Dry Covers on Historical Sulphidic Mine Waste – Long Term Statistic Performance Assessment of Surface Water Quality

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Abstract
In 1988 two sub-sections of a historic mine site with sulphidic ore residues (Fe, Cu, Zn, Cd and Pb) at Bersbo, Sweden, were covered with compacted illitic clay and cement stabilised fly ash, respectively. Changes in surface water composition during the period 1985-96, that is before and after the remediation, have been statistically evaluated with data from two adjacent locations (Lake Gruvsjön and the Kuntebo creek), and a downstream lake (Lake Risten). The analysis is focused on R and Q-mode clusters and time trends using concentrations of Al, Cd, Cu, Fe, Mn, Pb, and Zn, as well as pH (as a predictor in time trend). Data from Kuntebo and Lake Risten represent two main Q-mode clusters based on the sampling dates (before and after remediation). The Lake Gruvsjön data from 1989 form a third cluster. Two R-mode clusters are observed in the Kuntebo data, one including Al, Cd, Cu and Pb (1), and the other one including Fe, Mn, Zn and pH (2). Corresponding clusters of Lake Gruvsjön data include: Al, Cd, Cu, Pb, Zn, and Fe (1) and pH (2), respectively. Lake Risten data represent four clusters: Cu, Pb and Zn (1), Al, Cd and Fe (2), Fe (3), and pH (4). The predicted trends are compared with measured concentrations from the period after the remediation in 1988. A general conclusion is that the remediation strategy was effective and successful, based on the statistical analysis of data. The only exception is Fe that did not change significantly after remediation.

Key Words: ARD, sulphidic mine waste, covering, statistical analysis, Fe, Cu, Zn, Cd, Pb

Introduction
Historical sulphidic mine waste is the single largest source of environmental pollution of metals in Sweden. It has been estimated that some 8000 sites are in need for remediation according to national standards. Many of them are found at remote sites with low populations, while in other cases entire cities are built upon them. Several projects aim to determine the most favourable remediation strategy, but until now only a few sites allow for long term performance assessment. Overviews on the conditions after remediation are given by Karlsson and Bäckström (2003, 2005). A statistical evaluation of surface quality changes after remediation in 1988 of a historical sulphidic mine site in Bersbo, Sweden, has been made in the present project, as a follow-up of an extensive monitoring program.

Materials and Methods
The Bersbo site
Bersbo is located some 250 km SSW of Stockholm, Sweden, in the municipality of Åtvidaberg. Mining for copper is documented from the 14th century but probably began earlier. Rational mining was introduced in 1765, and during the peak production in 1850-1870 Bersbo was the largest copper producer in Sweden. Copper was refined in the nearby refinery in Åtvidaberg. The ore that could be extracted with the technology of the time ceased in the late 1800s, and the mining was stopped in 1902. For a more complete description see Allard *et al*. (1987).

The copper ore was mainly magnetic chalcopyrite that was confined to narrow veins together with pyrite, and sphaelerite in a leptite/granite host rock. Karlqvist and Qvarfort (1979) reported that the element concentrations were in the ranges 0.5-3% Cu, 1-3%, Zn, 0.5-1% Pb, around 20% Fe and 25% S. A detailed description of the minearlogy is given by Tegengren (1924). The grain size of the waste ranged from silt to rock, and the waste was put in the immediate vicinity of the shafts. These piles had a hydraulic

conductivity of around 10^{-3} m/s, and consequently, the material was in full contact with atmospheric oxygen and rainwater.
Because of a ridge that runs in north-south direction through the site, approximately half of the drainage from the waste is directed towards the east (the Kuntebo creek, K) and west (Lake Gruvsjön), respectively (Figure 1). There were two major waste piles with altogether some 700,000 m^3, covering 0.2-0.3 km^2. Both piles rest on compact illitic clay that minimises contact with the groundwater (Allard *et al.* 1991).

