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Effect of acid mine drainage on the chemical composition and fall velocity of fine organic particles

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Abstract

Allochthonous fine organic particles (0.45 μm–1 mm, FPOM) are a significant natural source for biotic production of alkalinity in acidic, post-mining waters. Hence, the depositional characteristics and the nature of FPOM are of interest to remediation. Our objectives were firstly, to determine interactions between allochthonous FPOM and water affected by mine drainage and secondly, to verify the consequence on particle fall velocity, a major factor in particle distribution in streams and lakes. In laboratory experiments we studied interactions between particles (shredded leaves) and two types of post-mining water (very acidic, pH 3.7; extremely acidic, pH 2.9). Particle fall velocity was assessed by gravity sedimentation analysis. Suspended particles released minerals (1 mass%) and organic compounds (10 mass%) within 25 h. Fe^{3+} was quickly reduced to Fe^{2+} in aerobic conditions by organic reductants. Most of the iron attached to the particles (88% in very acidic and 71% in extremely acidic water) when the particle concentration was high. The median particle fall velocity was in acidic, iron-rich water up to 3 times higher than in distilled water. We assume particle aggregation in the highly mineralised water to be the reason for the increase in particle fall velocity.

Keywords: Acid mine drainage; FPOM; Seston; Fall velocity; Settling velocity; Aggregation

1. Introduction

Surface waters in post-mining landscapes, such as in Lower Lusatia, Brandenburg, Germany, suffer from severe acidity. Oxidation of pyrite and marcasite in mining tips cause high concentrations of dissolved iron (2–126 mg l^{-1}) and sulphate (80–1400 mg l^{-1}) and low pH (2.5–4.7) in lakes and streams of the region (Mutz et al., 2002). The extreme acidity is a major target of rehabilitation measures. It can be partially neutralised by microbial reduction of Fe^{3+} and sulphate and subsequent storage of reduced sulphur compounds. This biotic production of alkalinity takes place at benthic surfaces and is limited by the presence of utilisable organic matter acting as an electron source (Peine and Pfeiffer, 1996; Blodau et al., 1998; Fyson et al., 1998). As the primary production is low (Beulker et al., 2002), the in-

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put, the transport and the deposition of allochthonous organic particles are key processes for the biotic production of alkalinity and hence of interest to remediation (Nixdorf and Deneke, 2002).

Most of the particulate organic material transported in streams and rivers is fine particulate organic matter (FPOM) with particle diameters between 0.45 μm and 1 mm (Webster and Meyer, 1997). These particles have various functions in the aquatic environment (Wotton, 1994). They are associated with nutrients, hydrophobic organic contaminants, and heavy metals (Karichkoff, 1984) and they are an energy source for invertebrates and microbes (Cummins, 1974). After being deposited on the stream bed, fine organic particles can control redox conditions and other physical and chemical factors of ecological importance (Sinsabaugh, 1997). The transfer of fine organic particles from water column to bed sediments is hence of general interest. This process, however, is not fully understood. Various characteristics of the stream, such as width, depth, slope, bed roughness, discharge, flow velocity, water temperature, and dissolved load influence the deposition of particles from the flow (Webster et al., 1987). In addition, characteristics of the particles, such as size, shape, density and their fall velocity, determine their deposition. The fall velocity in quiescent water is known to be a major factor of the deposition in the turbulent flow of streams (O'Melia, 1980; Webster et al., 1987; Reynolds et al., 1990; Prochnow et al., 1991a).

Suspended particles can interact with dissolved and particulate iron. Instream precipitation of iron onto particle surfaces and increasing proportion of aggregate particles were found after the influx of mine drainage into a river (Sullivan and Drever, 2001). Particle characteristics, such as the particle surface charge, also changed in rivers affected by acid mine drainage (Newton and Liss, 1987). Precipitation of iron on particles and a change in particle surface charge can alter the particle deposition. In several studies concerning tracer experiments on particle deposition in streams, the physico-chemical and the chemical interactions between particles and water were obviously not considered and the particle fall velocity was ascertained according to Stokes law (Minshall et al., 2000; Wanner and Pusch, 2000; Paul and Hall, 2002) or by measuring it in a fall cylinder without taking the water quality into consideration (Miller and Georgian, 1992). Experimental techniques to measure the particle fall velocity in a natural water sample facilitate the inclusion of particle water interactions into the determination of the fall velocity. Such methods were developed firstly for size analysis of soil and sediments and then adapted and applied to water samples, described by Puls et al. (1988), Puls and Kühl (1996, 1999), Prochnow et al. (1991b, 1996), Kozerski and Kern (1999).

We hypothesised that interactions between suspended allochthonous fine organic particles and the acidic, iron-rich water change the particle quality and alter their deposition rate. Our objectives were, firstly, to assess such interactions and, secondly, to check their effect on particle fall velocity as an important factor in particle deposition in acidic mining waters. To obtain these objectives, an experimental comparative approach was carried out for two types of acidic, iron-rich water from the post-mining region of Lower Lusatia, Brandenburg, Germany.

