



Resuspension studies in cylindrical microcosms: Effects of stirring velocity on the dynamics of redox sensitive elements in a coastal sediment

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Abstract. Effects of resuspension on the release of dissolved, redox sensitive elements (Fe, Mn) was studied in cylindrical microcosms. Effects from changing water stirring velocity in sediment pools were evaluated through measurements of pore water profiles of dissolved Mn, Fe and redox potential. Mn was a good natural marker to follow such effects. At current velocities below the threshold velocity for resuspension (37 cm s^{-1}), Mn release rates to overlying water were 100 times higher compared to steady-state values. Pulse increases in Mn concentration were the result of convective currents inside flow chambers. These results were strongly supported by measurements of Eh profiles in the sediment pore water. Furthermore, impacts from increasing stirring velocity were found down to 1.9 cm depth below the resuspended layer of sediment.

Introduction

In modern sediments, the redox potential decreases with depth below the sediment surface as a result of bacterial degradation of organic matter. At a certain depth, which depends on bottom-water composition (O_2 , NO_3^-), carbon flux, the composition and the accumulation rate of the sediment deposit, the redox potential falls below values at which oxidized forms of Mn and Fe are stable, and reductive dissolution of Mn and Fe-oxides takes place. The dissolution increases the pore water concentrations of dissolved Mn, Fe (and associated ions), resulting in concentration gradients which cause the ions to migrate through the pore water towards the sediment surface. The flux of ions within

the pore water and across the sediment-water interface can be calculated from the magnitude of the concentration gradients. However, in many instances the production of soluble ions takes place so close to the sediment surface, that it is difficult to measure the gradients with a resolution sufficient for the calculation of fluxes (Thamdrup et al. 1994; Davison et al. 1997; Brendel & Luther 1995). It is then preferable to study reactions at the sediment-water interface by measuring the fluxes directly, using either flux chambers which are placed over the sediment surface, or by laboratory incubations of sediment cores.

Aller (1980) observed that the fluxes of dissolved Mn and Fe from cores incubated in the laboratory were variable. Sometimes longer incubation times gave higher flux rates than shorter incubation times. He suggested that lower levels of dissolved O₂ were beginning to cause changes in pore water profiles or reaction distributions in the sediment.

Sediment resuspension is an important process in coastal areas, for example, it affects boundary layer structure (Gust & Walger 1976), re-distribution of sediment (Amos & Mosher 1985), enrichment of the water column by dissolved and particulate nutrients (Wainright 1990). Work on physical aspects of resuspension has been recently published (see *Hydrobiologia*, vol. 284 from 1994), and is abundant on metal release, however impact of resuspension in other sediment processes is still poorly understood (Vidal 1994; Laima et al. 1994; Sloth et al. 1996; Christiansen et al. 1997). Most available data on sediment-water fluxes concern exchanges through an undisturbed top sediment layer, and are largely obtained at standard levels of water motion, because of difficulties in reproducing environmental conditions. In stirred chambers, attention has been given to solute release mechanisms (Huettel & Gust 1992) and the redistribution of O₂ following changes in the flow velocity of the overlying water (Booij et al. 1991). These observations indicate that O₂ has an important effect on fluxes of redox-sensitive metals and associated species. However, the response of the sediment column to these flow changes has not been investigated in detail, and Eh potentials are rarely reported (Smayda 1990; Moore & Reddy 1994).

In this study we describe *in vitro* flow chamber experiments using stirring conditions where both O₂ and temperature are maintained close to the *in situ* values. It is investigated if resuspension causes changes in the sediment below the resuspended layers, measuring pore water concentrations of redox sensitive elements Fe and Mn, as indicators for these changes. Complementary redox potential measurements are used to indicate changes at the redox-boundary during the experiments. Where appropriate, emphasis is given to differentiate between natural variation effects and true resuspension effects.

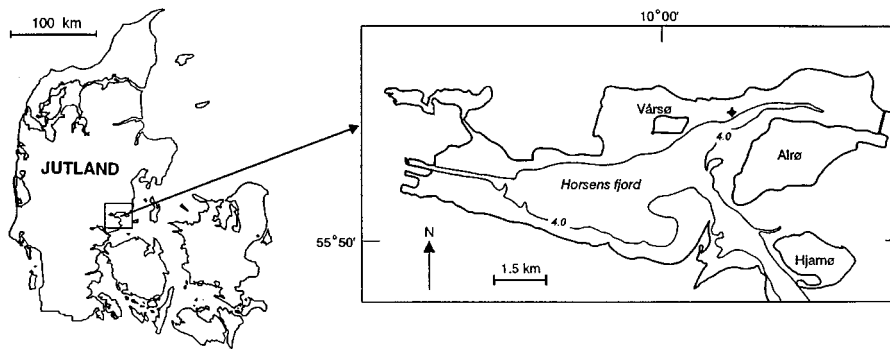


Figure 1. Horsens Fjord, Denmark. The cross indicates the sampling station.