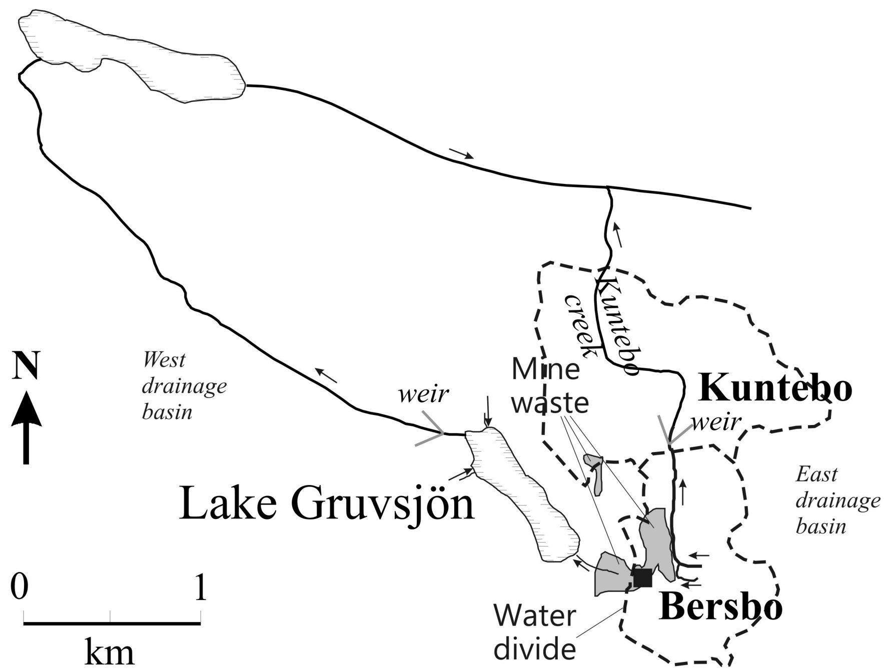

Figure 1. Map of the study area

The two catchments have different hydrological properties. The westerly one reaches the small Lake Gruvsjön after just some 100 m. The lake has an average depth of 8 m and a theoretical residence time of approximately 10 months. The easterly catchment is drained by the small Kuntebo creek (some 1 m wide) with highly variable discharge and retention times. The annual variation at location Kuntebo is from 0 l/sec during the dry summer and some 150 l/sec during intense snow melt. An ordinary late summer shower of 15 mm usually gave an increase from 1-2 l/sec to 30-40 l/sec, depending on original level of the water table. The corresponding flows for the outlet of Lake Gruvsjön are 1 l/sec and 1500 l/sec.
Peak flow occurs during snow melt, usually in April. Total precipitation reaches 650 mm of which some 200 mm forms surface water runoff. A comprehensive presentation of the hydrological conditions before the remediation is given by Sandén *et al*. (1987, 1997).
Rather coarse till dominates the soil, but in lower parts lenses of illitic clay are found. This is also reflected by the groundwater character which is of Ca^{2+}-SO_4^{2-}-CO_3^{2-} type with pH around 7.5 and an alkalinity of 2.5-3 meq/L (Allard *et al*. 1987). The higher vegetation consists of mixed stands with coniferous and deciduous tree species with a shrub layer of mainly *vaccinium* species as well as grasses and mosses on the soil.

Weathering in summary
Oxidative weathering of pyrite, as well as other sulphide minerals, has been summarised by Stumm and Morgan (1996):

$$FeS_2(s) + 3.5O_2 + H_2O \Rightarrow Fe^{2+} + 2SO_4^{2-} \quad (1)$$

$$Fe^{2+} + 0.25O_2 + H^+ \Rightarrow Fe^{3+} + 0.5H_2O \quad (2)$$

$$Fe^{3+} + 3H_2O \Rightarrow Fe(OH)_3(s) + 3H^+ \quad (3)$$

Reactions 1-3 are representative of the weathering pathway before the remediation since there was no limitation of either air or oxygen. The reactions are usually catalysed by bacteria which results in a rate some 10,000 higher than for the abiotic processes.

$$FeS_2(s) + 14Fe^{3+} + 8H_2O \Rightarrow 15Fe^{2+} + 2SO_4^{2-} + 16H^+ \quad (4)$$

$$ZnS(s) + 8Fe^{3+} + 4H_2O \Rightarrow Zn^{2+} + 8Fe^{2+} + SO_4^{2-} + 8H^+ \quad (5)$$

Reaction 4-5 are driven by Fe(III) as the electron acceptor, and presence of air is not required.