2. Methods

2.1. *Water sampling—study area, field methods, chemical analysis*

The sampling sites were situated at the stream Floßgraben in the post-mining landscape of Lower Lusatia (130 km southeast of Berlin), Germany. The stream Floßgraben is a part of the Schwarze Elster drainage system and flows into the mining lake chain of the former mining sites between Grünewalde and Plessa. Firstly, the water was sampled at the stream Floßgraben upstream from its confluence with the mining lake chain. Secondly, the water was sampled in two mining lake outlets below the confluence of the stream Floßgraben with the mining lake chain. The sites were expected to represent two types of water quality that are typical for the post-mining waters of the Lusatia (Nixdorf et al., 2003). Water A, at the site upstream the confluence with the mining lake chain, represents the "very acidic water" and water B, at the two sites at mining lake outlets, represents the "extremely acidic water", due to the influence of the mining lake chain.

To prove this expectation and assess the water quality, water samples were collected monthly from May 2001 to May 2002 at these sites. During the sampling the physico-chemical parameters pH, EC (electrical conductivity) and O_2% (oxygen saturation) were mea-

Table 1
Analytical methods used for characterising solutes in the water ([1]field water samples; [2]water samples from experiments) and for characterising the particle composition ([3]particle samples from experiments)

Parameter	Analytical method	Analytical instrument
^{1}Acidity ($K_{B4.3}$,$K_{B8.2}$)	Titration against NaOH	
^{1,2}DOC	Elemental analysis (DIN 38409-H3)	DIMATEC, DIMA-TOC 100
Anions ($^2Cl^-$, $^2Fl^-$, $^{1,2}SO_4{}^{2-}$–S)	Ion chromatography	Dionex, DX-100
Cations ($^2Na^+$, $^2K^+$, $^2Mg^{2+}$, $^2Ca^{2+}$)	Ion chromatography	Dionex, DX-100
$^{1,2}Fe_{total}$, $^1Fe_{diss,total}$, $^1Mn^{2+}$, $^{1,2}Al^{3+}$	Flame AAS	Perkin-Elmer 3100
$^2Fe_{diss,total}$, $^2Fe^{2+}$	Photometrical detection (510 nm) of the Fe(II)–phenanthrolin complex (DIN 38406-E1)	Dr. Lange, Cadas 100
Particle composition (^{3}C, ^{3}N, ^{3}S)	Elemental analysis	Elementar, Vario el
Particle composition (^{3}P, ^{3}Fe, ^{3}Mn, ^{3}K, ^{3}Ca, ^{3}Si)	Energy dispersive X-ray fluorescence analysis, (20 t pressed pellets, fundamental parameter method, reference material NIST 1575 Pine Needles)	Spectrace 5000, Noran Instruments

sured in the stream with a multiprobe (Hydrolab H20). Samples for chemical analysis were divided into sub-samples. The sub-samples were filtered for analysis of $SO_4{}^{2-}$–S and Fe^{2+}, acidified for analysis of Fe_{total} (total iron), filtered and acidified for analysis of $Fe_{diss,total}$ (dissolved total iron) and Al^{3+} immediately in the field. The water was filtered using a membrane filter (cellulose acetate, 0.45 μm, Macherey-Nagel) and acidified with HNO_3. The sub-samples for analysing $K_{B4.3}$, $K_{B8.2}$ (acidity), and dissolved organic carbon (DOC) were filled in glass flasks taking care to prevent bubbles. The methods for the chemical analysis of the water are given in Table 1.

The water for the laboratory experiments to determine the particle water interactions and the particle fall velocity was collected immediately before the experiments (March 2002: interactions between particles and water B, particle fall velocity from batch A in distilled water, water A and water B; May 2002: interactions between particles and water A, particle fall velocity from batch B in distilled water, water A and water B).

2.2. Particle preparation

To guarantee a standardised analogue for allochthonous fine organic particles, shredded birch leaves (*Betula pendula*) were used in the experiments. Fallen leaves were sampled from a forest floor in autumn 2001, dried at room temperature and shredded with a universal mill (IKA Universalmühle M20). Large particles were removed by sieving (<1 mm) and the smaller particles were homogenised. The effect of the acid mine drainage on the particle fall velocity was tested with two different batches of particles, which were obtained by shredding leaves with different periods.

2.3. Laboratory experiments—interactions between particles and water

For the investigation of the particle water interactions, the fine organic particles were exposed to 1.5 l of the acidic, iron-rich water for 25 h in glass beakers (2000 ml). Four parallel experiments were conducted with a particle concentration of $7\,g\,l^{-1}$. Particles were suspended in water A and water B with and without aeration. During exposure particles were kept in suspension with a magnetic stirrer. The pH value and O_2% were measured continuously. Water and particle samples were taken for chemical analysis before exposure and after 1, 15, and 25 h. The fine organic particles were separated from the water by centrifugation ($2700 \times g$, 4 min) and freeze-dried (−30 °C, 0.37 mbar, 24 h). The chemical composition of particles (C, N, S, P, Fe, Mn, K, Ca, Si) was assessed with the methods given in Table 1. The concentrations of $Fe_{diss,total}$, Fe^{2+}, Mn^{2+}, Al^{3+}, Cl^-, Fl^-, $SO_4{}^{2-}$, Na^+, K^+, Mg^{2+}, Ca^{2+} from the water samples were analysed after filtration by cellulose acetate filters (0.45 μm, Macherey-Nagel). The concentration of Fe_{total} in the water was determined before the exposure of particles to calculate the particulate iron suspended in the water. The concentration of DOC was analysed after filtration using a glass fibre filter (85/90 BF, Macherey-Nagel). Additional experiments were carried out with lower concentrations of particles ($1\,\mathrm{g\,l^{-1}}$, $0.1\,\mathrm{g\,l^{-1}}$) to examine the interaction between particles and water at concentrations closer to natural. In these additional experiments only the dynamics of $\mathrm{Fe_{diss,total}}$ and $\mathrm{Fe^{2+}}$ were considered. The analytical methods are given in Table 1. Additional experiments with artificial iron solutions and fine organic particles were conducted to clarify the observed results and to facilitate the discussion. For these experiments, the prepared fine organic particles were used and the concentrations of $\mathrm{Fe_{diss,total}}$ (partly) and $\mathrm{Fe^{2+}}$ were considered. The experimental conditions are introduced in the respective discussion on particle water interactions.

