

Geochemistry and partitioning of trace metals in acid sulphate soils in Sweden and Finland before and after sulphide oxidation

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Available online 12 February 2004

Abstract

Acid sulphate soils (ASS) are formed on sulphidic sediments along the Baltic coasts of Sweden and Finland. The sulphides in the fine-grained sediments were once formed in an anoxic environment some centimetres below an oxic sediment–water interface. In earlier investigations, high concentrations of many trace elements have been observed in streams draining such areas.

We compared the trace element contents in soils formed in sulphidic and non-sulphidic clays. The results show that the total contents of Cd, Ni, Mn, Co and Cu in the sulphidic sediments are not higher than in non-sulphidic clays from adjacent areas. However, the mobilisation of Cd and Ni is higher from acid sulphate soils than from soils formed on sulphide-free clays.

To understand the processes causing the mobilisation, we studied the partitioning of trace metals before and after oxidation of sulphidic sediments. Cadmium, Ni, Mn, Co, Zn, and to some extent Cu, were found to be mobilised from acid sulphate soils due to increased weathering caused by the low pH in these soils. Iron and Cr are only mobilised to a limited extent. Copper, Mo and Fe mobilised from weathering are precipitated in oxides that form in the oxidised Bg-horizons.

Nickel, Co, Mn, Mo and Cu were shown to be partly bound in pyrite and are mobilised from acid sulphate soils during the pyrite oxidation. However, mobilisation of trace elements from pyrite was not found to be an important quantitative contributor to trace element leaching.

Any elements that are mobilised are likely to be transported down through the characteristic cracks, which occur frequently in the oxidised horizons. Evidence of this was found at the groundwater table, where the most easily extractable fraction of mobilisable elements occurred at higher concentrations.

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Keywords: Acid sulphate soils; Iron sulphides; Trace elements

1. Introduction

Along the Baltic coasts of Sweden and Finland, acid sulphate soils (ASS) are formed on fine-grained

sulphidic sediments (Öborn, 1989; Åström and Björklund, 1996). The low pH in these soils is caused by oxidation of iron sulphides (van Breemen, 1982). High concentrations of certain trace elements (e.g. Ni, Cu and Mn) have been observed in streams draining areas dominated by ASS (Andersson et al., 1988; Ekelund et al., 1993; Åström, 2001; Åström and Björklund, 1996). It has been suggested

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that the weathering process is more intense in ASS than in fine-grained soils with higher pH (Palko and Yli-Halla, 1990).

In the present paper, we compare the total trace element content in sulphidic and sulphide-free sediments, to establish whether high sedimentary contents contribute to the intense leaching from ASS. Furthermore, we compare ASS with soils developed from clay sediments without sulphides, to establish to what degree the trace element mobilisation is due to conditions specific to ASS.

Little is known about the chemical processes involved in the redistribution and mobilisation of metals from ASS, even though there are some studies from Finland (Åström, 1998). We studied how Cd, Co, Ni, Fe, Mn, Cr, Zn, Mo and Cu are partitioned in the reduced sulphidic parent material and how this partitioning is different in the oxidised ASS. Several trace elements are associated with pyrite (FeS_2) (Huerta-Diaz and Morse, 1992; Sternbeck et al., 2000) and will be dissolved during the oxidation process. One aim of our investigation was to establish whether elements are mobilised from ASS due to release from oxidised pyrite or due to increased weathering of silicate minerals caused by the low pH.

Oxidation causes the formation of iron oxides and it is possible that certain metals are co-precipitated in, or adsorbed onto, these oxides, whereas others are leached out of the soil. A further aim was therefore to establish whether some elements are co-precipitated in oxides.

The reason for the low pH in ASS is the oxidation of iron sulphides, mainly FeS and FeS_2 (van Bree-men, 1982). There are, however, only a few investigations determining the iron sulphide content and forms (FeS and FeS_2) in anoxic sediments, which can potentially form ASS (Georgala, 1980; Sternbeck and Sohlenius, 1997). We therefore analysed the FeS content (in addition to total-S) in samples from one of the sites studied.

