Sediments as indicators of wetland efficacy in ameliorating acid mine drainage

by D. Limpitlaw*

Synopsis

As coal mining often results in the generation of acid mine drainage (AMD), careful planning and management of the water on such mines is required. A colliery in Mpumalanga recently sponsored independent research into the pollution-ameliorating potential of a natural wetland that has been receiving AMD for at least two decades. Samples of the sediment collected in the wetland were subjected to nine stages of sequential extraction in a determination of the distribution of the metal species present. The samples were then analysed for aluminium, iron, and manganese. The concentrations of sulphate-reducing bacteria (SRB) were also determined.

The SRB concentration was found to increase with distance downstream (away from the source of pollution). An analysis of the sediment speciation data indicated that metal immobilization may be optimal in areas where the organic adsorption of metals is at its greatest. The analysis also provided evidence of the biogeochemical improvement of the water quality as the AMD passed through the wetland environment.

The metal species present did not correlate strongly with the SRB populations; nor did the soluble species correlate with the species formed over long periods of time.

Introduction

The accelerated decline in environmental conditions over the past quarter of a century has resulted in ever-increasing environmental awareness amongst members of the public. In the mining sector, this has translated into increased emphasis on environmental management. In particular, the deleterious nature of coal, with its high acid-generating potential, requires careful control and management.

Traditional approaches to the amelioration of acid mine drainage (AMD) consists in the passing of polluted water through a plant where a source of alkalinity is added to the water. This raises the pH value of the water and reduces the concentrations of many toxic substances, particularly heavy metals (which are more soluble at lower pH values). Water-treatment plants such as these entail high capital and operating costs, and their efficacy requires regular monitoring.

During the past decade, researchers in the U.S.A. and elsewhere have been reporting on the use of both natural and constructed wetlands for the control of AMD. Many of these reports indicate that such passive systems are capable of improving water qualities. In South Africa, the track record has not been as distinguished. Few of the systems installed here have improved water qualities adequately, leading to a general mistrust of wetland technology[1].

Early attempts to harness the pollution amelioration potential of wetlands focused on the moss *Sphagnum*. Today many researchers believe that the primary medium for the removal of metals is associated with the sulphate-reducing bacteria encountered in the reducing environments of wetland substrates[1–4].

At a colliery in Mpumalanga, a natural wetland has been receiving AMD for the past two decades. A study was initiated on whether the system was improving the water qualities and whether any optimization was possible. This paper is concerned with one aspect of the study, namely the chemistry of the wetland sediment as affected by the presence of AMD.

Methodology

Sampling

Water qualities through the wetland were monitored for a period of twelve months, transects having been established across the wetland to facilitate sampling (Figure 1).

Samples of the sediment were collected in the odd-numbered transects in the wetland by use of a perspex corer 10 cm in diameter. Although more difficult to use than an auger-type sampler, this device minimized disturbance of the samples. The corer was pushed into the wetland sediment to a depth of approximately 20 cm. Four horizons were sampled, each occurring sequentially at 5 cm intervals below the sediment–water interface. The uppermost

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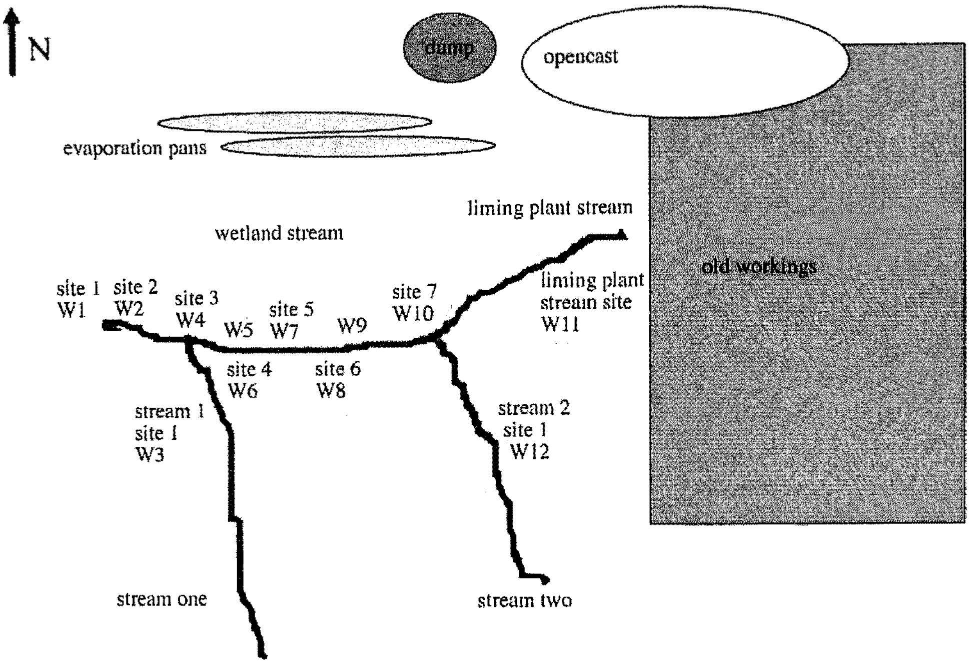