2.4. Laboratory experiments—particle fall velocity

The fall velocity (v_{fall}) of the fine organic particles was assessed using gravity sedimentation analysis (pipette method) according to modified DIN 66115 (DIN, 1997) and methods from Christensen (1992). The fall velocity was assessed in water A, water B and in distilled water as a control. Two parallel batches of shredded leaves were used that differed in relative composition according to the particle fall velocity. A particle suspension was prepared in distilled water in four steps. Firstly, 10.67 g air-dried particles were added to 400 ml distilled water. The suspension was then dispersed by sonification (duration: 4 min). Afterwards, the pH of the suspension was adjusted to the pH of the acidic water with H_2SO_4, to avoid iron precipitation from the acidic, iron-rich water during the mixing of this water with the suspension of particles. This step was only necessary for suspensions that were used for fall velocity experiments with water A or water B, respectively. Finally, the particles were kept in suspension for 15 h with a magnetic stirrer to remove gas bubbles from the particles. For fractionation, 375 ml suspension was transferred into a fall cylinder and filled up with distilled water or water A or water B. The particles were homogenously suspended in the fall cylinder. To obtain the total mass concentration of suspended particles in the fall cylinder a first sample was taken. The fall velocity distribution of the particles was assessed by taking samples after defined fall periods at a defined depth. The dry mass (105 °C, 24 h) and the loss on ignition (LOI; 550 °C, 3 h) were measured from the samples (DIN 38414-S2, DIN 38414-S3) (DIN, 1999). Seven fall velocity fractions were obtained: ≤ 1, 1–6, 6–24, 24–97, 97–389, 389–608, $>608\,\mathrm{cm\,h^{-1}}$. The mass percentage of every fraction of total mass was referred only to the LOI.

Assuming a lognormal distribution of the particle fall velocity (Prochnow et al., 1991b, 1996), the mean particle fall velocity was calculated as the median ($v_{\mathrm{fall,50}}$), Eq. (1), of this theoretical distribution, with its density function, Eq. (2). The parameters of the lognormal distribution ($\mu_{\mathrm{L}}, \sigma_{\mathrm{L}}$) were calculated with the program SISPE (developed by Prochnow) according to the procedure described by Prochnow et al. (1991b, 1996).

$$\mu_{\mathrm{L}} = \ln v_{\mathrm{fall,50}} \tag{1}$$

Table 2
Water chemistry of the water A, ($n = 12$) and the water B, ($n = 17$), from the small stream Floßgraben

Parameter	Unit	Water A	Water B
pH	–	3.7 (3.2–5.6)	2.9 (2.6–3.2)
Conductivity	($\mu\mathrm{S\,cm^{-1}}$)	427 (210–526)	1370 (1163–1570)
O_2 %	(%)	74 (25–94)	96 (89–100)
Acidity ($K_{B4.3}$)	($\mathrm{mmol\,l^{-1}}$)	0.5 (0.0–2.2)	2.7 (2.1–3.4)
Acidity ($K_{B8.2}$)	($\mathrm{mmol\,l^{-1}}$)	1.10 (0.5–2.9)	3.4 (2.9–4.2)
DOC	($\mathrm{mg\,l^{-1}}$)	4.4 (1.1–8.7)	3.2 (0.7–6.6)
$\mathrm{SO_4^{2-}}$–S	($\mathrm{mg\,l^{-1}}$)	69 (31–117)	274 (227–425)
$\mathrm{Fe_{total}}$	($\mathrm{mg\,l^{-1}}$)	2.3 (1.0–4.2)	29.3 (17.9–38.5)
$\mathrm{Fe_{diss,total}}$	($\mathrm{mg\,l^{-1}}$)	2.1 (0.2–3.7)	29.1 (22.4–38.3)
$\mathrm{Fe^{2+}}$	($\mathrm{mg\,l^{-1}}$)	0.9 (0.2–1.4)	3.2 (1.3–6.2)
$\mathrm{Al^{3+}}$	($\mathrm{mg\,l^{-1}}$)	2.0 (0.3–4.1)	2.6 (1.9–3.1)

The given values are mean and (minimum to maximum).

$$f(v_{\mathrm{fall}}) = \left(\frac{1}{(\sqrt{(2\pi)}\sigma_{\mathrm{L}} v_{\mathrm{fall}})}\right)\exp\left(-\frac{(\ln v_{\mathrm{fall}} - \mu_{\mathrm{L}})^2}{2\sigma_{\mathrm{L}}{}^2}\right) \qquad (2)$$

3. Results

3.1. *Water quality at stream Floßgraben*

The water in the stream Floßgraben is acidic and iron-rich (Table 2). High concentrations of dissolved iron buffered the water at low pH values. The water quality at the stream Floßgraben differed corresponding to the prevailing water sources. The stream Floßgraben, upstream from its confluence with the mining lake chain, is fed by seepage from shallow groundwater and interflow from forrested banks. Downstream the confluence with the mining lake chain the acidity of its water was significantly higher. The increase of the acidity was accompanied with lower pH, approximately 3 times higher concentration of $SO_4{}^{2-}$–S and more than 10 times higher concentration of Fe_{total}, followed by 3 times higher EC values. These differences in water quality confirmed the expectation, that water A and water B represent different types of acidic water. Water A corresponds to the class ‘very acidic, pH below 4’ and water B corresponds to the class ‘extremely acidic, pH below 3.2’, according to the classification for acidic mining lakes (Nixdorf et al., 2003). As a result of these clear differences in water quality we carried out all the experiments with water A and water B.

