,2	95,7	96,9	97,5	96,1	96,7	96,2	97,3	98,9	100,0	97,1

Table SI.7 SLRS-5 quality control data.

Element	Mean of 6 measurements / ng L-1	CRM Spected value / ng L-1	CRM spected error / ng L-1
V	324	317	33
Cr	227	208	23
Co	61	50	*
As	431	413	39
Cd	7.20	6.00	1.4
Ni	531	476	64
	Mean of 6 measurements / µg L-1	CRM spec value / µg L-1	CRM spec error / µg L-1
Al	51.9	49.5	5.00
Mn	4.34	4.33	0.18
Fe	94.2	91.2	5.80
Cu	18.5	17.4	1.30

Table SI.8: Additional information of instrument LDL and uncertainty (% error at 95% confidence interval) for the different analyses carried out.

Analysis and details		Al	V	Cr	Mn	Fe	Co	Ni	Cu	Zn	As	Cd	Pb
Step 1 from Sequential Extractions. 1 M MgCl$_2$ (1% v/v HNO3)	LDL / ppb	23.5	0.363	1.34	2.00	26.50	0.069	0.610	1.10	2.73	0.298	0.137	0.238
	% uncertainty	4.14	2.81	1.93	5.15	4.2	0.92	1.15	1.37	1.40	4.19	0.89	2.32
Step 2 from Sequential Extractions. 1 M NaOAc (1% v/v HNO3)	LDL / ppb	36.4	0.26	0.60	3.65	60.5	0.18	1.58	6.99	2.46	0.58	0.19	0.38
	% uncertainty	4.50	3.64	2.87	5.24	4.20	3.43	4.48	8.33	5.70	4.41	2.21	3.18
Step 3 from Sequential Extractions. 0.04 M NH$_2$OH·HCl in 25% v/v HOAc (1% v/v HNO3).	LDL / ppb	55.07	0.20	0.91	1.19	0.13 ppm	0.17	2.72	2.45	4.83	0.54	0.15	0.23
	% uncertainty	3.74	2.67	2.71	2.48	5.44	3.30	3.76	4.05	3.69	3.11	2.91	2.94
Step 4 from Sequential Extractions. HNO$_3$ + 30% H$_2$O$_2$ + 3.2 M NH$_4$OAc (1% v/v HNO3)	LDL / ppb	122	0.53	0.96	3.44	0.06 ppm	0.25	1.86	2.32	1.73	1.08	0.22	0.25
	% uncertainty	5.55	3.14	2.88	3.43	3.27	2.53	2.95	2.66	2.74	1.09	1.15	1.31
"High salinity water" samples (S3 and S4) from filtered river water and resuspension experiment (x50 fold)	LDL / ppb	7.91	0.14	0.09	0.87	2.16	0.35	0.027	1.35	6.04	0.24	0.03	-
	% uncertainty	0.45	0.48	0.26	0.48	0.80	0.58	3.72	0.68	1.28	1.39	1.00	-
"Low salinity water" samples (S1 and S2) from filtered river water and resuspension experiment	LDL /ppb	0.974	0.003	0.016	0.1	0.651	0.001	0.096	0.249	0.701	0.005	0.014	0.059
	% uncertainty	2.0	1.5	1.0	3.1	1.7	2.7	8.9	1.2	5.9	5.1	3.0	8.4

The following table (Table SI.9) shows the summary of the low detection limits taken into account the dilution of the samples that were analysed. The subsamples for ICP-MS analysis were diluted (x5) with MilliQ water and acidified (1 % v/v HNO_3). The volume of the samples recovered for ICP-MS analysis (after centrifugation and filtration) varied, so the dilution factor was corrected by weight difference. The low detection limit reported below is the more conservative (taken from the more diluted sample analysed).