Figure 1—Layout of sampling sites in the wetland

sample was labelled 1 and so on to the lowest sample, which was labelled 4. Sediment was collected over a two-day period and then taken to the Agricultural Research Council, Pretoria, for analysis. Samples were subjected to 9 stages of sequential extraction, each designed to extract a more strongly bound species. Extractions were conducted for aluminium, iron, and manganese.

Extractions

The following laboratory procedures, reported by Murray and Jooste[5], were employed.

1. *Soluble fraction.* The sample is shaken for 16 hours with 20 ml of water. All the easily soluble components are dissolved by the solvent (distilled water). The simple inorganic salts, most probably formed when the sample was dried, are determined. Examples include $FeSO_4$, $MnSO_4$, and $CaSO_4$ (gypsum).
2. *Neutral-salt exchangeable fraction.* The sample is shaken with 20 ml of 0.5 M $Ca(NO_3)_2$ for 16 hours. The cations adsorbed onto solid surfaces are replaced by Ca^{2+} as a result of an ion-exchange reaction. Species such as Mn^{2+}, Fe^{2+}, and $FeOH^+$, which are adsorbed onto surfaces more weakly than Ca^{2+}, are likely to be determined.
3. *Pb-displaceable fraction.* The sample is shaken with 20 ml of 0.05 M $Pb(NO_3)_2$ and 0.1 M $Ca(NO_3)_2$ for 16 hours. In this stage, the cations adsorbed onto solid surfaces are replaced by Pb^{2+} in an ion-exchange reaction. The species that are adsorbed less strongly than Pb^{2+} are determined, including Mn^{2+}, Fe^{2+}, and $FeOH^+$.

   Stages 2 and 3 determine the difference between the strongly and the weakly adsorbed species.
4. *Acid-soluble fraction.* The sample is shaken with 20 ml of 0.44 M CH_3COOH and 0.1 M $Ca(NO_3)_2$ for 8 hours. Carbonates, poorly crystalline metal hydroxides, and amorphous FeS are dissolved. The species likely to be determined include $FeCO_3$, $MnCO_3$, $Mn(OH)_2$, FeS and, possibly, amorphous precipitates of FeO.
5. *Mn-oxide occluded fraction.* The sample is shaken with 20 ml of 0.01 M $NH_2OH{\cdot}HCl$ and 0.1 M HNO_3 for 30 minutes. The manganese oxides are dissolved in this stage, with the subsequent release of particles cemented together or enclosed by manganese oxides. The iron oxides do not dissolve. The manganese oxides such as MnOOH and MnO_2, as well as any occluded species that would otherwise have been determined in stage 4, are likely to be determined. The iron species that may be determined include those released by dissolution of the surrounding manganese oxides, which would also have been determined in stage 4 if they had been available.
6. *Organically bound fraction.* The sample is shaken with 20 ml of 0.1 M $K_4P_2O_7$ for 24 hours. The organic material is dissolved during this stage. All the cations previously taken up by plants or bound to humic substances are released. The manganese and iron bound in plant cells (soluble or bound to organic ligands) and the metals complexed to humic substances are released.
7. *Amorphous Fe-oxide occluded fraction.* The sample is shaken with 20 ml of 0.175 M $(NH_4)_2C_2O_4$ in darkness for 4 hours. The amorphous iron oxides are dissolved, releasing the iron in this form, as well as any other encapsulated particles.
8. *Crystalline Fe-oxide occluded fraction.* The sample is shaken with 20 ml of 0.175 M $(NH_4)C_2O_4$ and 0.1 M $H_2C_2O_4$ at 85°C under ultraviolet irradiation for 3 hours. The crystalline iron oxides are attacked, permitting the determination of iron forms such as goethite (α-FeOOH), hematite (α-Fe_2O_3), and maghemite (γ-Fe_2O_3).
9. *Residual fraction.* The sample is shaken with 1 ml of aqua regia (1 part of concentrated HNO_3 to 3 parts of concentrated HCl) and 10 ml of hydrofluoric acid (HF)

at 110°C for 2 hours in a Parr digestion bomb. All the remaining solid matter is now dissolved, and the clay minerals, quartz, magnetite, and any undissolved iron and manganese silicates are determined. This extraction represents long-term immobilization of the iron.