3.2. *Interactions between particles and water*

To illustrate the particle water interaction, the dynamic of water quality during the exposure of the particles in the acidic, iron-rich water is presented first followed by the comparison of particle quality before and after exposure in the acidic, iron-rich water.

3.2.1. *Change in water quality during particle exposure*

The very highly concentrated suspended particles caused strong consumption of oxygen. Without aeration O_2% declined to less than 5% during the 25 h of the experiment, whereas with aeration the saturation of O_2 stayed at the initial level (Fig. 1a and b). However, aeration during the experiments did not affect the water particle interaction. (Fig. 1c–f, Fig. 2a–f). Therefore, the results of the aerated and non-aerated experiments (Tables 3–5, Figs. 1c–f and 2a–f) are presented together.

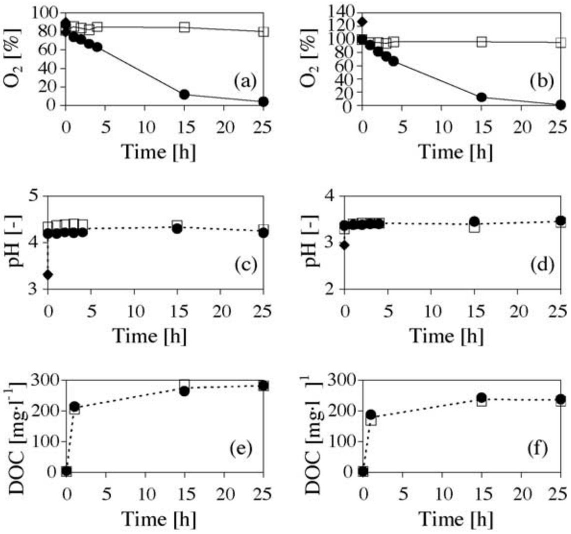

Fig. 1. Change of the physico-chemical parameters: oxygen saturation (O_2%) (a and b) and pH (c and d) and of the dissolved organic carbon (DOC) (e and f) during the exposure of the fine organic particles in water A and water B. The (●) symbol represents experiments without aeration, the (☐) symbol represents experiments with aeration and the diamond represents the initial water.

Despite the differences in the initial chemical quality of water A and B (Table 2), the suspended particles induced similar changes of the water quality in both types of water (Fig. 1c–f, Fig. 2a and b, Fig. 2e and f), excluding Fe^{2+} (Fig. 2c and d). The most significant changes occurred within the first hour after particle addition.

The pH increased very quickly during the 10 min, in which the particles were added and stirred into the water (Fig. 1c and d). The increase was stronger in water A then in water B. The pH then stayed almost constant.

The concentration of DOC increased during the exposure of the fine organic particles (Fig. 1e and f) and revealed a leaching of the suspended particles. The concentration of DOC was slightly higher in water A, whereas the increase in the other substances was nearly identical in both types of water. The concentrations of Mn^{2+}, Na^+, K^+, Mg^{2+}, Ca^{2+}, Cl^-, and F^- in solution increased also due to the leaching (Table 3).

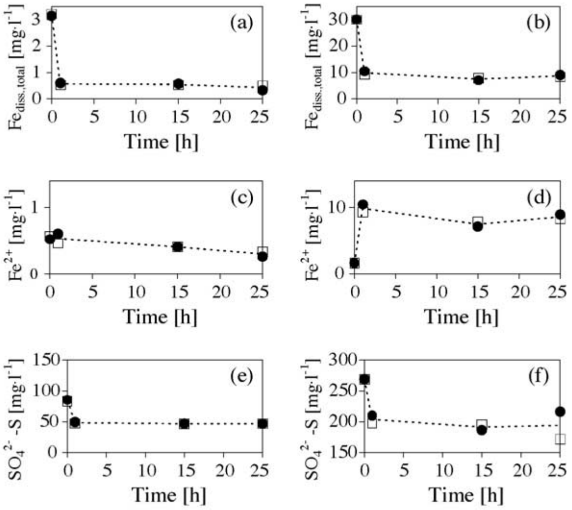

Fig. 2. Change of water chemistry during exposure of the fine organic particles in water A and water B: dissolved total iron ($Fe_{diss,total}$) (a, b); Fe^{2+} (c, d); SO_4^{2-}–S (e, f). The (●) symbol represents experiments without aeration and the (☐) symbol represents experiments with aeration.