There are several studies dealing with the environmental development of the present Baltic Sea as it is reflected in the chemical composition of Holocene sediments (Sohlenius and Westman, 1998; Sternbeck et al., 2000). Less is known about the formation of sediments uplifted above the present sea level. The formation of the sulphidic sediments investigated here is therefore discussed briefly.

2. Materials and methods

2.1. Site description

In both Sweden and Finland fine-grained clay (and silt) sediments, which were originally formed at the bottom of the Baltic Sea, are often used as arable land. These sediments were deposited after the latest deglaciation and have been uplifted due to the ongoing isostatic land upheaval. The most widespread glacial and postglacial clays have low contents of organic carbon and iron sulphides. In certain areas, however, the clay sediments contain organic material and iron sulphides. These sediments are often referred to as gyttja clay (3–6% organic matter). Such sediments were mainly deposited in the Litorina Sea, from about 7000 to 4000 BP (Fromm, 1965). The ASS developed during the oxidation of the uppermost, drained, part of these naturally formed sediments, often after drainage for agricultural purposes (Öborn, 1991). The ASS in northern Sweden have a lower clay content than corresponding soils in Central Sweden (Öborn, 1989).

The present investigation deals mainly with samples from six sites, five in Sweden and one in Finland (Table 1). Three of the sites sampled are ASS underlain by sulphidic sediments; Hylinge, Hacksta and Mustasaari. The three other soils are formed on clays where iron sulphides are absent and are therefore not acidic. However, the results in Table 2 were compiled from a total of 16 sites situated along the former and present Baltic coast in Central Sweden.

All the sites sampled are situated on arable land where the ground water table has been artificially lowered by drainage. At one of the ASS sites, Hacksta farm, a core sample was also taken from an adjacent fen where the groundwater table is situated close to

Table 1
Geographical data from the six sampled sites

Site	Latitude	Longitude	m a.s.l.	Texture	
Ammantorp	64°94'95"N	15°50'90"E	10–15	Clay	Sweden
Bergshammar	65°89'20"N	15°60'15"E	8	Clay	Sweden
Vagnstad	64°91'30"N	15°47'10"E	15	Clay	Sweden
Hylinge	64°93'20"N	15°48'90"E	5–10	Gyttja clay	Sweden
Hacksta	59°32'N	17°23'E	0	Gyttja clay	Sweden
Mustasaari	63°05'N	21°71'E	0	Gyttja clay	Finland

Table 2

The average total contents of Cd, Ni, Cu, Co, Mn and TiO₂ in the B and C-horizons from clay sediments with and without iron sulphides

	Gyttja clay with iron sulphides, <i>n</i> =41, 10 sites		Clay without iron sulphides, <i>n</i> =25, 6 sites	
Horizon	B	C	B	C
Cd (mg/kg)	0.087 ± 0.02	0.1640 ± 0.03	0.154 ± 0.080	0.1870 ± 0.083
Ni (mg/kg)	31.7 ± 3.5	43.9 ± 5.6	42.1 ± 10.6	42.9 ± 10.0
Cu (mg/kg)	31.90 ± 3.2	35.2 ± 6.7	38.0 ± 8.4	37.3 ± 8.4
Co (mg/kg)	36.0 ± 2.9	40.9 ± 3.5	38.5 ± 6.6	41.5 ± 5.1
Mn (mg/kg)	440 ± 70	700 ± 150	484 ± 157	697 ± 48
TiO ₂ (%)	0.7400 ± 0.04	0.738 ± 0.060	0.742 ± 0.068	0.759 ± 0.075

Standard deviation is shown in the ± columns.

the soil surface. This fen was assumed to provide a picture of the soil conditions before drainage.