Bacteria

Most metals in AMD can be removed when the environmental prerequisites for sulphate-reducing bacteria (SRB) are met. These include the presence of carbon in a form suitable for bacterial assimilation[6], anaerobic conditions, the occurrence of suitable attachment points (as provided by hydrophitic vegetation), and the presence of a source of sulphate.

SRB in the wetland sediments

Two horizons within the wetland were sampled for the presence of SRB. The upper horizon extended from the soil–water interface to a depth of 20 cm (0 to 20 cm). The lower horizon began 15 cm below the upper and extended downwards for a further 15 cm (35 to 50 cm). Samples were collected with an auger type of sampler. The analyses were carried out to a dilution series limit of 10^3, and the presence of SRB was reported according to the following simple scale:

1. Negligible
2. Positive
3. Substantial
4. Greater than 10^3.

The analyses were carried out by Mattek at the Council for Scientific and Industrial Research, Johannesburg.

For both horizons, the lowest concentrations of SRB occurred just before the confluence with stream 1 (site 3). High concentrations for both horizons coincided further upstream at site 5, although the lateral variation was greater for the upper horizon, with values increasing towards site 5a (on the side of the wetland closest to the mine). The maximum presence of SRB was recorded across this transect for the lower horizon.

The presence of SRB in wetland sediments is an indicator of conditions suitable for bacterial sulphate reduction. These bacteria require organic carbon, suitable points of attachment, and a source of sulphate. Where any of these requirements are not satisfied, the bacterial counts will be low.

Results

Average metal accumulation over the wetland

The concentration of each of the three metal species determined in the fractions described previously was expressed as a percentage of the total concentration of that metal in the sample.

Aluminium

The bulk of the sediment aluminium (70%) occurred in the residual fraction (Figure 2). The only other significant form of aluminium was the crystalline Fe-oxide occluded fraction (18%). The amorphous Fe-occluded and organically bound fractions both contained an average of 3% of the aluminium in the system. The remainder of the metal was distributed between the other five fractions.

Iron

The iron generally occurred in the residual fraction (43%), although significant amounts were also present in the crystalline Fe-oxide occluded (31%), amorphous Fe-oxide occluded (10%), and organically bound (11%) fractions. The formation of pyrite as predicted by Hedin *et al.*[3] was determined in the acid-soluble extraction. There is not a major iron sink in the wetland (Figure 3).

Manganese

Manganese was the only metal to demonstrate noticeable accumulation in the first two fractions: 4% of the sediment manganese was contained in the soluble fraction, and 15% in the exchangeable fraction (Figure 4). The residual and crystalline Fe-oxide occluded fractions were high (55% and 15% respectively).