The concentration of $Fe_{diss,total}$ decreased from the initial 3.2 to 0.4 mg l^{-1} in water A and from 30.1 to 8.7 mg l^{-1} in water B and remained at these low levels throughout the following experimental period (Fig. 2a and b). The concentration of Fe^{2+} decreased slightly in water A, but clearly increased in water B. However, in both types of water, the proportion of the Fe^{2+} to $Fe_{diss,total}$, increased after particle addition. This increase was from 17 to 71% in water A and from 5 to 99% in water B. The increase of this proportion in the water A was due to the decrease of $Fe_{diss,total}$, whereas the increase in water B was because of the decrease of $Fe_{diss,total}$ and a change of the remaining $Fe_{diss,total}$ into Fe^{2+}. A reduction of Fe^{3+} to Fe^{2+} and a decrease of $Fe_{diss,total}$ was also observed for lower particle concentrations in the aerated water B (Fig. 3a and b). Both processes were clearly related to the particle concentration. For 1 g particles per litre all Fe^{3+} was reduced to Fe^{2+}, whereas for 0.1 g particles per litre 73% of $Fe_{diss,total}$ was not reduced. The kinetics of this transformation indicated that the reduction of Fe^{3+} to Fe^{2+} was almost completed within 1 h.

Table 3
Change in water chemistry during 25 h of exposure of the fine organic particles in water A and water B

Parameter	Time	Al^{3+}	Mn^{2+}	Na^+	K^+	Mg^{2+}	Ca^{2+}	Cl^-	F^-
Unit (mg l^{-1})									
Water A	Initial	2.2	0.2	6.4	2.6	4.8	34.7	16.6	0.1
	Final	0.8	3.5	7.6	28.9	13.6	47.6	28.9	0.5
Water B	Initial	3.0	0.9	6.3	3.9	12.5	112.8	14.8	0.1
	Final	2.1	4.6	7.2	31.7	22.7	117.7	29.8	1.6

The values are means of experiments with aeration and without aeration.

Table 4
Change in particle composition during 25 h of exposure of the fine organic particles in water A and water B

Parameter	Unit	Initial	Final	
			Water A	Water B
C	(mg g^{-1} particles)	515	511	514
N	(mg g^{-1} particles)	11.3	11.2	11.8
S	(mg g^{-1} particles)	1.4	1.3	1.3
P	(mg g^{-1} particles)	1.6	0.9	1.2
Fe	(mg g^{-1} particles)	0.4	1.1	4.5
Mn	(mg g^{-1} particles)	1.2	0.6	0.3
K	(mg g^{-1} particles)	3.5	0.6	0.5
Ca	(mg g^{-1} particles)	12.7	12.7	9.7
Si	(mg g^{-1} particles)	5.6	5.5	4.7

The values are means of experiments with aeration and without aeration.

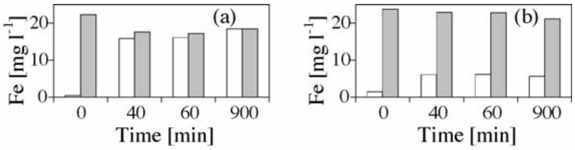

Fig. 3. The change of Fe^{2+} (white bars) and $Fe_{diss,total}$ (grey bars) during exposure of the fine organic particles in water B with aeration. The particle concentration was 1 g l^{-1} (a) and 0.1 g l^{-1} (b).

$SO_4{}^{2-}$–S decreased from 84.5 to 46.7 mg l^{-1} in water A and from 268.7 to 194.0 mg l^{-1} in water B (Fig. 2e and f). Al^{3+} also diminished in water A from 2.2 to 0.8 mg l^{-1} and in water B from 3.0 to 2.1 mg l^{-1}.

3.2.2. *Changes in the particle composition during particle exposure*

The particles lost similar quantities of easily leachable compounds in both types of water. Dissolved organic compounds accounted for a 10% mass loss in water A and a 9% mass loss in water B (Fig. 1e and f) and the leaching of Mn, Na, K, Mg, Ca, Si, Cl and F accounted for additional 1% mass loss in both types of water (Tables 3 and 4). Although iron attached to the particles (Table 4), particles in water B accumulated substantially more iron than particles in water A.

The changes in the particle composition compared to the changes in the water are shown in Table 5. It was not possible or intentional to get completely balanced values between the changes in water and in the particle composition, because different analytical methods were used. However the changes in both should refer to the same processes, indicated by differences in the sign, that is to say, plus or minus. The comparison confirmed the leaching of mineral elements for K and Mn. The loss of Fe_{total} was accompanied by an attachment of iron to the particles. A corresponding increase in sulphur on particles could not be found for the loss of sulphate from the water.

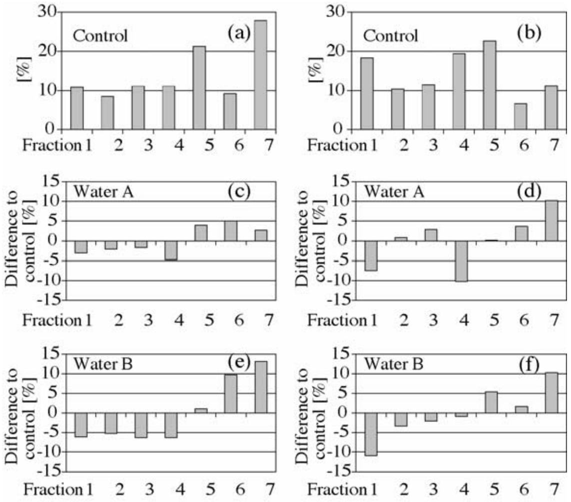

Fig. 4. (a and b) The fall velocity distribution of fine organic particles in distilled water (control) is presented as proportions of total ash free dry mass. (c–f). The difference of the fall velocity fractions in water A (c and d) and B (e and f) from the fall velocity fractions in distilled water, according to: Difference to control = fraction water type A (or B) − fraction control. The fractions are: (1) ≤1, (2) 1 to 6, (3) 6 to 24, (4) 24 to 97, (5) 97 to 389, (6) 389 to 608 and (7) >608 cm h^{-1}.