Sampling and methods

Core sediment sampling

Sediments from Horsens Fjord, Denmark (55.52.437 N; 10.02.677 E) were sampled in May 1996 at 3 m water depth, using a 'Haps' bottom corer (Kanneworff & Nicolaisen 1973). The sampling position was placed in a sheltered bay in the northern part of Horsens Fjord (Figure 1). Resuspension at the site is generally governed by surface waves as the dominating wind direction in the area is southwest, giving a maximum fetch (Lund-Hansen & Eriksen 1998). Sediment was sandy with a mean grain diameter of 0.009 cm. In situ O_2 concentration in bottom water was 13.1 mg l^{-1} (107% of air saturation) bottom salinity was 24.5 psu and temperature was 5°C . Sediment samples were taken with Plexiglass tubes (9.5 cm i.d., 50 cm long) and with smaller coring tubes (5.4 cm i.d., 20 cm long). The sediment surface was covered by a 0.2–0.3 cm thick benthic algae mat also including planktonic forms. Sediments with visible macrofauna were discarded. Sediments and water were transferred to the laboratory within a few hours after sampling and were kept in a temperature controlled room (4°C). Water content (105°C , 24 h), porosity and organic matter content (550°C , 2.2 h) were determined in duplicate (Table 1). All experiments described below were carried out in the cold room at darkness.

Resuspension assays

The following experimental set-up was designed to study fluxes in undisturbed sediments and under different stirring velocities at circular flow motion (Figure 2). Sediment cores with water collected at the station were prepared in the following way: Sediment height was 15 cm and the water phase was 10 cm. A four bladed propeller was placed 6 cm above the sediment surface

Table 1. Sediment characteristics (0–10 cm) for the sampling site (55.52.437 N; 10.02.677 E). All measurements were done in duplicate. Horsens Fjord, Denmark, May 1996.

Depth (cm)	H ₂ O (ml g ⁻¹ wet wt)	Density (g cm ⁻³)	Org. matter (% dry wt)	Porosity (vol vol ⁻¹)
0–1	0.65 ± 0.0	1.29 ± 0.0	7.23 ± 0.8	0.843
1–2	0.49 ± 0.0	1.45 ± 0.1	4.05 ± 0.4	0.702
2–3	0.47 ± 0.0	1.46 ± 0.1	4.27 ± 0.4	0.692
3–4	0.45 ± 0.0	1.47 ± 0.1	3.85 ± 0.0	0.657
4–5	0.45 ± 0.0	1.47 ± 0.1	3.94 ± 0.4	0.655
5–6	0.48 ± 0.0	1.50 ± 0.1	4.66 ± 0.0	0.728
6–7	0.48 ± 0.0	1.37 ± 0.1	4.63 ± 0.7	0.660
7–8	0.57 ± 0.0	1.25 ± 0.1	6.77 ± 0.7	0.712
8–9	0.53 ± 0.1	1.20 ± 0.1	5.71 ± 1.9	0.635
9–10	0.65 ± 0.0	1.30 ± 0.2	8.36 ± 0.7	0.839

and the rotation speed was controlled by an electric motor (Lund-Hansen et al. 1995). Bottom water collected at the sampling site was pumped continuously over the sediment and pumped out to obtain a residence time of 12 hours (flowrate 50 ml h⁻¹). An O₂ microelectrode was placed in a cell at the chamber outlet and the electrode output signal was read through a computer. Readings were done in clear water, thus avoiding possible changes in electrode performance when measuring O₂ in the different chambers. No difference in signal was observed in the water inflowing to the electrode cell and in the chamber water. Sometimes, O₂ was also measured manually (Winkler method) in fresh outflow samples to check for electrode performance. During the first hours of incubation, outlet water samples were collected intensively until reaching a steady-state in dissolved Fe and Mn concentrations. In vitro resuspension was simulated by step increases of blade rotation (0.5 volt step during 30 minutes, range 0.5–3 volt), corresponding to stirring velocities of 2.3–13.8 cm s⁻¹, which reached their maximum about 3.0 cm distance from core edge measured 0.1 cm above sediment (Figure 3). Motor rotation was then regulated to obtain symmetrical, step decreasing velocities. It was found that the above velocity range did not cause resuspension of sediment. In assay 2, higher pulse increases were applied (1.0 volt during 30 min, range 1–8 volts), or 4.6–36.8 cm s⁻¹ of maximum stirring velocity. Motor rotation was then regulated to 1.1 cm s⁻¹, which continued for some days. Control flow chamber (Fc) was incubated with gentle stirring of overlying water (1.1 cm s⁻¹), to avoid anoxic stratification, and the O₂ concentration in overlying