Table SI.9: Summary of LDL for "low salinity" (0-4) and "high salinity" (≥4) samples

	Al	V	Cr	Mn	Fe	Co	Cu	Zn	As	Cd
Low salinity LDL / ppb	8	0.02	0.1	0.8	5.5	0.01	2.1	6	0.04	0.1
High salinity LDL / ppb	50	1.0	0.6	5.5	13.6	2.2	8.5	38	1.5	0.2

SI.5 Changes in pH and Eh during sediment resuspension
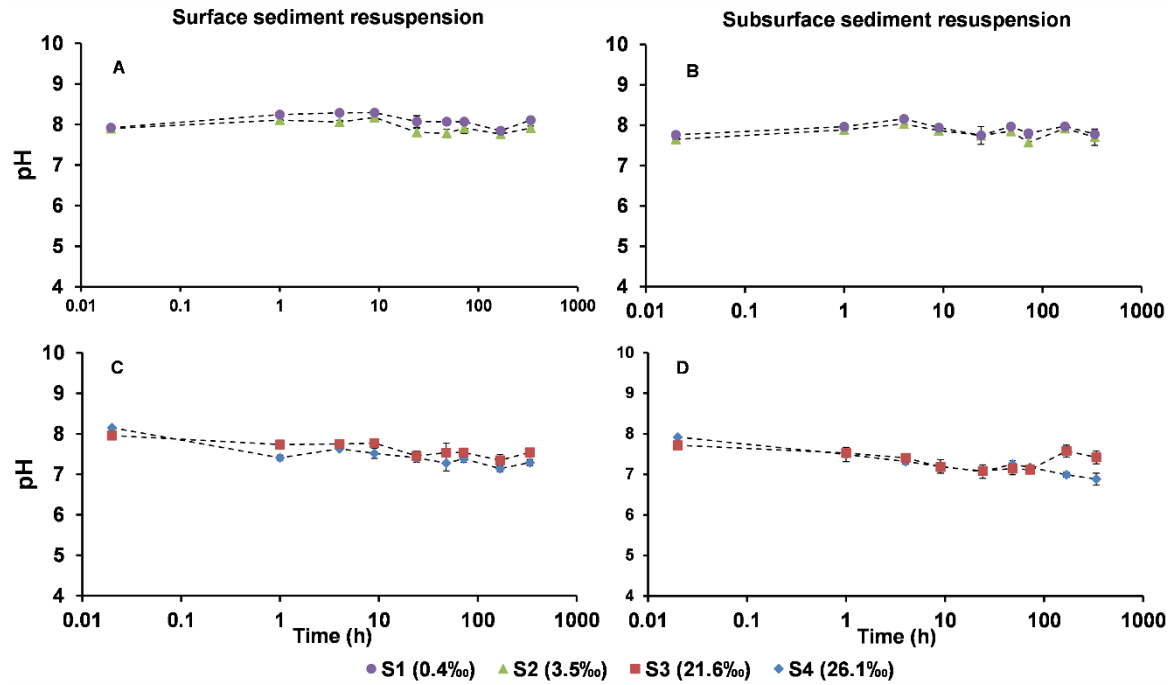

Figure SI.1: pH changes during the resuspension experiments using surface (A) and subsurface (B) sediments from the inner estuary sites; and surface (C) and subsurface (D) sediments from the outer estuary sites.