3.3. *Particle fall velocity*

The particle batch A consisted of a higher proportion of fast falling particles than particle batch B (Fig. 4a and b). Both batches of particles fell faster in the acidic, iron-rich water than in distilled water. The increase in the particle fall velocity is shown by the increase in the $v_{fall,50}$ (Table 6). The increase in the mean fall velocity was caused by a shift in the relative proportions of

Table 5
Changes in the particle composition compared to the changes in the water during 25 h of exposure

Parameter	Unit	Exposition in water A		Exposition in water B	
		Difference in		Difference in	
		Water	Particle composition	Water	Particle composition
Ca	(mg l^{-1})	13	0	5	−21
K	(mg l^{-1})	26	−21	28	−21
Mn	(mg l^{-1})	3	−4	4	−6
Fe	(mg l^{-1})	−3	5	−25	29
S	(mg l^{-1})	−38	−1	−75	−1

Table 6
Parameters of the particle fall velocity distribution in distilled water (control), in water A (very acidic water) and in water B (extremely acidic water)

Parameter	Particle concentration (C)	Lognorm parameter μ_L	Lognorm parameter σ_L	Median $v_{fall,50}$
Unit	(LOI mg l^{-1})	–	–	(cm h^{-1})
Control, batch A	5510	4.8338	3.279	125.69
Water A, batch A	5095	5.3543	3.125	211.51
Water B, batch A	5292	5.9804	3.041	395.60
Control, batch B	4141	3.5719	3.290	35.58
Water A, batch B	3728	4.6508	3.193	104.67
Water B, batch B	3308	4.6429	2.904	103.84

the particles from slow falling to fast falling fractions (Fig. 4c–f). This shift was stronger in water B than water A.

4. Discussion

4.1. Interactions between particles and water

The leaching of ions and DOC from the particles and the association of iron to the particles were established for very acidic and extremely acidic water and it was connected with a reduction of iron in the extremely acidic water.

The shredding of the leaves increased their leaching as expected. The shredded particles were leached faster and about 100% more DOC than intact birch leaves exposed in extremely acidic water (Siefert and Mutz, 2001). The slightly higher level of DOC found in the leachate in the less acidic water A, matched the results of Siefert and Mutz (2001), who found more DOC leachate in neutral water compared to very acidic water.

In contrast to the leaching, approximately two-thirds of the $Fe_{diss.total}$ and $SO_4{}^{2-}$–S were removed from the water. The removed $Fe_{diss.total}$ attached to the particles (Table 5). Such attachment could be due to precipitation of amorphous Fe-oxyhydroxides (a) or precipitation of Fe-sulphides (b) or adsorption of Fe^{2+} (c) on the particles. These three ways will be discussed.

The precipitates of amorphous Fe-oxyhydroxides (a), for example ferrihydrite and Schwertmannite, are known to associate with organic particles (Bigham et al., 1994, 1996; Cornell and Schwertmann, 1996). Such a formation of iron oxides was documented on living and dead bacteria, which served as passive nucleation elements for iron oxides (Ferris et al., 1989). Precipitation of iron onto particle surfaces was also found in streams under the influence of acid mine drainage (Sullivan and Drever, 2001). A precipitation of Schwertmannite at the measured pH (Fig. 1c and d) is possible (Bigham et al., 1996) and would explain the loss of $SO_4{}^{2-}$–S from the water. However, we found no visible ochreous precipitation in the water or coating the particles and we could measure a remarkable increase of iron on the particles but no increase of S (Table 4). In addition, all remaining iron in the water B was reduced to Fe^{2+} in the experiments with the high particle concentration within 1 h.

The reduction of Fe^{3+} to $Fe^{2+}$and the suspension turning black support the assumption of precipitation of Fe-sulphides (b). But this assumption cannot be accepted, because a precipitation of Fe-sulphide would also increase the content of S in the particle composition. That was not the case, see Table 4. The control experiment b with $FeCl_3$ (Table 7) confirmed that the reduction of Fe^{3+} to Fe^{2+}also took place without sulphate in the initial water.

The adsorption of Fe^{2+} on the particles (c) is probable. To confirm definitely this adsorption theory, the loss of Fe^{2+} from an artificial solution ($FeCl_2$ or $FeSO_4$) should be observed in a further experiment. For an adsorption, the particle surfaces must have been negatively charged (Newton and Liss, 1987). And this is the case for the most organic particles without iron coatings in the water environment (Newton and Liss, 1987).