The studied acid sulphate soil profiles in Central Sweden and the Finish Mustasaari profile are characterised by a dark brown plough layer (Ap, sometimes Hp horizon) overlaying greyish yellow or yellowish grey Bg and BCg horizons with a strongly developed structure which is angular blocky down to about 50 cm from where vertical cracks and prismatic aggregates are dominating. Brown and yellowish brown (presumably Fe-oxyhydroxides) mottles are common in the Bg and BCg horizons and cutans have formed on ped surfaces and in old root channels. The C-horizon (Cr) is situated below the groundwater table and is recognised by grey or olive grey colours and few black dots or stripes of Fe-sulphides. Öborn (1989) has described acid sulphate soils in Central Sweden in detail. They are mainly classified as Gleyic Cambisols (FAO, 1988), or Humaquepts or Dystrichrepts (Soil Survey Staff, 1992), since pH in the Bg horizons is no longer low enough to qualify as sulphuric horizon (Öborn, 1994). Most of the acid sulphate soils in these areas have been artificially drained for 100 years or more. The acid sulphate soils in Finland have been described and classified according to international systems by Yli-Halla (1997) and Yli-Halla et al. (1999).

2.2. Sampling

The ASS were sampled in open pits whereas underlying reduced sediments, below the groundwater table, were sampled with a Russian peat corer. The soil profiles were described according to FAO guidelines for soil profile description (FAO, 1990). The samples were taken from three groups of horizons, the oxidised

horizons (ASS) and the underlying transitional (CBg and Cg horizons, TZ) and the reduced zones (Cr horizons, RZ). Samples were taken at dense intervals around the groundwater table, where drastic changes in pH and redox potential were expected to occur.

The core samples from the reduced zone were wrapped firmly in plastic and frozen as fast as possible to avoid oxidation.

2.3. Chemical analyses

The pH was measured on fresh samples after shaking the samples in de-ionised water (1:2.5) at room temperature (Swedish Standard Institute, 1994). The remaining samples were dried at 35 °C and sieved through a 2-mm mesh for metal and total C and S analyses. Total concentrations of C and S were measured on fine-grained samples using a Leco CHN 932.

The total metal content was analysed to quantify the magnitude of mobilisation. The majority of the samples was finely ground (agate mortar) and analysed by X-ray fluorescence (XRF). An exception was Cd which was present in too low concentrations for the XRF analyses and therefore was analysed by atomic adsorption with graphite furnace (Perkin Elmer AAS 4110 ZL) after extraction with 7 M boiling HNO₃ (Swedish Standard Institute, 1997). The samples from Hacksta and Mustasaari were fused in a carbon crucible with a flux (lithium metaborate) at 1000 °C. The “bead” which formed was dissolved in HNO₃ and the metal concentrations were determined with ICP-MS.

Sequential extractions were carried out on samples from the three ASS sites. This was done to determine the partitioning of metals. These samples were taken at the same depths as the samples analysed for total

element contents. The following steps were included in the sequential extraction: *Step 1* NH_4NO_3 (water soluble and exchangeable), *Step 2* Na-pyrophosphate (organically bound), *Step 3* oxalate acid and ammonium oxalate pH 3.25 (oxides), *Step 4* (crystalline oxides) 0.5 M HCl and *Step 5* conc. HNO_3 (metals in pyrite). The first three steps are described thoroughly by [Elsokkary and Låg \(1978\)](#) and a method described by [Huerta-Diaz and Morse \(1992\)](#) was used to determine metals bound in pyrite.

The metal contents were determined with ICP-MS except Step 2, which was analysed using atomic absorption with a graphite furnace.

Step 2 was only analysed for Ni, Cd, Cu and Zn. It was not possible to analyse Zn in Step 3, Cd in Steps 3–5 and the total Mo content.

In three samples from Hacksta, FeS–S was analysed as acid volatile sulphide (AVS) in a nitrogen atmosphere (6 M HCl; [Cornwell and Morse, 1987](#)). The pyrite content in RZ was calculated as the difference between total-S and AVS-S.

3. Results

3.1. Total geochemistry of acid sulphate and non-acid sulphate clay soils

The ASS are characterised by low pH (3.9–4.5) and low total S content ([Fig. 1](#), white dots). Both pH

and total S increase in TZ, where the groundwater table is situated ([Fig. 2](#)). There is almost no sulphur in ASS, indicating that the sulphides have been oxidised and most of the S has been leached out of the profile. The content of organic carbon does not vary below the Ap horizon. The soils formed from sulphide-free sediments are characterised by low S content and pH between 7 and 8 throughout the profiles ([Fig. 1](#), black dots). The low S content below the groundwater table indicates the absence of iron sulphides.