Remediation

In 1987-89 the site was remediated by filling all available shafts with mine waste and sealing them as well as putting the remaining material in two piles that were contoured for optimum stability (Lundgren and Lindahl 1991). All exposed soil was limed. The waste on the westerly side was covered with a sealing layer of 0.25 m cement stabilised fly ash and the easterly one with 0.5 m compacted illitic clay from the area. For both materials the target permeability was 10^{-9} m/s or lower. Identical 2 m protective layers of till was put on the impervious layers. Vegetation was allowed to establish, and after a couple of years it consisted of grasses, shrubs and plants of Scots pine. A complete technical description of the technical procedure is given in Lundgren (1990) and Lundgren and Lindahl (1991).

Surface water monitoring

The strategy for monitoring of surface water quality relied upon weekly samples from some 15 locations along the watercourses. Water discharge was measured continuously during the period 1983-88 at the weirs, and the water level at the outlet of Lake Risten was noted on sampling occasions. The weirs were also equipped with automatic samplers that collected up to four daily samples. With exception of automatic sampling all samples were collected manually in 1 L plastic (polyethylene or polypropylene) bottles that were acid washed and thoroughly rinsed with 18 MΩ-water. Samples for metal analysis were acidified with nitric acid (Suprapur). After 1994 water quality measurement became less frequent because of economical constraints. The number of sampling sites and sampling frequencies were reduced successively and after 1996 only a few observations are available. The database contains some 50,000 quality observations at present.

Analytical

Electrical conductivity and pH were measured in the laboratory with conventional electrodes. Metals included in the study were Al Ca, Cd, Cu, Fe, K, Mg, Mn, Na, Pb and Zn. Occasionally also As, Cr and Ni were included. Before 1991 the metals were quantified by atomic absorption spectrophotometry (PerkinElmer 5000 Zeeman), either flame or furnace. This technique was replaced by ICP-MS (PerkinElmer Elan Sciex 5000) in 1991 after validation of the analytical performance. Metal analysis was performed on acidified original samples as well as on 0.40 µm filtrates (not discussed here since the data that were used for statistical analysis are non- filtered data).

Statistical analyses

Statistical methods such as clustering analyses (Q- and R- mode) as well as time series analyses were used to classify the samples with respect to concentrations of Al, Cd, Cu, Fe, Mn, Pb, Zn, as well as pH.

Hierarchical Cluster analysis

Hierarchical Cluster analysis is a common multivariate technique in geochemical statistical studies. Cluster analysis can be applied as an “exploratory data analysis tool” to better understands the multivariate behavior of a data set. The principal aim of this technique is to partition multivariate observations into a number of meaningful, multivariate homogeneous groups. By applying this technique, a large amount of data reduces into a few clusters. Interpreting the clusters is easier, as each cluster contains some observations (samples or variables) that are similar to each other. The outcomes of this cluster analysis are shown by a special plot called dendrogram. The hierarchical clustering method uses the dissimilarities /similarities or distances between objects when forming the clusters. Therefore, two important measurements for this output are distance and linkage measures. It should be noted that diverse techniques can yield different groupings, even when using exactly the same data.

Time series analyses

A time series is a set of observations obtained by measuring a single variable regularly over a period of time. The form of the data for a typical time series is a single sequence or list of observations representing measurements taken at regular intervals. These sets of data have a natural temporal ordering. This makes time series analysis distinct from other common data analysis problems, in which there is no natural ordering of the observations.

Time series analysis comprises methods for analyzing time series data in order to extract meaningful statistics and other characteristics of the data. It is also distinct from spatial data analysis, where the observations typically relate to geographical locations. One of the most important reasons for doing time series analysis is to try to forecast future values of the series. A model of the series that explained the past values may also predict whether and how much the next few values will increase or decrease. The ability to make such predictions successfully is important. Methods for time series analyses may be divided into two classes: (1) frequency-domain methods that include spectral analysis and recently wavelet analysis, and (2) time-domain methods that include auto-correlation and cross-correlation analysis.

Time series models will generally reflect the fact that observations close together in time will be more closely related than observations further apart. Models for time series data can be classified into three main methods such as the autoregressive (AR) models, the integrated (I) models, and the moving average (MA) models. These three classes depend linearly on previous data points.