But in which way the Fe^{3+} was reduced to Fe^{2+}? Three general hypotheses will be discussed, supported by the control experiments with artificial solutions

Table 7
Experiments to investigate Fe^{3+} reduction processes in artificial Fe(III)solutions by reductive substances (16 h leachate of the organic particles (b, ca, cc) and oxalate acid (cb))

No.	Process	Experimental conditions (C_P–concentration of leached particles) (C_{OA}–concentration of oxalate acid)	Initial Fe^{2+} (mg l^{-1})	Initial Fe^{3+} (mg l^{-1})	Final Fe^{2+}
b	Fe-sulphide formation	C_P (1 g l^{-1}); light; not sterilised			
		(a) $FeCl_3$ solution	0.7	20.3	4.2
		(b) $Fe_2(SO_4)_3$ solution	0.4	15	3.1
ca	Microbial reduction	$Fe_2(SO_4)_3$, pH 2,8; C_P (100 mg l^{-1}); light			
		(a) Not sterilised	0	5	0.9
		(b) Sterilised	0	5	1.3
		$Fe_2(SO_4)_3$, pH 2,8; C_P (1 g l^{-1}); light			
		(c) sterilised	0	5	4.1
cb	Chemical reduction	$Fe_2(SO_4)_3$, pH 2,8; C_{OA} (50 mg l^{-1})			
		(a) Light	0	10	9.5
		(b) Dark	0	10	9.7
cc	Photo-chemical reduction	$Fe_2(SO_4)_3$, pH 2,8; C_P (100 mg l^{-1}); sterilised			
		(a) Light	0	5	1.3
		(b) Dark	0	5	0.9
		$Fe_2(SO_4)_3$, pH 2,8; C_P (1 g l^{-1}); sterilised			
		(c) Light	0	5	4.1
		(d) Dark	0	5	3.9

The duration of the experiments was 240 (b), 210 (ca, cc) and 60 min (cb).

(Table 7). A reduction of Fe^{3+} to Fe^{2+} in the oxygenated water could have been caused by microbial processes (ca) or via chemical reductants (cb), accelerated in sunlight by photo-processes (cc) (Pronk and Johnson, 1992; Voelker et al., 1997; Herzsprung et al., 1998).

The sterilisation of the particle leachate (autoclave, 20 min, 125 °C) did not decrease the Fe^{3+} reduction for leachate from the particle concentration 100 mg l^{-1}. The sterilised particle leachate from 1 g l^{-1} reduced the most of Fe^{3+} within the 210 min. This means a microbial reduction could be excluded (ca).

The reduction of Fe^{3+} must be caused by chemical reductants (cb). The chemical reduction of Fe^{3+} is known. Bloomfield (1957) demonstrated abiotic dissolution of synthetic iron oxides by reduction and complexation reactions even under aerobic conditions with extracts from leaves. As an example for chemical reduction, experiment cb showed that oxalate acid is able to reduce most of the Fe^{3+} from our artificial solution. From the exclusion of the other possibility (ca) and from the citied literature we conclude that the reduction from Fe^{3+} to Fe^{2+} is caused by chemical reductants; for example oxalate, fulvic acid, hydroxy and amino carboxylic acids and phenols (Voelker et al., 1997; Cornell and Schwertmann, 1996), which are to be expected in the litter layer on forest floor (Blume, 1965).

A photochemical acceleration of this process seems to be low. The reduction of Fe^{3+} to Fe^{2+} from the $Fe_2(SO_4)_3$ solution by particle leachate was increased slightly if the light was not excluded in experiment cc (Table 7).

The removal of the $SO_4{}^{2-}$–S from the water could not be found as a corresponding increase on the particles (Tables 4 and 5). The formation of gaseous H_2S is one possibility to explain the discrepancies. A reduction of $SO_4{}^{2-}$–S to H_2S is accompanied by an oxidation of organic material and an increase in pH (Peine, 1998) and could also explain the suspension turning black. To clarify the fate of the $SO_4{}^{2-}$–S further experiments and a detection of H_2S are necessary.

To summarise, the main processes of the observed water particle interaction were firstly a release of DOC from the organic particles, which contained chemical reductants which in turn were able to reduce the Fe^{3+} to Fe^{2+} and the reduced Fe^{2+} attached to the particle surfaces by adsorption.

Table 8
Comparison of the particle fall velocity of the studied fine organic particles and of in situ seston from streams and the sea, reported by other studies

Mean fall velocity (median, $v_{\text{fall},50}$)[1] (modal, $v_{\text{fall,modal}}$)[2] (cm h^{-1})	Particles (sampling site, characteristic)	Reference
35.58 and 125.69[1]	Fine organic particles	This study
Between 1 and 9[1]	Old branch River Spree, Seston	Engelhardt et al. (1996)
Between 7 and 15[1]	Channel Oder-Spree, Seston	Prochnow et al. (1991b)
Between 1 and 22[1]	River Spree, Seston	Prochnow et al. (1996)
Between	North Sea, particulate matter	Jago et al. (1993)
–72 and 1800[2]	–resuspension	
–0.036[2]	–background	
–3.6 and 1080[2]	–phytoplankton	
Between 0.36 and 36[1]	German Bight, suspended particulate matter	Puls and Kühl (1996)

4.2. *Particle fall velocity*

The fall velocity of the fine organic particles from this study in comparison to fall velocities from in situ seston of streams and of the sea is shown in Table 8. The median fall velocity from batch B in distilled water (36 cm h^{-1}) falls into line with the highest median fall velocities of suspended sediments in the River Spree (Prochnow et al., 1996). A higher median fall velocity such as for batch A is documented for marine particulate material by several studies shown in Table 8.

The median particle fall velocities increased for both batches in the acidic iron-rich water. The higher increase in the fall velocity in the water B (extremely acidic) compared to water A (very acidic), suggests that it is related to the acidity and the concentration of iron. The increase in particle fall velocity in acidic, iron-rich water could have been caused by two processes: firstly, the rise in particle density in acidic, iron-rich water and secondly by the formation of aggregates from smaller particles.