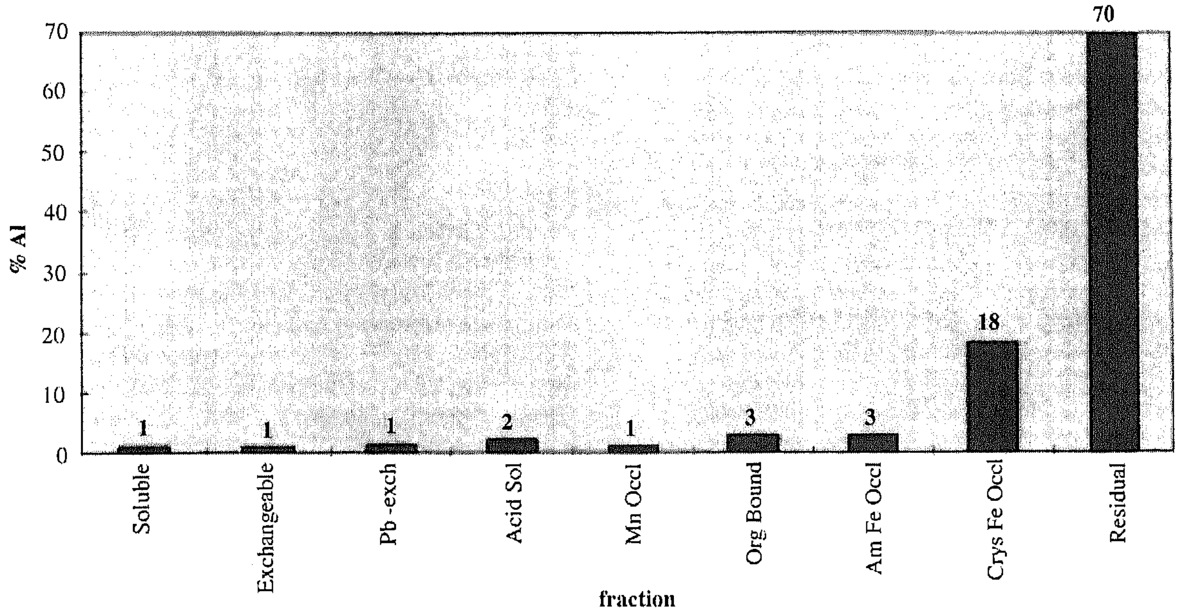

Figure 2—The average distribution of sediment aluminium

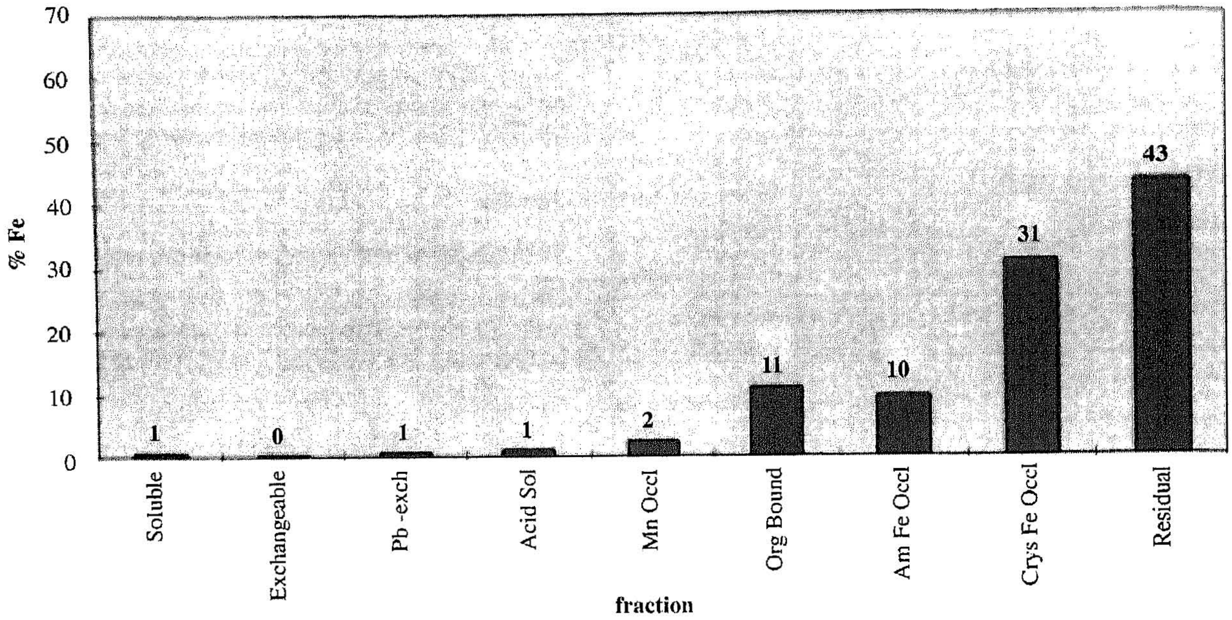

Figure 3—The average distribution of sediment iron

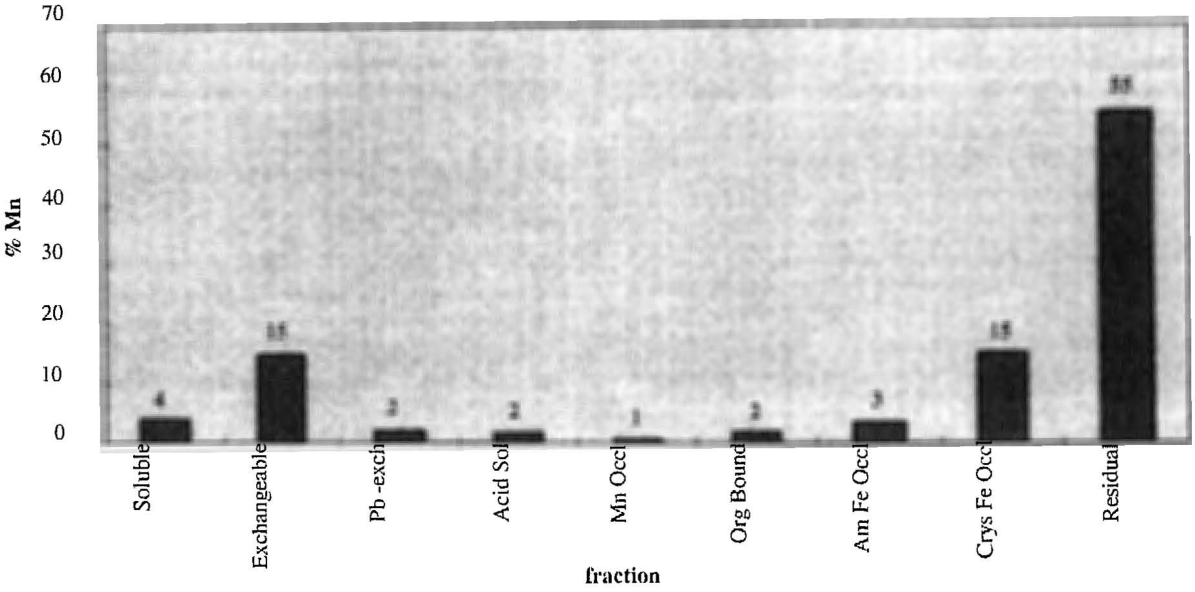

Figure 4—The average distribution of sediment manganese

Average accumulation at each transect

Aluminium

The transect averages did not differ radically from the pattern of aluminium accumulation identified in the values averaged over the entire wetland (Figure 5). The only noticeable differences were elevated accumulations in the organically bound fraction at site 1 (6%) and in the amorphous Fe-oxide occluded fraction at site 7 (6%).

Iron

The transect averages for iron accumulation were more variable. For most of the system, the residual iron fraction was dominant, except at the confluence of stream 2 and the liming plant stream (site 7) and in the middle of the wetland at site 5 (Figure 6). At site 7, most of the iron occurred in the crystalline Fe-oxide occluded fraction (55%) and, at site 5, 40% of the sediment iron was in that form. Organically bound iron generally accounted for between 10 and 19% of the metal but, at site 7, only 5% of the iron occurred in that form. The iron in stream 2 occurred in the Mn-oxide occluded form to a greater degree than elsewhere (7%).

Manganese

The residual manganese was lowest at site 7 (39%) and greatest at the final transect, site 1 (69%) (Figure 7). The crystalline Fe-oxide occluded fraction was greatest at site 7 (29%), and the exchangeable fraction greatest at the site of a freshwater seep, site 3 (27%).