In the C-horizon, there are no significant differences in the total trace element contents (Cu, Ni, Mn, Co and Cd) between soils with and without sulphides ([Table 2](#)). A comparison between the average total contents in the B- and C-horizons shows that Cd, Ni, Mn, and to a small extent Cu and Co, have been leached out from the ASS ([Table 2](#)). Manganese is the only element that has been leached out from soils formed from sulphide-free sediments ([Table 2](#)). The average TiO_2 content shows small variations within the soils, which suggests that the original sediments were homogeneous with respect to their mineral and element contents ([Table 2](#)). In all soils, the surface horizon is high in total Cd, which is due to application of Cd-contaminated P-fertiliser and atmospheric deposition (cf. [Eriksson et al., 1997](#)).

The total metal contents show that Cd, Ni, Mn, Co and Cu have been mobilised from ASS ([Figs. 2 and 3](#)). However, Cu is not mobilised from ASS to the same extent as the other elements.

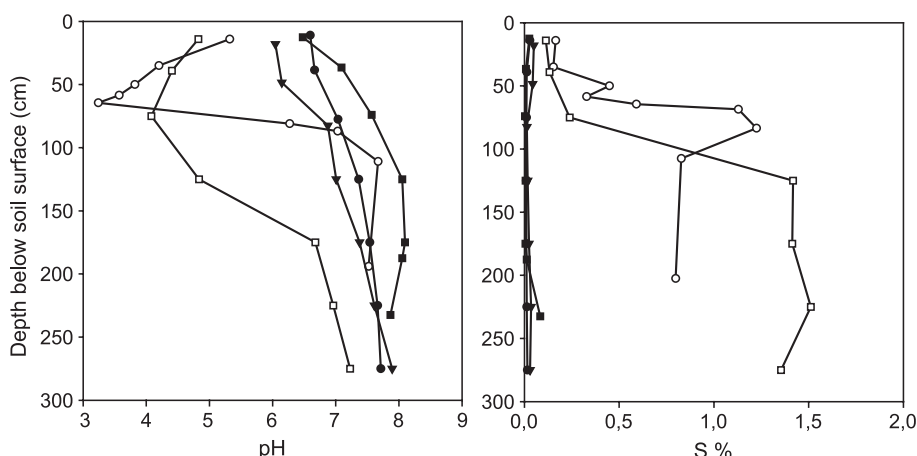


Fig. 1. The pH and total contents of S from six soils developed in clay sediments. The acid sulphate soils formed in gytja clay are shown with white symbols and the soils formed in sulphide-free clays with black symbols.