Figure SI.2: Eh changes during the resuspension experiments using surface (A) and subsurface (B) sediments from the inner estuary sites; and surface (C) and subsurface (D) sediments from the outer estuary sites.
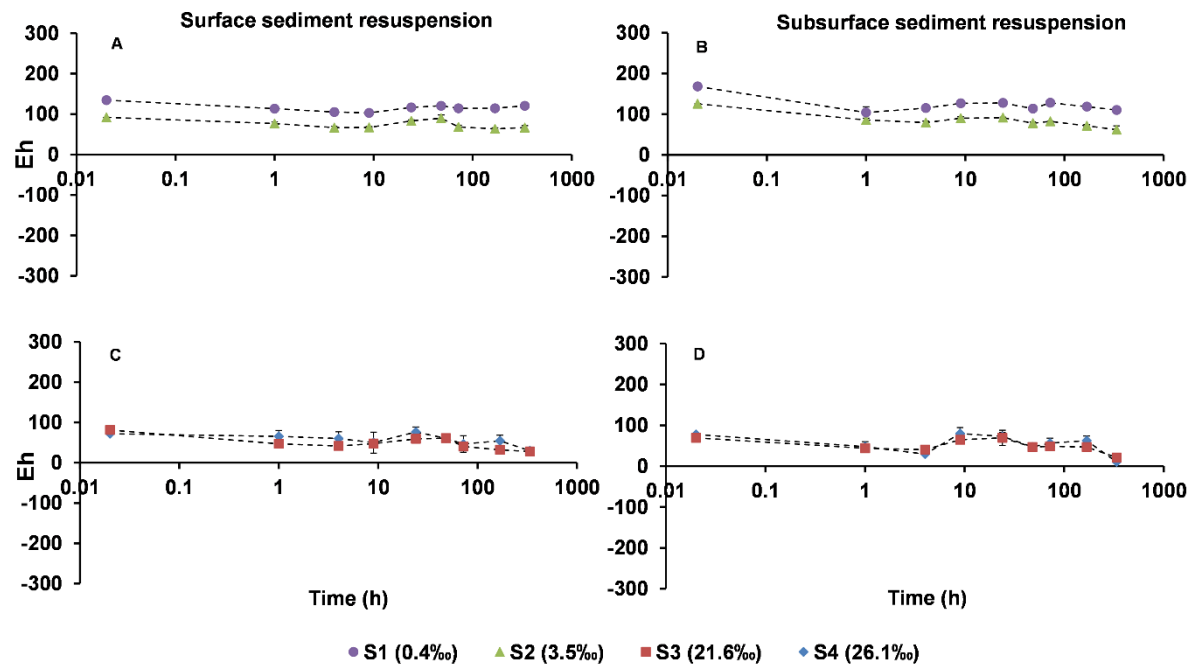
SI.6 Sediment grain size characteristics

Table SI.10 Sediment grain size (as the upper bound diameter of the sample at 50% of cumulative percentage of particles by volume), water content (%) (both from this study); classification, sorting classification and mean silt content (%), from Mortimer et al. (1999).