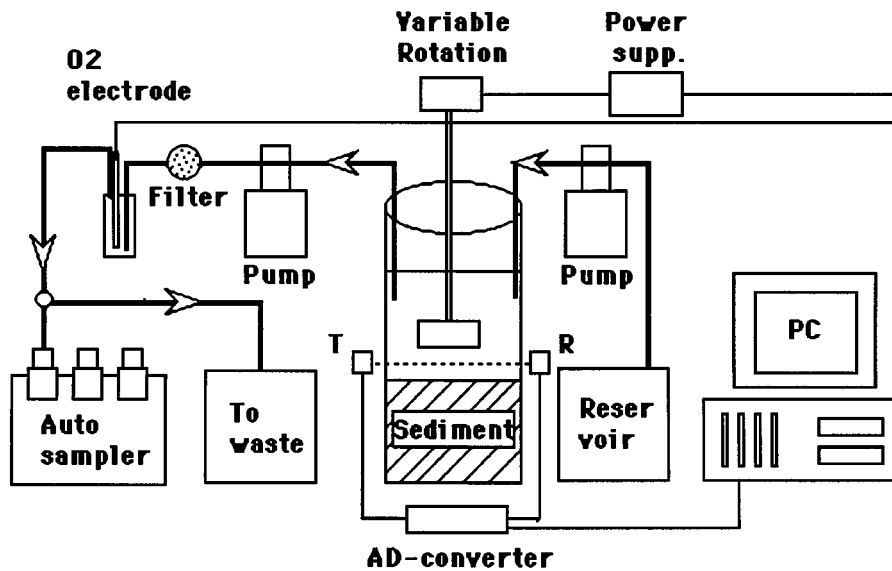


Figure 2. Experimental set-up in the laboratory assays of resuspension. Using this set-up it was possible for a range of stirring velocities in water, to collect water samples for measurement of concentrations of solutes and to measure electrode based parameters in sediment overlying water.

water was periodically checked. Sediment cores ($n = 2$) with no replacement or stirring of overlying water, were incubated in parallel with flow chambers.

Sampling of chamber water and sediment pore water

Flow water from resuspension chambers was sampled using an automatic Model 2700 Autosampler (ISCO inc., USA), otherwise water was manually sampled from the outlet into 50 or 100 ml flasks. Flux water samples were filtered through $0.45 \mu\text{m}$ cellulose acetate filters, acidified with ultrapure HCl to pH 2–3 and stored at 4°C until analysis. At selected time intervals, flow chambers and control cores were transferred into a N_2 -filled glove box, and 1.0 cm sediment slices (0–10 cm) were scooped off. Sediment slices in air tight containers were transferred to a Sorvall RC-5B refrigerated super-speed centrifuge for pore water removal ($5340 \times g$, 30 min). Pore water supernatants were filtered in the glove box, acidified with HCl to minimize oxidation of Fe(II), and stored at 4°C until analysis.

Analysis of water samples

Mn dissolved in pore water was measured using an air-acetylene AAS, whereas Mn concentrations in flux samples were measured by Graphite

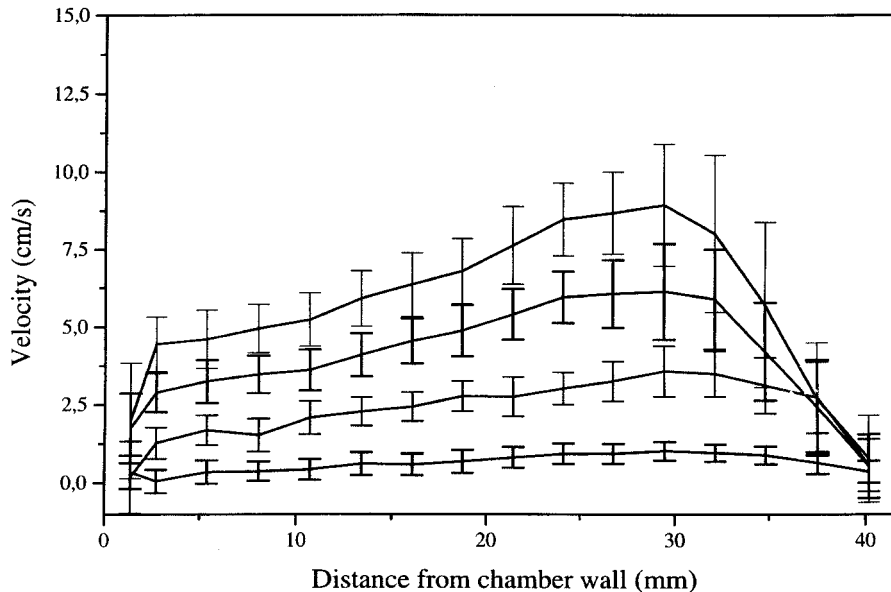


Figure 3. Distribution of water velocity inside resuspension flow chambers for 2.3, 4.6, 6.9 and 9.2 cm s^{-1} of near bottom velocity. Each velocity value is the average of 500 data points as measured 1 mm above bottom with a laser Doppler anemometer.