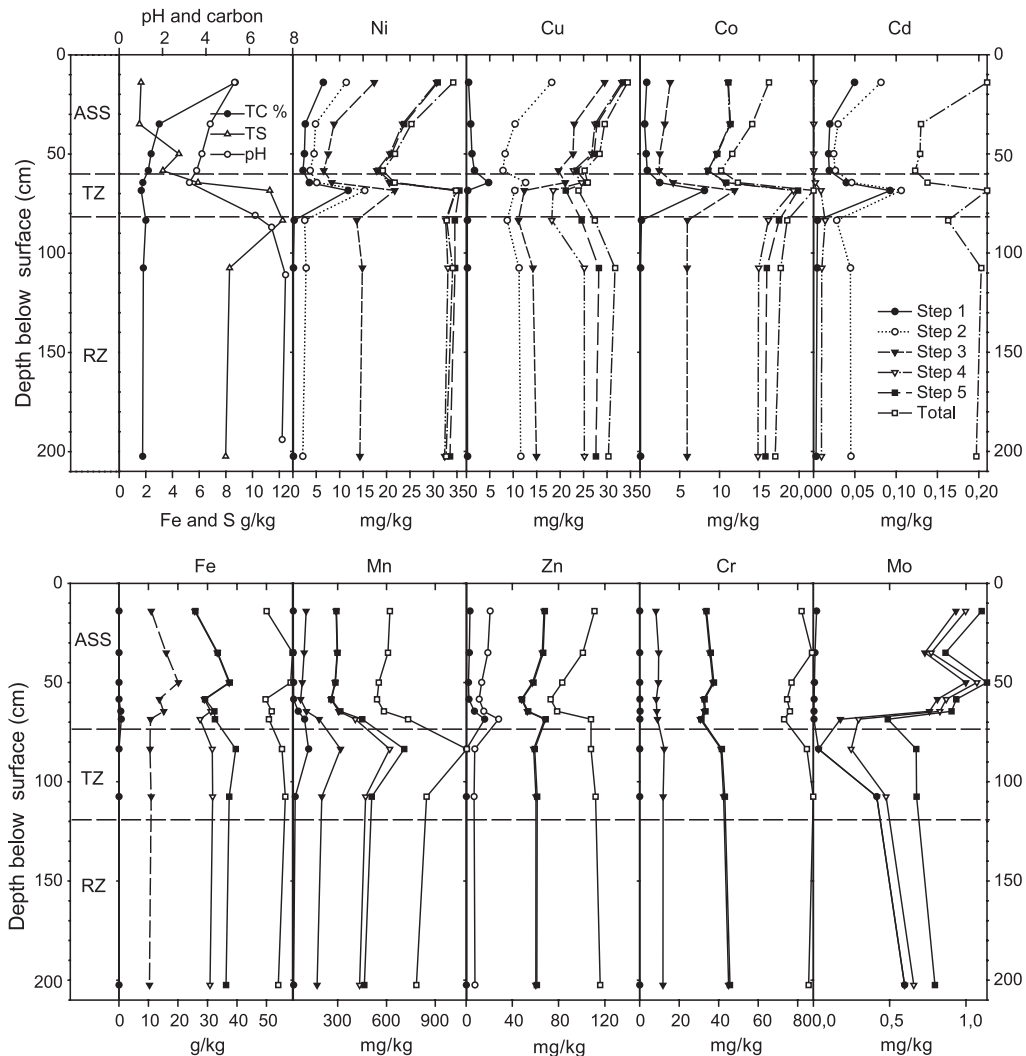


Fig. 2. Depth distribution of pH, tot-C and -S, Ni, Cu, Co, Mn, Fe, Cr Mo and Cd in an acid sulphate soil profile from Hacksta, close to Lake Mälaren in Central Sweden. Steps 1 through 5 show results from sequential leaching and are cumulative.

3.2. Partitioning of trace metals before and after sulphide oxidation

In the most easily extractable fraction (step 1), the highest concentrations of all elements were detected in the transitional zone. For Cr and Fe, Step 1 is only a small fraction of the total content, whereas for Cd Step 1 is the most important source in TZ. That is also reflected in a peak in the total Cd content in TZ.

In ASS, there is less Cd in the organic fraction (Step 2) than in underlying reduced sediments. The

other elements analysed in Step 2 (Zn, Cu and Ni) do not show any significant differences between ASS and RZ.

In ASS, Fe, Mo and Cu show relatively high contents in Step 3, indicating that these elements are bound in oxides, which were formed in this zone. One sample from Mustasaari was taken from the iron oxide crust, which covers cracks in ASS. Iron in Step 3 from that sample is high (4%), which shows that iron oxides are dissolved in that step. In RZ Co, Mn and Ni show higher contents in “oxides” (Step 3) com-