| Site and sample		Sediment D50 (µm)	Water content (%)	Sediment classification	Sorting classification	Mean silt (%)
Boothferry (S1)	Surface	53	42	not available	not available	not available
	Subsurface	37	35			
Blacktoft (S2)	Surface	47	42	Very fine sand/coarse silt	Poorly sorted	58
	Subsurface	47	39			
Paull (S3)	Surface	16	55	Coarse silt	Poor to moderate	86
	Subsurface	19	41			
Skeffling (S4)	Surface	13	54	Coarse silt	Moderate	90
	Subsurface	16	37			
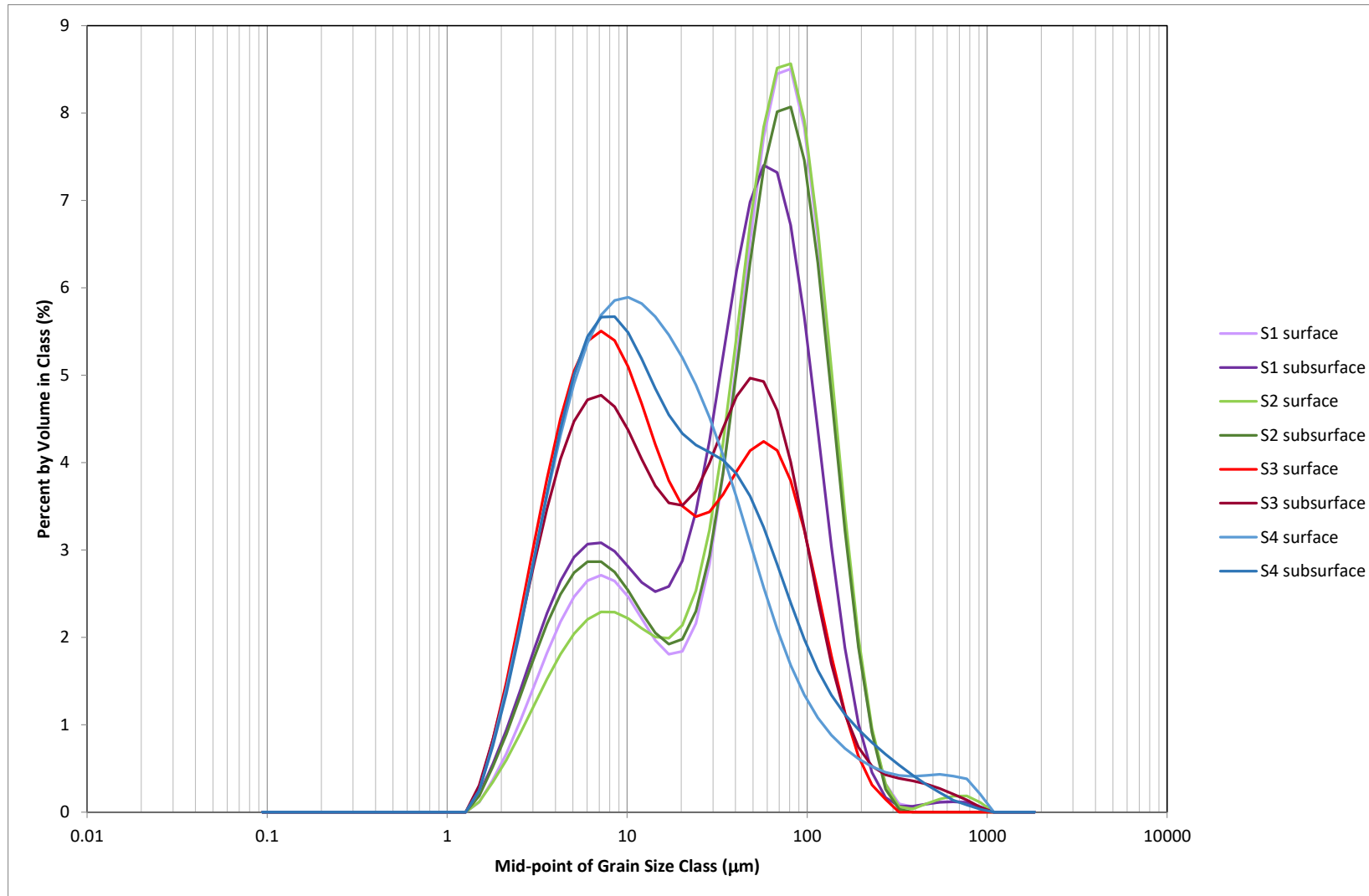
Figure SI.3: Granulometry of the estuarine sediments as a percentage (by volume) of the grain size (µm) classes.

SI.7 Nutrient and metal fluxes

Table SI.11: Calculated nutrient and metal fluxes during a resuspension experiments carried out with surface and subsurface sediments. The fluxes have been calculated for the two time-windows discussed in the manuscript. Green cells indicate positive fluxes (to the water column) and red cells indicate negative fluxes (to the sediment).