Furnace AAS according to Slavin et al. (1982), using $\text{Mg}(\text{NO}_3)_2$ as matrix modifier (Table 2). Concentrations were determined with a five-point calibration curve. Calibration standards were prepared from commercial standard of Mn(II) in 0.5 M HNO_3 , diluted with artificial seawater to get the same salinity as the water samples (25 psu). Each sample was run in duplicate or until a 3% standard coefficient of variation was obtained. Dissolved Fe in pore water was measured spectrophotometrically (Collins et al. 1959). Primary standard for Fe was $(\text{NH}_4)_2\text{Fe}(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$, the detection limit was 0.002 mM and the relative standard deviation on replicates was below 1% ($n = 3$). The dissolved Fe pool is interpreted as Fe(II), because the solubility of Fe(III) is low ($< 10^{-8}\text{M}$) at natural conditions ($6 < \text{pH} < 8$). Iron which had been oxidized to Fe(III) during storage, was reduced to Fe(II) with HONH_3Cl . Concentration data in outlet samples were corrected for possible adsorption effects onto container walls, tubings and overlying water, as measured in a control system without sediment. All materials used had been previously stored during 12 hours in a 0.02 M H_2SO_4 -bath and all chemicals were of analytical grade.

Release rate of solutes at steady-state were calculated from the mass balance $VdP/dt = JA - QP$, where dP/dt is the change of solute concentration with time, J is the solute flux to overlying water, V is the volume of overlying

Table 2. Atomic Spectroscopy standard conditions used for analysis of dissolved Mn (Perkin Elmer, Germany). Diluent used to obtain data was 0.2% HNO₃. Detection limits were determined using Mn standards in dilute aqueous solution, and were based on a 98% confidence level (3 standard deviations).

	Flamme- AAS Model 5100 PC	GF- AAS Model 4100 ZL
Wavelength, nm	279.5	279.5
Matrix modifier		0.005 mg Pd + 0.003 mg Mg(NO ₃) ₂
Pretreatment temp., °C		1300
Atomization temp., °C		1900
Atomization time, s		3
Detection limits, mg/L	0.0015	0.00009
Sensitivity check, mg/L	2.5–25.0	0.01

water, Q is the water flow rate and A is the area of sediment column. The approximation of Boers et al. (1984) was used for short sampling intervals.

Eh measurements

Redox potential was measured manually using a home made platinum electrode, consisting of a Pt wire (0.05 cm diameter) mounted in a Pasteur pipette (0.1 cm external diameter, 10 cm tip length). The pipette glass was melted around the Pt wire, leaving 0.2 cm of Pt exposed. Potentials were measured using a standard pH-meter (PHM 62 Radiometer), and a saturated calomel electrode as reference (K401 Radiometer). All results are given relative to the calomel electrode. Eh relative to the hydrogen electrode can be found by adding 241.5 mV. A redox profile was measured as follows: The Pt-electrode was cleaned in conc. HNO₃ and mounted on a manipulator, which enabled us to control the position of the electrode tip within 0.1 mm resolution. The Pt- and the reference electrode were plunged in the sediment overlying water, until a stable reading was reached (10–20 min). The Pt-electrode was then carefully pressed in 0.1 cm steps down into the sediment, whereas the reference electrode was left in the overlying water. Rather than searching for a stable reading and in order to avoid or minimize contamination, the Pt-electrode was left exactly 10 seconds at each depth before reading the Eh value. The Pt-electrode only makes a hole with 1 mm diameter (area 0.008 cm²), so it will not significantly affect the concentration of ions measured in the pore water extracted from the sediment core (area 71 cm²). Natural variation effects were investigated by measuring Eh randomly in dif-