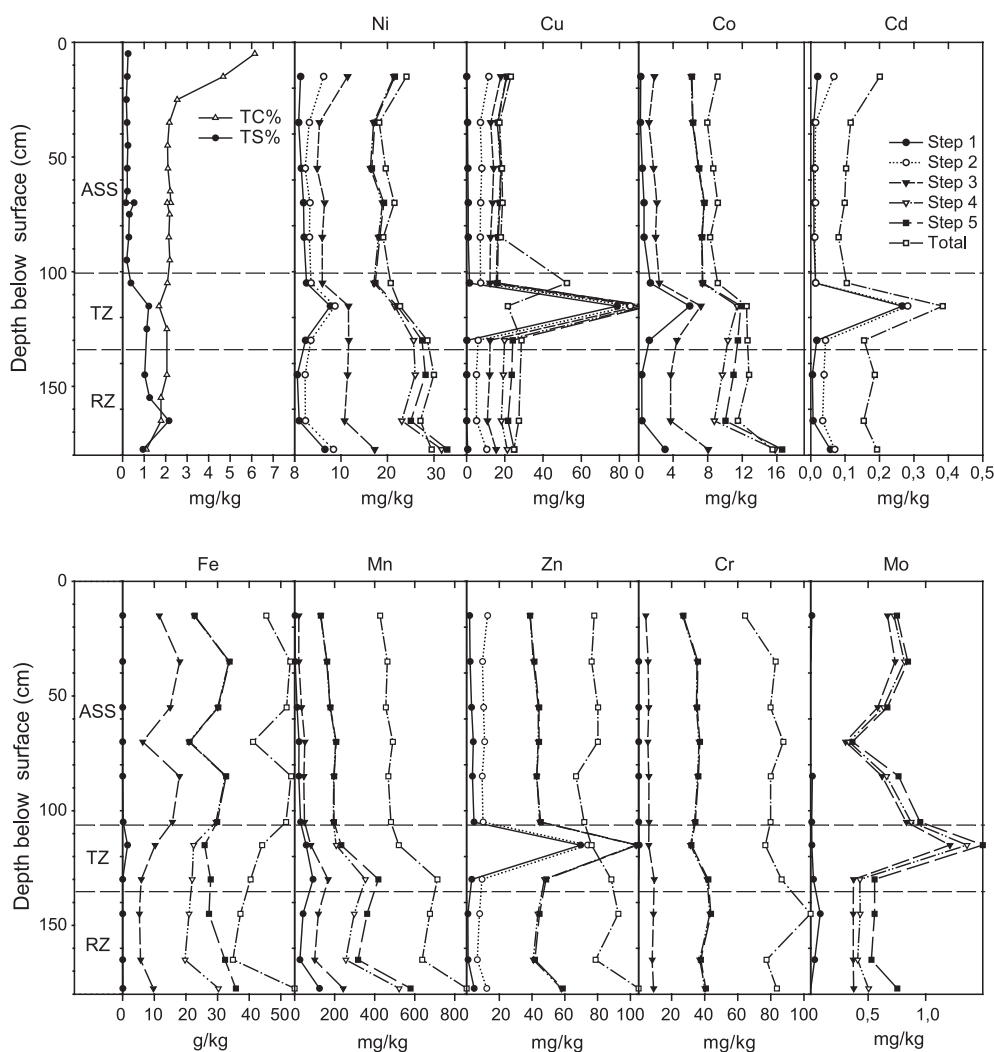


Fig. 3. Depth distribution of tot-C and -S, Ni, Cu, Co, Mn, Fe, Cr Mo and Cd in an acid sulphate soil profile from Mustasaari, close to Vaasa in Finland. Steps 1 through 5 show results from sequential leaching and are cumulative.

pared to the oxidised ASS. Since oxides are unlikely to occur in RZ, these elements were probably extracted in Step 3 due to the low pH (3.25) of that leachate.

In Step 4, Ni, Mo, Cu and Mn occur in lower contents in ASS compared to underlying RZ.

In RZ Ni, Mn, Co and Cu occur in pyrite (Step 5) and are consequently mobilised during oxidation. Cd, Cr and Zn are absent in pyrite.

In one sample from Mustasaari (Zn and Cu in TZ), the sum of the sequential leachings is higher than the

total content. However, the total and sequential leachings were carried out on parallel samples and the discrepancy may be due to heterogeneity in the soils.