Combinations of these approaches produce autoregressive moving average (ARMA) and autoregressive integrated moving average (ARIMA) models. The autoregressive fractionally integrated moving average (ARFIMA) model generalizes the former three.

Data are generally divided into two series: An estimation period, and a validation period. A model can be developed on the basis of the observations in the estimation period and the model can be tested and evaluated in the validation period. The results are shown by plots and tables that may include stationary R-square, R-square (R^2), root mean square error (RMSE), mean absolute error (MAE), mean absolute percentage error (MAPE), maximum absolute error (MaxAE), maximum absolute percentage error (MaxAPE) and normalized Bayesian information criterion (BIC). The plots would represent residual autocorrelations and partial autocorrelations. Results for individual models may be expressed in terms of forecast values, fit values, observed values, upper and lower confidence limits, residual autocorrelations and partial autocorrelations.

The data in this study originate from the samples that were taken from three sampling locations: The two lakes (Lake Gruvsjön and Lake Risten) and the creek (Kuntebo). Focus of the study is on the samples that were taken during the period 1985-96. Missing data were replaced and a seasonal effect test was done prior to the statistical analysis. The data were analyzed by SPSS 17. Since the remediation processes have been from 1987 to 1989, the data were divided into two groups, representing the estimation and the validation period, respectively. The samples that were taken before 1985-87 were considered as belonging to the estimation period; and the samples that were taken in the next years were considered as belonging to the validation period. Analytical parameters such as Al, Cd, Cu, Fe, Mn, Pb, Zn and pH were determined separately in each lakes and in the creek.

Prior to processing the data by times series analyses, a model type was selected. A model statistics table shows the goodness of the chosen model assessed from different parameters as mentioned above. Table 1 shows such results; SPSS 17 examines various model types and suggests the optimal one

Table 1. Model types and model fit statistics

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Gruvsjön	Model Type	Model Fit statistics		Ljung-Box Q(18)	
		R-squared	Statistics	DF	Sig.
Al	ARIMA(0,0,0)	0,73	41,23	18,00	0,00
Cd	ARIMA(0,0,0)	0,72	17,58	18,00	0,48
Cu	ARIMA(0,0,0)	0,64	13,26	18,00	0,78
Fe	ARIMA(0,1,1)	0,45	11,24	17,00	0,84
Mn	ARIMA(0,0,0)	0,81	22,09	18,00	0,23
Pb	ARIMA(0,0,0)	0,87	24,77	18,00	0,13
Zn	ARIMA(0,0,0)	0,67	17,61	18,00	0,48

Kuntebo	Model Type	Model Fit statistics		Ljung-Box Q(18)	
		R-squared	Statistics	DF	Sig.
Al	Winters' Multiplicative	0,51	14,23	15,00	0,51
Cd	Simple Seasonal	0,61	27,88	16,00	0,03
Cu	Winters' Multiplicative	0,79	23,71	15,00	0,07
Fe	Simple Seasonal	0,19	11,70	16,00	0,76
Mn	Simple Seasonal	0,49	17,38	16,00	0,36
Pb	Winters' Multiplicative	0,68	21,57	15,00	0,12
Zn	Winters' Multiplicative	0,75	7,48	15,00	0,94

Risten	Model Type	Model Fit statistics		Ljung-Box Q(18)	
		R-squared	Statistics	DF	Sig.
Al	Winters' Multiplicative	0,72	31,11	16,00	0,01
Cd	Simple Seasonal	0,63	14,89	16,00	0,53
Cu	Winters' Multiplicative	0,77	17,65	16,00	0,34
Fe	Simple Seasonal	0,83	27,86	16,00	0,03
Mn	Simple Seasonal	0,61	19,03	15,00	0,21
Pb	Winters' Multiplicative	0,88	21,55	16,00	0,16
Zn	Winters' Multiplicative	0,84	22,00	15,00	0,11

Results

All mentioned statistical analyses were used for the three sampling sites, Lake Gruvsjön, Kuntebo and Lake Risten. None of the data sets show significant seasonal effects regarding to statistical analysis .

Lake Gruvsjön

The samples from this lake were divided into three main clusters by Q- mode cluster analysis (Figure 2). Cluster one includes the samples that were taken in 1990s (from 1990-95); the second cluster as well as the third cluster include the samples that were taken 1988-95. Based on R- mode clustering, the analytical parameters are divided into two main clusters: Cluster one includes Al, Cd, Cu, Mn, Pb and Zn, and cluster two includes Fe and pH (Figure 3).