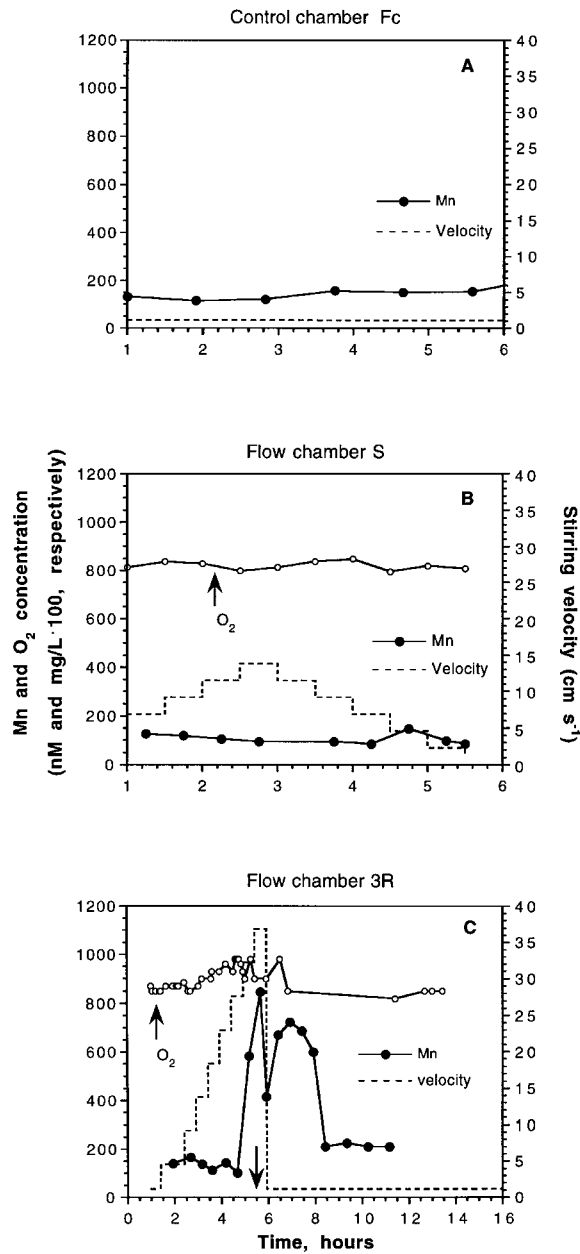


Figure 4. Flow chamber experiments. Effects of stirring velocity of overlying water on Mn fluxes in a coastal sediment. Flow chamber Fc (A) was run with very low velocity (1.1 cm s^{-1}). Flow chambers 2R and 3R were exposed to velocities in the range of, respectively, $2.3\text{--}13.8$ down to 1.15 cm s^{-1} (B) and $4.6\text{--}36.8$ down to 1.1 cm s^{-1} (C). The upper sediment stratum (~ 0.5 to 1 cm) was resuspended at 36.8 cm s^{-1} . Oxygen concentrations (mg l^{-1}) in the water phase during resuspension are shown. Arrow indicates the point of resuspension causing disruption of the uppermost sediment stratum.

Table 3. In vitro resuspension experiments. Measured Mn flux rates at different stirring velocities of overlying water and comparison with a control sediment. Horsens Fjord, Denmark.

Flow chamber	Condition	Flow velocity cm s ⁻¹	Flux of dissolved Mn nmol m ⁻² h ⁻¹
3R	Before resuspension	1	$1.8 \cdot 10^{-3}$
	Increasing velocity	5–24	$-5.9 \cdot 10^{-3}$
	Increasing velocity	28–37	0.19
	Resuspension	37	(fluctuated)
	After resuspension	1	$1.9 \cdot 10^{-3}$
Fc	Control	1	$1.9 \cdot 10^{-3}$

ferent control cores, whereas resuspension effects were investigated through time-series measurements of Eh on fixed positions in the same core.

The influence of resuspension on Eh profiles was assessed through readings at two positions situated only 0.5 cm apart. At both positions, Eh profiles were measured before resuspension, “during” resuspension at 36.8 cm s⁻¹ and after the end of resuspension. The term “during” here refers to a reading done a few minutes after a short stop of motor rotation, as redox profiles could not be read while the stirring device was rotating. Readings continued for two days following the stop of motor rotation.

Results and discussion

Resuspension, solute fluxes and pore water profiles

No major changes in the concentrations of O₂ and Mn were observed for applied low and moderate stirring velocities (Figure 4A–4B). There was no define pattern for dissolved Fe, fluctuations observed were in the same magnitude, independently of core condition (not shown). At stirring velocities above 20 cm s⁻¹, there was a smooth increase of O₂ concentration in chamber water up to the point of resuspension (Figure 4C). Chamber experiments were run at darkness and water residence time was constant with time. A possible explanation for this increase of O₂ concentration, was a temporarily fall in the sediment oxygen demand at these water stirring velocities. It was necessary to increase the velocity up to 36.8 cm s⁻¹ to observe a sudden disruption and resuspension of the uppermost few mm of sediment. At the time of resuspension, there was a fall in O₂ concentration, before reaching