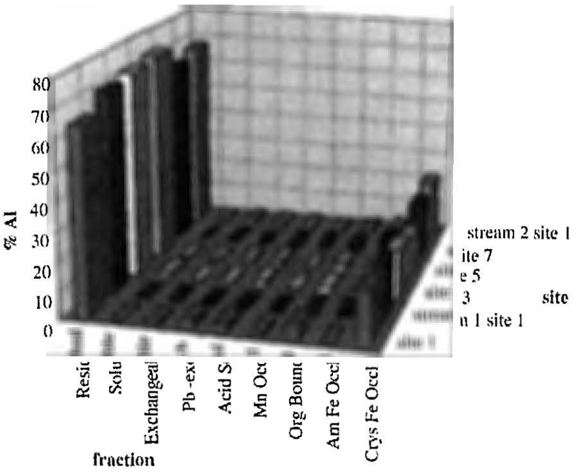

Figure 5—The distribution of sediment aluminium by transect

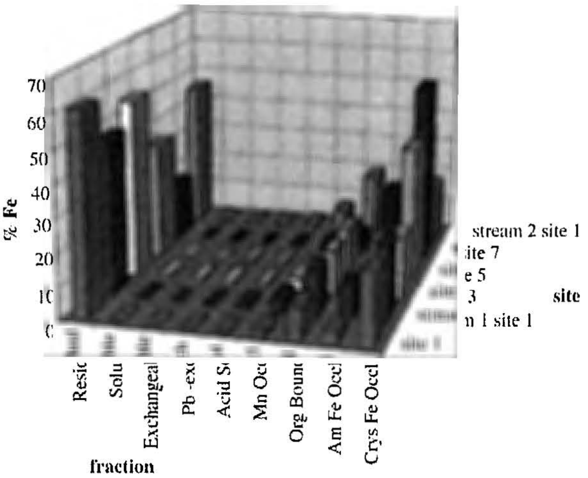

Figure 6—The distribution of sediment iron by transect

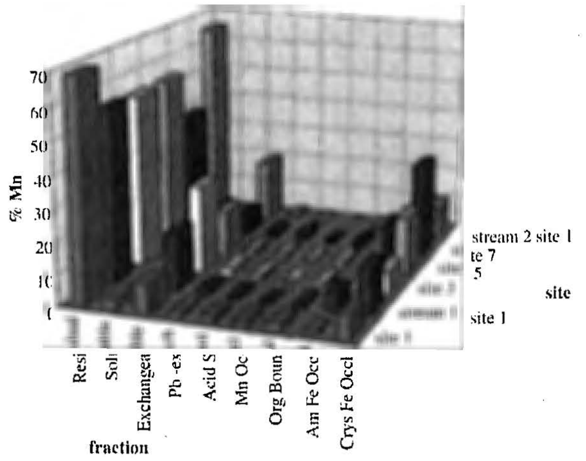

Figure 7—The distribution of sediment manganese by transect

Although the differences in the mineralogical characteristics were not quantified, the percentage of iron and manganese in the residual fraction was low at sites with poor water quality (such as W10* at site 7), and where in the iron oxide fraction was high. At sites with better quality water, such as W3† (site 1) in stream 1, the residual fraction was dominant. Based on this observation, the sediment speciation data for site 1, the final transect in the wetland, indicated a change in the chemical regime of the sediment when compared with that at site 7, at the beginning of the system. Biogeochemical processes had thus acted to alter the chemical characteristics of the wetland's water in contact with the sediment.

The species composition at most sites corresponded closely to the average for the wetland. The exceptions were at sites 1, 3, and 7. This can be explained in terms of the locations of these sites.

- Site 7 was at the beginning of the system, where two streams of different qualities mixed. This fact is demonstrated by the elevated mass of aluminium occurring in the amorphous Fe-oxide occluded fraction. Iron at this site occurred predominantly in the crystalline Fe-oxide occluded fraction, and there was more manganese in this last-mentioned fraction than at any other point in the system.
- Site 3 contained a freshwater seep, a fact that seemed to influence the composition of only the manganese species. At that point, the exchangeable fraction was larger than at any other point in the system, which is possibly indicative of the removal of metals from the polluted wetland water as it comes into contact with the unpolluted seep water.
- Site 1 occurred at the end of the system. Modelling of the water speciation showed that the wetland water was most stable at that point[7]. Greater amounts of aluminium occurred in the organically bound fraction than elsewhere in the system.

Correlation of metal species

The mechanisms controlling the distribution of metal in the wetland sediments remain unclear. The data on sediment speciation were subjected to a simple covariance test to determine whether there are any relationships between the various species of each metal. The test statistic (from the Cambridge Tables, table no. 13) was ±0.3075 at a 1% level of confidence ($N = 57$, $\nu = 55$). The values of r (the correlation coefficient) within this range implied that there was no correlation between the variables being compared (Table I).

Some understanding of the thermodynamic properties of various species is necessary for an interpretation of the correlation data. Using a speciation model (JESS), Murray and Jooste[5] determined the rate at which various compounds form in wetland sediments. They divided the compounds into two categories: those forming in the short term (minutes to days), and those forming in the medium term (days to decades). A third possible category, minerals and compounds forming over longer periods, is not discussed here.

The following solids can be expected to form in the short term[5]:

- ferrihydrite ($5Fe_2O_3 \cdot 9H_2O$), determined in the soluble fraction

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* The mean determinations at W10 over the 12-month period of water sampling were as follows: pH 4, electrical conductivity: 6 mS/m.

† The mean determinations at W3 over the 12-month period of water sampling were as follows: pH 5, electrical conductivity: 126 mS/m.

Table I

Correlation coefficients

Species compared	r (Al)	r (Fe)	r (Mn)
Amorphous Fe-oxide occluded / acid soluble	0.9102	0.5505	0.6657
Amorphous Fe-oxide occluded / crystalline Fe-oxide occluded	0.7742	0.6213	0.4745
Amorphous Fe-oxide occluded / Mn-oxide occluded	0.5527	0.5747	0.6455
Amorphous Fe-oxide occluded / Pb displaceable	0.2786	0.6981	0.6422
Crystalline Fe-oxide occluded / acid soluble	0.9011	0.3931	0.3458
Crystalline Fe-oxide occluded / Mn-oxide occluded	0.6436	0.7058	0.3798
Crystalline Fe-oxide / Pb displaceable	0.2597	0.7199	0.3078
Neutral salt exchangeable / acid soluble	−0.1748	0.7937	0.6311
Neutral salt exchangeable / amorphous Fe-oxide occluded	−0.2308	0.1580	0.2408
Neutral salt exchangeable / crystalline Fe-oxide occluded	−0.0930	0.2837	0.4382
Neutral salt exchangeable / Mn-oxide occluded	−0.0867	0.4756	0.5491
Neutral salt exchangeable / Pb displaceable	0.4683	0.6067	0.7189
Organically bound / acid soluble	0.8056	0.8597	0.6420
Organically bound / amorphous Fe-oxide occluded	0.8145	0.5636	0.5148
Organically bound / crystalline Fe-oxide occluded	0.5911	0.7083	0.6720
Organically bound / Mn-oxide occluded	0.6717	0.7319	0.8332
Organically bound / neutral salt exchangeable	−0.1031	0.6588	0.4461
Organically bound / Pb displaceable	0.6517	0.8462	0.5086
Soluble / acid soluble	−0.1740	0.5793	0.8646
Soluble / amorphous Fe-oxide occluded	−0.1901	−0.0311	0.1808
Soluble / crystalline Fe-oxide occluded	−0.0427	0.2243	0.4397
Soluble / Mn-oxide occluded	−0.1616	0.2581	0.8793
Soluble / neutral salt exchangeable	−0.0356	0.6593	0.5358
Soluble / organically bound	−0.2272	0.4840	0.6515
Soluble / Pb displaceable	−0.1702	0.3824	0.8023