3.3. Iron sulphides—content and forms

In the three sulphidic samples from Hacksta analysed for AVS, only a small fraction of the sulphur emanated from FeS (1.4%, 1.3% and 2.2%). However, in the fen 12–17% of the sulphur emanated from FeS in the two uppermost samples (7–11 and 11–15 cm).

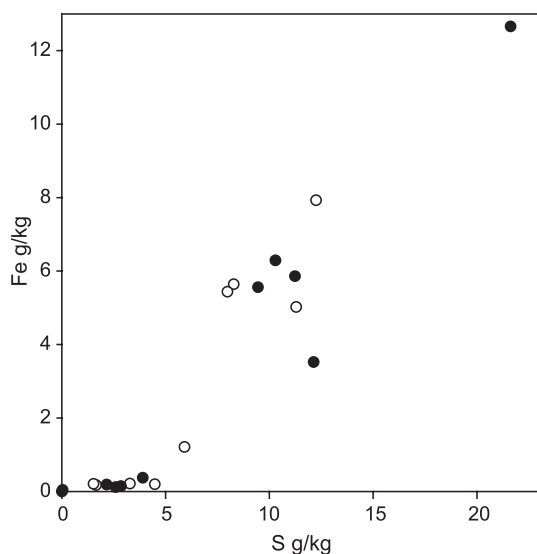


Fig. 4. Fe in pyrite (step 5) and total sulphur. The white and black symbols represent samples from Hacksta and Mustasaari, respectively.

These two samples had a total S content of 0.12% and 0.15%, whereas underlying sediments had a total S content of ca. 1% and contained less AVS-S. In RZ, there is a correlation between Fe in pyrite (Step 5) and total S. Calculations show that at least 70% of the S emanates from pyrite (Fig. 4). It could, therefore, be concluded that pyrite is the most important sulphide carrier in these sediments. It is possible that some of the Fe from pyrite was not included in Step 5, which would underestimate the pyrite content. Some of the B-horizon samples (Fig. 3) contained sulphur but almost no Step 5 iron.

4. Discussion

Sulphide-containing clays were not found to have higher contents of Cu, Mn, Co, Ni and Cd than sulphide-free clays in neighbouring areas. The high concentration of these elements in drainage water from ASS (e.g. Åström and Björklund, 1995, 1996) is therefore presumably due to high mobility in the soil rather than high sediment trace element contents.

The results presented in Table 2 show that Ni and Cd have been leached out of ASS due to the acid

conditions. Manganese seems to be leached out of both ASS and sulphide-free clays. The reason for the high degree of leaching of Mn from neutral soils is not known. However, earlier investigations have shown that the Mn concentration in stream water from ASS areas is high compared to areas with sulphide-free clays (Åström and Björklund, 1995, 1996; Andersson et al., 1988). That indicates a higher mobility of Mn in ASS than in other clay soils. Furthermore, the results presented in Figs. 1 and 2 show clearly that most elements (e.g. Mn) have been leached out from the ASS. Cr and Fe seem to be less affected by mobilisation.

The results from the sequential extraction (Step 5) show that all the elements measured except Zn, Cr and Cd are incorporated in pyrite. However, the oxidation of pyrite could not explain the magnitude of mobilisation of metals from the oxidised horizons. Increased weathering of other minerals due to the low pH must contribute to the leaching. Morse and Luther (1999) have shown that Zn and Cd are not incorporated into pyrite but may form discrete sulphide minerals, while Cr does not react with sulphide. Cd and Zn are probably mobilised from ASS due to the low pH or partly due to the oxidation of discrete sulphide minerals. In RZ, where pH is high, certain elements occur in high contents in Steps 3 and 4. Both these leachates are acidic and the lower Step 3 and 4 content of these elements in ASS may therefore be due to the acid conditions in that horizon. All this supports the concept of mobilisation of certain elements from ASS due to increased weathering caused by acid conditions.

An earlier investigation (Öborn, 1991) showed that the clay mineralogy has changed due to the weathering in ASS.

The mobilisation of Cd from ASS is not only caused by weathering of minerals, however, but also by but release from binding associated to organic matter (Step 2).