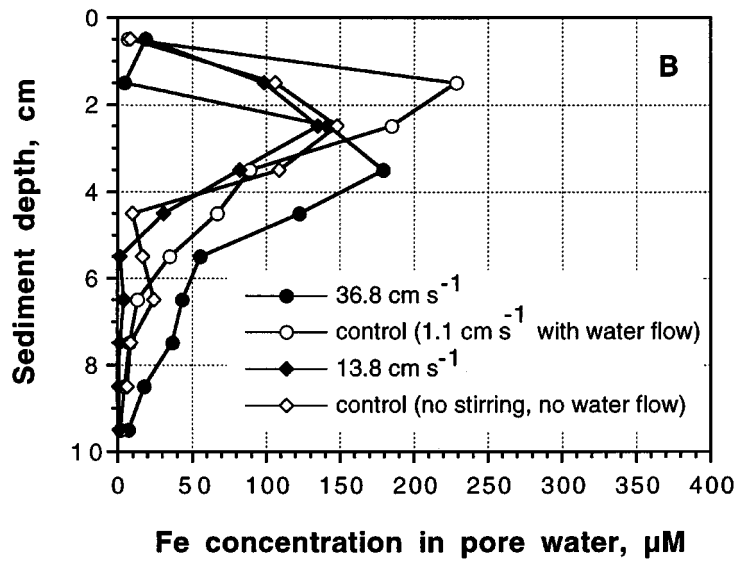
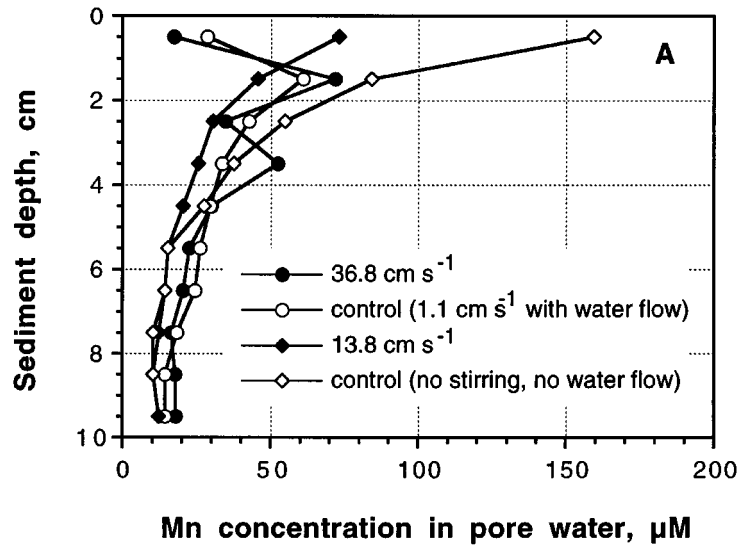


Figure 5. Depth sediment pore water profiles for dissolved Mn (A) and for dissolved Fe (B) obtained after resuspension assays. Profiles are shown for flow chambers 3R (36.8 cm s^{-1}), 2R (13.8 cm s^{-1}), Fc (1.1 cm s^{-1} , with water flow) and core 2C (no stirring or water flow).

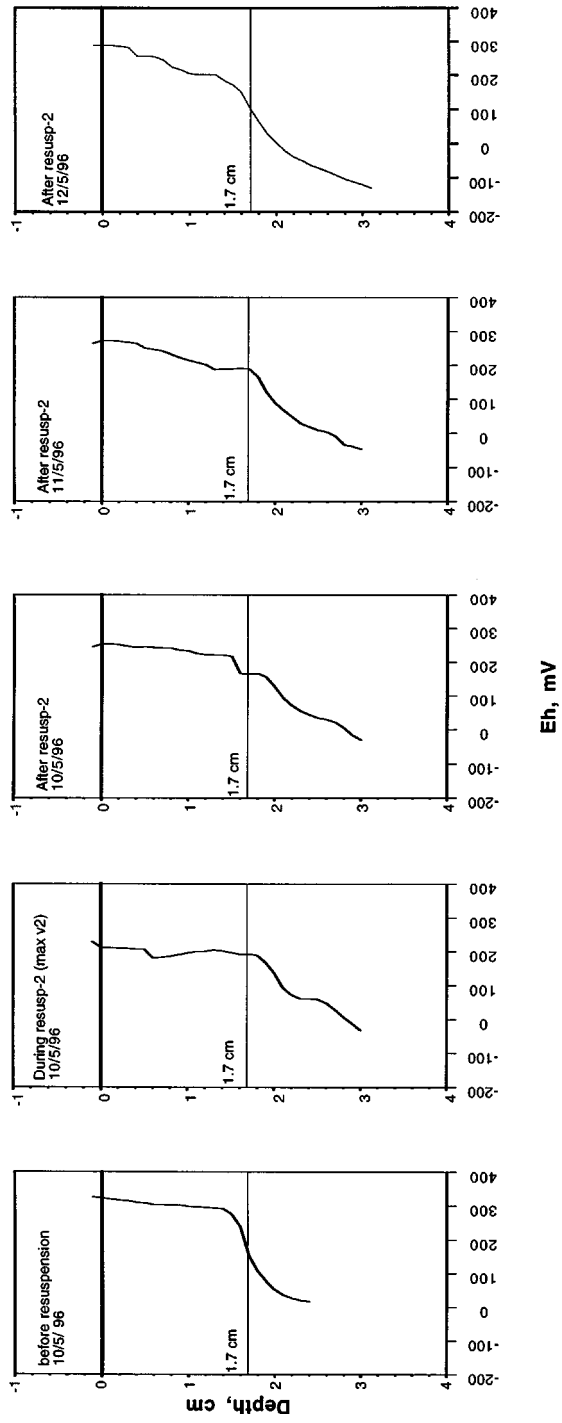
a steady-state. A likely explanation for the fall in O_2 concentration was an increase of sediment activity from the direct exposure of reduced sediment to oxygenated overlying water.