- goethite (α-FeOOH), determined in the crystalline Fe-oxide occluded fraction[*]
- amorphous iron oxides and sidderite ($FeCO_3$), determined in the amorphous Fe-oxide occluded and acid-soluble fractions
- oxides of manganese (MnO_2), determined in the Mn-oxide occluded fraction
- amorphous iron sulphides, determined in the acid-soluble extraction.

In addition, species determined in the neutral-salt exchangeable and Pb-displaceable fractions form in the short term.

Solids forming over longer periods can be expected to form in the medium term[5]:

- jarosite ($KFe(OH)_5(SO_4)_2$)
- hematite (α-Fe_2O_3), determined in the crystalline Fe-oxide occluded fraction
- maghemite (γ-Fe_2O_3), determined in the crystalline Fe-oxide occluded fraction
- pyrite (FeS_2)
- oxides of manganese (MnO_2), determined in the amorphous Fe-oxide occluded fraction.

In this wetland system, the species determined for aluminium behaved differently from those determined for iron and manganese. The author initially ascribed this difference to the high concentration of aluminium in the clays occurring through the wetland. The correlation of the other aluminium species with the residual aluminium[†] was poor, with coefficients ranging from 0.2 to 0.4. Only the crystalline Fe-oxide occluded and the soluble fractions correlated with the residual aluminium, and thus the aluminium content of the clay minerals did not control the distribution of most of the aluminium species. The seemingly inexplicable correlation of the short- and long-term species should be regarded as a caveat in the interpretation of the results of this analysis.

The soluble fraction consists of species precipitated from sediment water during the drying of the sediment samples. These precipitates are thus indicative of the water qualities of the sediment and represent very recently formed metal species. It is therefore unlikely that they would correlate with species formed over long periods of time, such as those determined during the crystalline Fe-oxide occluded extraction. This assertion is based on the fact that the system is not in equilibrium and has been subjected to an increasing pollution load over the past four years[*]. The soluble extraction for aluminium did not correlate with that for any other species. The soluble iron species did not correlate with the Mn-oxide occluded, the amorphous Fe-oxide occluded, or the crystalline Fe-oxide occluded fractions. The soluble manganese fractions correlated with all the other manganese species except the amorphous Fe-oxide occluded. The lowest correlation coefficient generated for the soluble manganese fraction was for the crystalline Fe-oxide occluded fraction (0.4). This correlation appears to support the above assertion.

The neutral salt exchangeable and the Pb-displaceable fractions were a measure of the quantities of metal adsorbed onto surfaces. The high correlation between these two fractions for all three metals indicated that the fractions can be grouped together for the purpose of modelling (the two

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[*] Although goethite forms in the short term, the crystalline Fe-occluded fraction is assumed to be generally representative of medium-term species.

[†] The residual aluminium was treated as if it represented sediment mineralogy (clays).

[*] The increasing pollution load was confirmed by an inspection of historical records of water quality and by ongoing water monitoring.

fractions showed differing degrees of adsorption). The correlation coefficients for these fractions were 0.5 for aluminium, 0.6 for iron, and 0.7 for manganese. If these correlated with the soluble fraction, it could be argued that the most adsorption occurs in sediments where the quality of the sediment water is poor. This assertion is partially validated by the correlation factors in the sediment data. The data for the iron and manganese fractions correlated well, but not those for the aluminium fraction.

The iron sulphides present in the substrate were determined in the acid-soluble fraction. The manganese determined by this extractive technique is likely to occur in the carbonate form[5]. For aluminium, this fraction correlated very well with the two Fe-oxide occluded fractions (correlation coefficients of 0.9), while the manganese acid-soluble fraction correlated to this degree only with the soluble fraction ($r = 0.9$). For iron, only the acid-soluble and organically bound species had coefficients of 0.9. Thus, despite the fact that the iron and manganese acid-soluble fractions correlated with all the other fractions, it is tempting to conclude that the formation of iron sulphides in the wetland sediment is greatest in the areas of high organic adsorption.