Calculated fluxes(mmoles/m2/day)														
	NO_3^-		NH_4^+		Mn^{2+}		SO_4^{2-}		$*Fe^{2+}_{(s)}$		Zn		Cu	
	surface	subsurface	surface	subsurface	surface	subsurface	surface	subsurface	surface	subsurface	surface	subsurface	surface	subsurface
S1														
0-1h	-2.2	12.1	-1.8	7.3	1.1	6.3	110.0	73.3	-79.8	0.0	3.5	0.2	0.0	0.4
1h-48h	0.3	0.3	0.2	-0.1	0.0	-0.1	1.5	1.5	-17.7	-31.0	-0.1	0.0	0.0	0.1
S2														
0-1h	49.9	69.4	-2.2	1.8	0.4	4.2	36.7	183.3	21.3	-637.5	2.4	-0.3	-0.1	0.2
1h-48h	0.0	0.0	0.2	0.0	0.0	-0.1	10.7	3.1	-7.8	-12.8	-0.1	0.0	0.0	0.0
S3														
0-1h	-9.2	-15.0	-2.9	45.8	9.9	2.5	36.7	586.7	70.1	416.5	190.3	127.0	71.7	46.5
1h-48h	0.7	0.1	0.1	-0.8	-0.2	0.0	18.3	18.3	-14.6	-101.4	-4.9	-16.0	-1.6	-0.9
S4														
0-1h	1.8	-1.5	-4.8	18.7	14.0	-0.7	-623.4	-770.1	-1341.0	-271.4	586.6	344.1	220.3	120.8
1h-48h	1.2	0.1	0.1	-0.5	0.0	-0.2	26.7	56.5	-5.3	-126.3	-3.8	-9.0	-4.9	-2.9

* In the case of $Fe^{2+}_{(s)}$ is not a flux since it represents iron transformation (i.e. redox transformations) within the sediments

The calculated fluxes (for the short timescale, 0-1 hrs of resuspension) can be used to give crude estimates of the role played by the intertidal mudflats in the nutrient budget of the estuary when we upscale those fluxes to the whole estuary (Table SI.12). The intertidal area can be subdivided into outer estuary (Humber Bridge to Spurn, which would include the middle estuary in Mortimer et al., 1998) middle estuary and inner estuary (Humber Bridge to Trent Falls), giving areas of 99 and 16 km2 respectively.

For the nitrogen species, we have calculated the amounts of nitrate and ammonia that are removed or released to the water column during the resuspension. Resuspension events were not considered in other works that have calculated nitrogen fluxes on the Humber estuary (Sanders et al., 1997, Mortimer et al., 1998; Barnes & Owens 1998). Here we compare our estimations with those reported by Mortimer et al. (1998).

The resuspension of surface sediments would be a source of nitrate in the inner estuary but nitrate is mainly consumed in the outer estuary (sink) (23.8 mmoles/m^2/day average in the inner estuary and −12.1 mmoles/m^2/day average in the outer estuary). This contrasted with Mortimer et al., (1998) fluxes of nitrate, which were negative also in the inner estuary (−13.4 mmoles/m^2/day). Nitrate fluxes increased when we consider the resuspension of subsurface sediments. For the ammonium, our estimations show a completely different scenario if surface or subsurface sediment are being resuspended. For surface sediments, both, inner and outer estuary will be a sink for ammonium (−2 and −3.9 mmoles/m^2/day respectively). However subsurface sediment resuspension will be a major source of ammonium (4.6 and 32.3 mmoles/m^2/day), which agrees with the conclusions in Mortimer et al., (1998). Overall, intertidal sediments seem to be a sink for DIN when surface sediment resuspension is considered, but a major source when subsurface sediments are mobilised. So if major resuspension events are more frequent, general considerations about nitrogen and other important elements budgets should be taken into account.

Table SI.12: Estimated nitrate and ammonium fluxes for the Humber Estuary.

Surface sediment resuspension					
	Average NO$_3^-$ flux (mmoles/m^2/day)	Average NH$_4^+$ flux mmoles/m^2/day	NO$_3^-$ kmoles/day	NH$_4^+$ kmoles/day	Overall DIN kmoles/day
inner estuary	23.8	−2.0	358	−30	
outer estuary	−12.1	−3.9	−1198	−381	
whole estuary			−840	−411	**−1252**
Subsurface sediment resuspension					
	Average NO$_3^-$ flux (mmoles/m^2/day)	Average NH$_4^+$ flux mmoles/m^2/day	NO$_3^-$ kmoles/day	NH$_4^+$ kmoles/day	
inner estuary	40.8	4.6	611	69	
outer estuary	−3.9	32.3	−381	3195	
whole estuary			230	3263	**3494**

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