Mn concentrations varied with the applied stirring velocity, resulting in alternating Mn flux sign from effluxes at low stirring velocities, via influxes at intermediate velocities and effluxes again above 28 cm s^{-1} (Figure 4 and Table 3). At velocities below 28 cm s^{-1} , there was a simultaneous increase of O_2 concentration and decrease of Mn concentration. This suggests the occurrence of Mn oxidation, decreasing the concentration of dissolved Mn in the water (Table 3). At velocities above 28 cm s^{-1} , concentrations increased abruptly until a maximum was reached at the point of resuspension. At these velocities, normal diffusional processes in and above the sediment-water interface, were certainly superimposed by physical forces increasing pore water exchange which in turn resulted in high Mn release rates to overlying water. Different hydraulic pressure gradients inside the chamber may cause radial flows to develop, which, in case of permeable sands, may go through the upper mm's of the sediment (Huettel & Gust 1992). In our sediments, these effects were observed down to 1.9 cm (see below). Dissolved Mn is an excellent natural marker for such flows, as it is often present in high concentrations in the pore water of the uppermost sediment strata. Furthermore, the oxidation of dissolved Mn^{2+} is slow as compared to dissolved Fe^{2+} (Wilson 1980; Roekens & van Grieken 1983), giving time to measure the high concentration level before the Mn^{2+} is oxidized and precipitates. After resuspension, velocity was set at 1.1 cm s^{-1} and the Mn concentration fluctuated for some hours, before reaching a steady-state. This is in agreement with others (Santschi et al. 1990; Sundby et al. 1986), who observed similar fluctuations in stirring interrupted systems with regulated O_2 concentrations.

Pore water profiles

Figure 5 shows depth sediment profiles of pore water Mn and Fe after the end of both resuspension assays, and comparison with two control cores with different conditions of overlying water flow. The Mn concentration in the 0–1 cm sediment stratum decreased significantly in all flow chambers, as compared to the batch, O_2 limited core (Figure 5A). This again suggests the occurrence of oxidation/precipitation of Mn in upper microlayers of sediment. With respect to Fe, resuspension apparently did not cause changes in the top 0–1 cm, but below this layer (Figure 5B). These results are supported by the Eh measurements (see below).