The particle density can increase by a shift toward a higher inorganic proportion of the particles according to Eq. (3) (Minshall et al., 2000). Therefore, the predominant leaching of organic compounds from the particles could have increased their density. However, as more leaching in the extremely acidic, iron-rich water (water B) compared to the distilled water and to the very acidic, iron-rich water (water A) was not observed nor expected (Siefert and Mutz, 2001), this could not explain the much higher increase in the fall velocity measured in water B. Another process that increased the inorganic proportion of the particles was the attachment of iron. This attachment process was insignificant, because although the fall velocity increased, the increase was less than 2%, calculated according to Eq. (3) and Stokes law Eq. (4):

$$\rho_{\text{particle}} = p\rho_{\text{o}} + ((1 - p)\rho_{\text{i}}) \tag{3}$$

$$v_{\text{fall}} = \frac{(2gr^2(\rho_{\text{particle}} - \rho_{\text{water}}))}{9\eta} \tag{4}$$

where p = LOI (%), ρ_{particle} = particle density (g cm^{-3}), ρ_{o} = organic density (1.25 g cm^{-3}), ρ_{i} = inorganic density, (2.5 g cm^{-3}), v_{fall} = fall velocity (m s^{-1}), g = 981 cm s^{-2}, r = particle radius (cm), ρ_{water} = water density (g cm^{-3}), and η = dynamic viscosity (mPa s^{-1}). We therefore, conclude that the clear increase in the mean particle fall velocity in the acidic, iron-rich water was not caused just by an increase in the particle density.

From this we conclude that the increase of the median particle fall velocity in the acidic, iron-rich water is due to particle aggregation. Particle aggregation results in a formation of larger particles which fall faster than a single particle, according to Eq. (4). Processes of formation of aggregates and the research of these processes were summarised by Eisma (1992) for suspended matter and reviewed by van Leussen (1988) for estuarine suspended matter. Frequently reported processes are aggregation by turbulence, by organisms, by exudates from bacteria and phytoplankton, by sticky organic compounds (fulvic acid), by adherence of positively to negatively charged particles, by differential settling, by high particle concentration, by precipitation, polymerisation, adsorbance onto particles and flocculation of dissolved inorganic or organic matter and by salt flocculation. The last three processes, (aggregation by high particle concentration, by precipitation and by salt flocculation) should be discussed to see, if they are able to explain the aggregation of fine organic particles in our quiescent water and therefore cause the stated differences between the fall velocity experiments.

Firstly, the total particle concentration was different for every experiment. However, the increase in the fall velocity from distilled water, to water A and to water B was not correlated for both batches with the particle concentration (Table 6).

Secondly, water A and water B contained dissolved and particulate iron and the distilled water did not. An aggregation of organic particles in acidic, iron-rich waters with Fe-oxyhydroxides serving as electrostatic glue between the particles is known (Sullivan and Drever, 2001) and a precipitation of such Fe-oxyhydroxides was shown by Ferris et al. (1989). In our study however, the precipitation of Fe-oxyhydroxides on particle surfaces was prevented by the reduction from Fe^{3+} to Fe^{2+} and so an aggregation in this way can be excluded.

Thirdly, water A and water B differed to the distilled water in their low pH and their high content of ions, which appears in the high electrical conductivity. From the salt aggregation research it is known, that a high salinity reduces the repulsive forces between particles through a neutralisation of originally negative charged particles by a cloud of positively ions around every particle (Eisma, 1992). A high hardness and also a low pH reduce the electrophoretic mobility of particles, making them less repulsive and increase their aggregation (Gerritsen and Bradley, 1987). Newton and Liss (1987) found a change from obviously negatively to positively charged particles in the iron-rich Carnon River, UK with decreasing pH and increasing electrical conductivity. We conclude from these references that the higher median particle fall velocity was due to a particle aggregation based on the low pH and the high electrical conductivity of water A and B compared to the distilled water. Fall velocity experiments in artificial acidic solutions would be helpful to confirm the discussed aggregation processes.

5. Summary and conclusion

In the experiments for investigation the interactions between particles and acidic, iron-rich water the fine organic particles released organic compounds. The Fe^{3+} was reduced to Fe^{2+} in extremely acidic water and it could be shown that the Fe^{2+} attached to the fine organic particles when the particle concentration was high enough. Experiments with artificial iron solutions clarified the way of iron attachment to the fine organic particles and the way of iron reduction. Excluding the precipitation of amorphous Fe-oxyhydroxides or the precipitation of Fe-sulphides, we conclude that Fe^{2+} attached to the particles by adsorption. To confirm this adsorption theory categorically, the loss of Fe^{2+} from an artificial solution should be observed in a further experiment. It could be further shown that the Fe^{3+} was reduced to Fe^{2+} by chemical reductants and not by microbial processes and, furthermore, that the photochemical processes were not important for the yield in Fe^{2+}. A removal of $SO_4{}^{2-}$–S from the water could be shown, however it was not accompanied by an increase of sulphur on the particles. Also, additional experiments with artificial iron solutions and an analytical detection of gaseous H_2S would be useful to clarify the fate of sulphur.

The median particle fall velocity increased in acidic, iron-rich water. We discussed a rise in particle density in acidic, iron-rich water and an aggregation process as a reason for this increase. The calculated increase in the particle density was insignificantly low and could not explain the increase of particle fall velocity. We concluded, the increase is due to aggregation and three possible aggregation processes were discussed. The particle water interactions must be considered in the investigation of fall velocities, especially in acidic, iron-rich mine drainage water.

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