The organically bound fraction represents metals that have undergone organic physicochemical binding. For iron and manganese, this fraction correlated with all the other fractions. For aluminium, only the soluble and neutral-salt exchangeable fractions did not correlate with the organically bound species. The correlation between the organically bound fraction and the two Fe-oxide occluded fractions suggests that organic binding is an important process in the long-term immobilization of metals*. The same can be said for the adsorption fractions.

Correlation of metal species with total carbon and SRB in the sediment

Coefficients were calculated for the correlation of the three metal species with the carbon and SRB. In order to simplify the analysis, the total metal species at each site were used (i.e. the four horizons were added together and averaged). The total sediment carbon was expressed as a percentage of the sediment by mass for each horizon, and thus an average value was used for each site. The SRB were not sampled at the same depth intervals as the metals. The upper SRB sample was taken from the same horizon of the wetland sediment (0 cm to approximately 20 cm below the soil-water interface) as those for the metals. The correlation was calculated for this sample and for the total SRB sample, which included a sample taken from 35 cm to 50 cm below the interface. Each population consisted of 15 samples ($N = 15$ and $\nu = 13$).

Most of the determinants did not demonstrate a significant degree of correlation when compared with the SRB population or the total carbon present.

- The crystalline Fe-oxide occluded fraction determined for aluminium correlated with the carbon at a confidence level of 5% ($r = 0.4781$).
- The organically bound fraction correlated with the carbon for all three metals at 2.5%. (Al: $r = 0.6324$, Fe: $r = 0.5877$, Mn: $r = 0.5744$).
- The neutral salt exchangeable fractions determined for the aluminium and iron correlated with the carbon at the 5% level (both $r = 0.5312$).
- The Pb-displaceable fractions determined for the aluminium and iron correlated with the carbon. The coefficient generated for the aluminium showed the highest correlation of any metal species with the carbon ($r = 0.8344$). This is significant at a 0.1% level of confidence. The iron correlated at a 2.5% level of confidence ($r = 0.6338$).

These correlations underscore the importance of the organic binding of metals in wetland systems. Organic carbon would have been a more informative measure of this organic material, but facilities for its analysis were not available.

No correlation could be found between the upper and the total SRB sample populations or between any metal species and the total SRB sample population. The crystalline Fe-oxide occluded fraction determined for iron was the only metal species to correlate with the upper SRB sample population ($r = 0.5598$ at 2.5%).

Correlation coefficients were also generated for the concentrations of the various determinants and the Cartesian co-ordinates of their sampling locations. The X-values decreased away from the mine (northern) side of the wetland. The Y-values decreased downstream (east).

- Both SRB sample populations demonstrated a significant correlation with the X-value (SRB total: $r = 0.5378$, SRB upper: $r = 0.5906$; both at 2.5%). Thus, the SRB presence increased with an increase in X-value; more SRB were present on the mine side of the wetland. This may be due to the higher availability of sulphate in the sediments as a consequence of the polluted seepage that enters the wetland from the mine.
- The SRB upper sample population was inversely proportional to the Y-values ($r = -0.5072$ at 5%). Although the total SRB sample population did not demonstrate this correlation at a suitable level of confidence ($r = -0.3063$), it appears that the SRB presence in the wetland decreased as the Y-value increased; thus the SRB increased with the distance downstream.
- The only metal species to show a correlation with either of the Cartesian co-ordinates was the soluble fraction determined for aluminium ($r = -0.5139$ at 5%). This implies that the soluble aluminium concentrations in the sediment increased with the distance downstream.

Conclusions

The following trends were identified.

- The soluble species generally showed no correlation with the species that formed over long periods of time; this is consistent with a system in a state of flux.
- The neutral salt exchangeable and Pb-displaceable fractions correlated well with the soluble fractions; this was expected since species formed in the short term are also measured in these two extractions.
- The formation of iron sulphide in wetland sediments may be linked to the organic adsorption of iron.

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* Organically bound metals may be available to SRBs, which would release iron under reducing conditions, allowing the formation of pyrite[6].

- The concentrations of metal included in the sediments by organic adsorption correlated well with the other species formed in both the short and medium terms; metal immobilization may therefore be optimal in areas of greatest organic adsorption.

The initial samples of surface water indicated that little chemical alteration occurred with distance downstream along the wetland's axis. The analysis of the wetland sediments presented a complex picture of metal binding. A comparison of the fraction ratios along the axis of the wetland indicated a gradual change in the condition of the sediment. Increases in the residual fraction implied that more metal was bound in stable species at the end of the wetland than at the beginning. The wetland was thus immobilizing the pollutants.

More work is required before any definitive statement regarding the efficacy of this system can be made, but it is clear that this wetland, by biogeochemical processes, is altering the chemical composition of the water flowing through it.

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