Flow chamber 3R, hole 1



Flow chamber 3R, hole 2

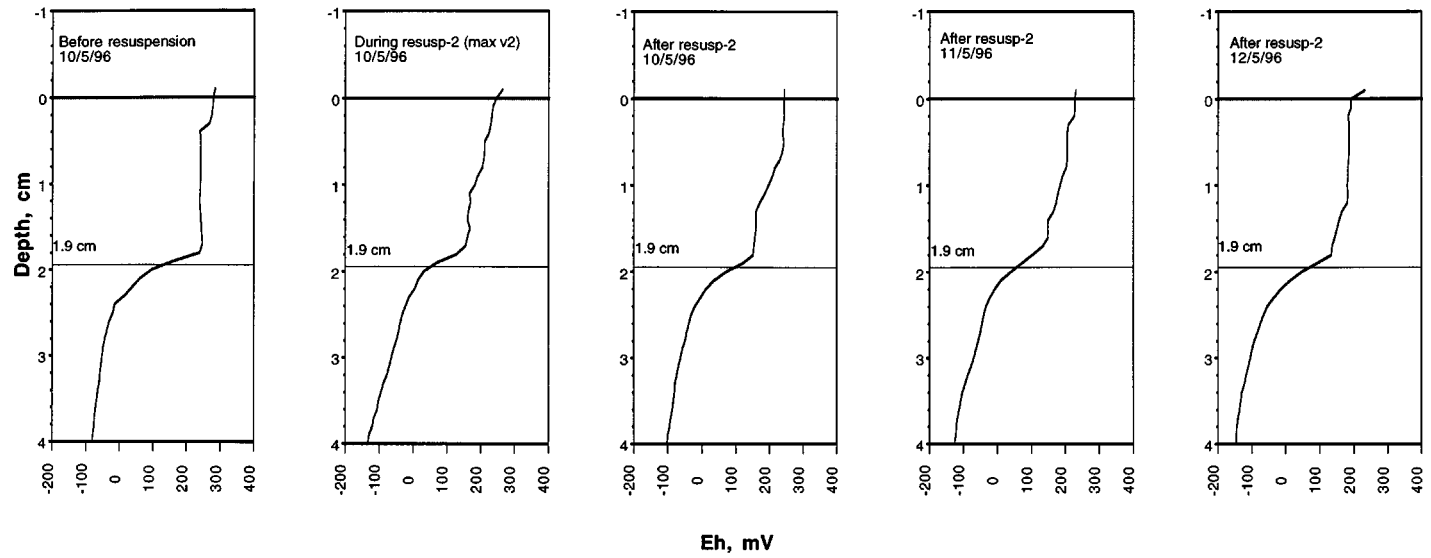


Figure 6. Flow chamber 3R. Time serie depth Eh profile measured before resuspension, “during” resuspension at 36.8 cm s^{-1} and three times after the stop of resuspension. Measurements were done in two positions situated only 0.5 cm apart. The line representing the redox-jump is shown on graph for both situations.

Eh profiles and resuspension effects

The heterogeneous character of this sediment resulted in high variability between single Eh profiles in different control cores and between Eh profiles in different positions in the same sediment core (not shown). Variability of Eh profiles did not decrease by choosing a shorter distance between measuring positions in sediment. To avoid problems related to the inhomogeneity of sediment and in order to investigate resuspension effects, we chose to make multiple Eh readings in the same position. We investigated the usefulness of this approach in a control exchange experiment, in which a redox-profile was measured randomly in a non disturbed core. The small hole left by the Pt-electrode was then manually filled-up with oxic surface water and the redox profile was again measured, resulting in higher Eh values. Repetitive Eh measurements showed that the old redox-profile was re-established within 20 hours (not shown). Thus, to investigate changes due to disturbance, redox potential were measured frequently.

Figure 6 shows results from time series measurements of two depth redox profiles in core 3R before, during and after the induced resuspension. The profiles were all measured exactly at the same two positions in the sediment core and were not influenced by the inhomogeneity of the sediment. It is seen, that before resuspension there was one large jump in the redox potential at 1.7 cm depth (Figure 6). The redox jump most likely reflects changes in the Fe(II)/Fe(III) and Mn(II)/Mn(III, IV) equilibria in the pore water (Whitfield 1972). At 36.8 cm s^{-1} the position of the redox-jump had moved from 1.7 cm to 1.9 cm, and the redox profile was more flat. During the next two days the position of the jump moved slowly back towards 1.7 cm. The sharp redox profile measured before resuspension was not fully re-established two days after resuspension. This breakdown of the redox profile is likely due to porewater convection requiring some time to equilibrate with the surrounding sediment.

Porewater exchange induced by the resuspension was not evenly distributed across the sediment. In similar measurements of redox profiles in another position (only 0.5 cm apart) in core 3R no changes in depth of redox jump were observed (Figure 6). So, visualization of true effects of resuspension on Eh depended on electrode position in sediment. The distribution of pore water exchange is probably influenced by local sediment structure (Webster et al. 1996), by animal burrows and perhaps even by the holes left by the Pt-electrode (0.1 cm diameter).

Conclusion

Our laboratory experiments on resuspension have shown the following points:

- (1) Dissolved Mn can be used as a good natural marker to follow the effects of water stirring in sediments and overlying water.
- (2) The increase in stirring velocity of overlying water below the point of resuspension, resulted in dramatic changes in the concentration of dissolved Fe and Mn below the resuspended layers of sediment, and the movement of the redox boundary from 1.7 cm to 1.9 cm in depth.
- (3) The observed increase in Mn concentration in the overlying water below the point of resuspension was the result of pore water convection effects, which changed the composition of sediment pore water and the release rate of Mn to sediment overlying water.

In situ pore pressure measurements should be encouraged, to give direct evidence of advection of pore water fluids in the sediment (Fang et al. 1993; Glud et al. 1996) and their influence on concentration gradients of chemical species in aquatic microenvironments (Davison et al. 1997).

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