There are several possible reasons for these differences in mobilisation between elements.

Cu and Fe have probably been weathered in ASS but the limited mobilisation from ASS is likely to be the effect of precipitation in oxides (Step 3). Mo was also precipitated in oxides but, since the total content was not analysed, it is not known if this element is mobilised from ASS. Cu and Mo levels are not

enhanced in the Fe-oxide layer from Mustasaari, which implies that these elements are not bound in the same oxides.

Most Cr is probably bound in minerals that are hardly weathered, which is shown by the fact that a large proportion of this metal was only leached by the total digestion.

In TZ Cr, Fe and especially Cu show peaks in the most mobile fraction. This indicates that these elements are also mobilised, although not in such quantities that it is reflected in the total contents. Furthermore, Fe-oxide clogging in drainage pipes from ASS shows that this metal is mobilised (Palko, 1994; Mácsik, 1999).

The Step 1 peak is situated close to the groundwater table. The elements in the peak were probably transported down from the oxidised zone by water percolating through the cracks in ASS. Metals from these peaks can probably be further transported in soil cracks and drainage pipes to surrounding streams. This is shown by the high concentrations of, e.g. Ni and Mn in drainage and stream waters from areas with ASS (Andersson et al., 1988; Åström and Björklund, 1995, 1996).

Pyrite is the most important S carrier at the site investigated (Hacksta). The two uppermost samples from the fen contained less total sulphur and more FeS than the other sulphidic samples. A fluctuating groundwater table probably causes that. When the water is low the sulfides are oxidised and sulfate is leached out of the soil. When the water is high, reducing conditions are established and FeS is formed. Sediments in which a large proportion of the sulphides is bound in FeS are often coloured black. This is not the case in the gytja clays investigated here (except the samples from the fen) and it is therefore suggested that FeS₂ is the most important sulphide mineral at all the sites investigated. This is in contrast to Northern Sweden where the sediments are coloured black by FeS (Georgala, 1980).

Iron, sulphur or organic material may limit sulphide formation (Berner, 1984). Manganese is only incorporated into pyrite when the Fe available for pyrite formation is low (Huerta-Diaz and Morse, 1992). Mn is present in the pyrite from the sediments investigated here, which implies that iron rather than sulphur and organic matter was the limiting factor during sulphide formation.

It has been shown that pyrite in sediments deposited at anoxic bottoms is enriched in Mo (up to 100 mg/kg in Step 5; Sternbeck et al., 2000). The sediments investigated here show no such enrichment, which suggests deposition at oxic bottoms (Sternbeck et al., 2000). Furthermore, in the sediments investigated here the Cd content in pyrite is not enhanced, which might be in sediments deposited at anoxic bottoms (Sternbeck et al., 2000). That suggests that the pyrite in our samples was formed below an oxic sediment–water interface after sediment deposition.

5. Conclusions

In samples from Sweden and Finland, the trace element contents are not found to be higher in sulphidic clay sediments than in clays, which lack iron sulphides.

There is an intense leaching of Cd and Ni from ASS compared to soils formed in clay sediments with no iron sulphides.

Cd, Ni, Mn, Co, Zn and to some extent Cu are mobilised from ASS whereas Cr and Fe are only mobilised to a limited extent.

Ni, Co, Mn, Mo and Cu are partly bound in pyrite and are mobilised from ASS after oxidation of that mineral. However, the most important factor causing mobilisation from ASS is increased weathering due to low pH.

Cu, Mo and Fe were found to be bound in the oxides formed in ASS, which decreases the mobility of these elements.

In ASS, elements are able to move rapidly in soil cracks down to the groundwater table, in which the contents of most easily leached elements are shown to be enhanced.

The analytical results also indicate that the gytja clays were deposited at oxic bottoms and the iron sulphides were formed in the sediment after deposition.

Acknowledgements

The Swedish Geological Survey (SGU) financially supported this study. Pål Andersson and Kent

Andersson assisted during fieldwork. Gunilla Lundberg carried out many of the laboratory analyses.

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