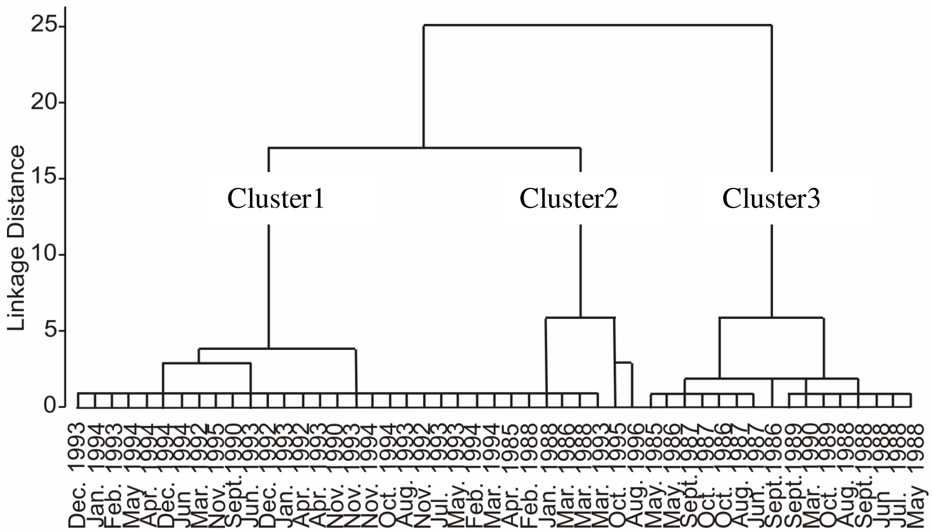

Figure 2. Q- mode cluster analysis of Lake Gruvsjön. The samples from this lake were divided into three main clusters. Cluster one includes the samples that were taken in 1990s (from 1990-95); the second cluster as well as the third cluster include the samples that were taken 1985-96.

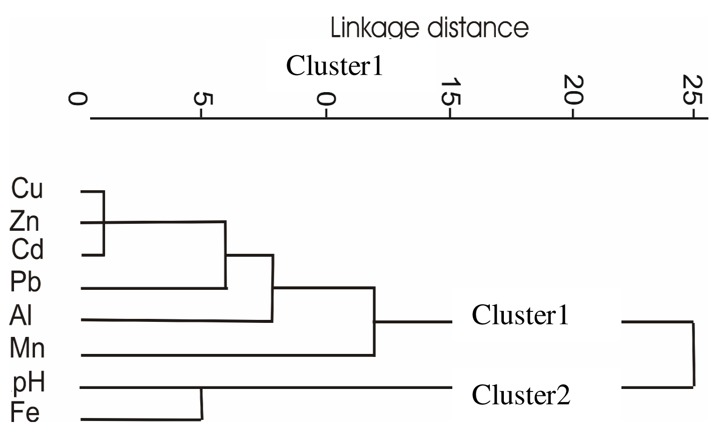

Figure 3. R- mode cluster analysis of Lake Gruvsjön. The analytical parameters are divided into two main clusters: Cluster one includes Al, Cd, Cu, Mn, Pb and Zn; and cluster two includes Fe and pH

Based on time series analyses, Al, Cu and Zn have similar behavior. The measured concentrations had both increasing and decreasing changes before the remediation period. This increasing and decreasing of the concentrations of the elements are also visible during the remediation period (1987-89). However, after this time the concentrations decreased noticeably. The forecasted concentration for the period of remediation is higher than the measured concentration. The behaviour of Cd is similar to that of the mentioned elements except two peaks of concentrations that were measured after remediation period in

1990 and 1991. There are similarities between the behavior of Mn and Pb. There is not a noticeable difference between the measured concentration before and after remediation period. However, two peaks of concentration were measured in 1990 and 1991. The forecasted value for Pb is very close to the measured one. However, the forecasted value of Mn is less than the measured one. The behaviour of Fe is unique and has no similarities to the other elements. The measured concentration of Fe increased noticeably after the remediation period, and a peak of concentration was measured in 1995. Figure 4 shows the described trends.

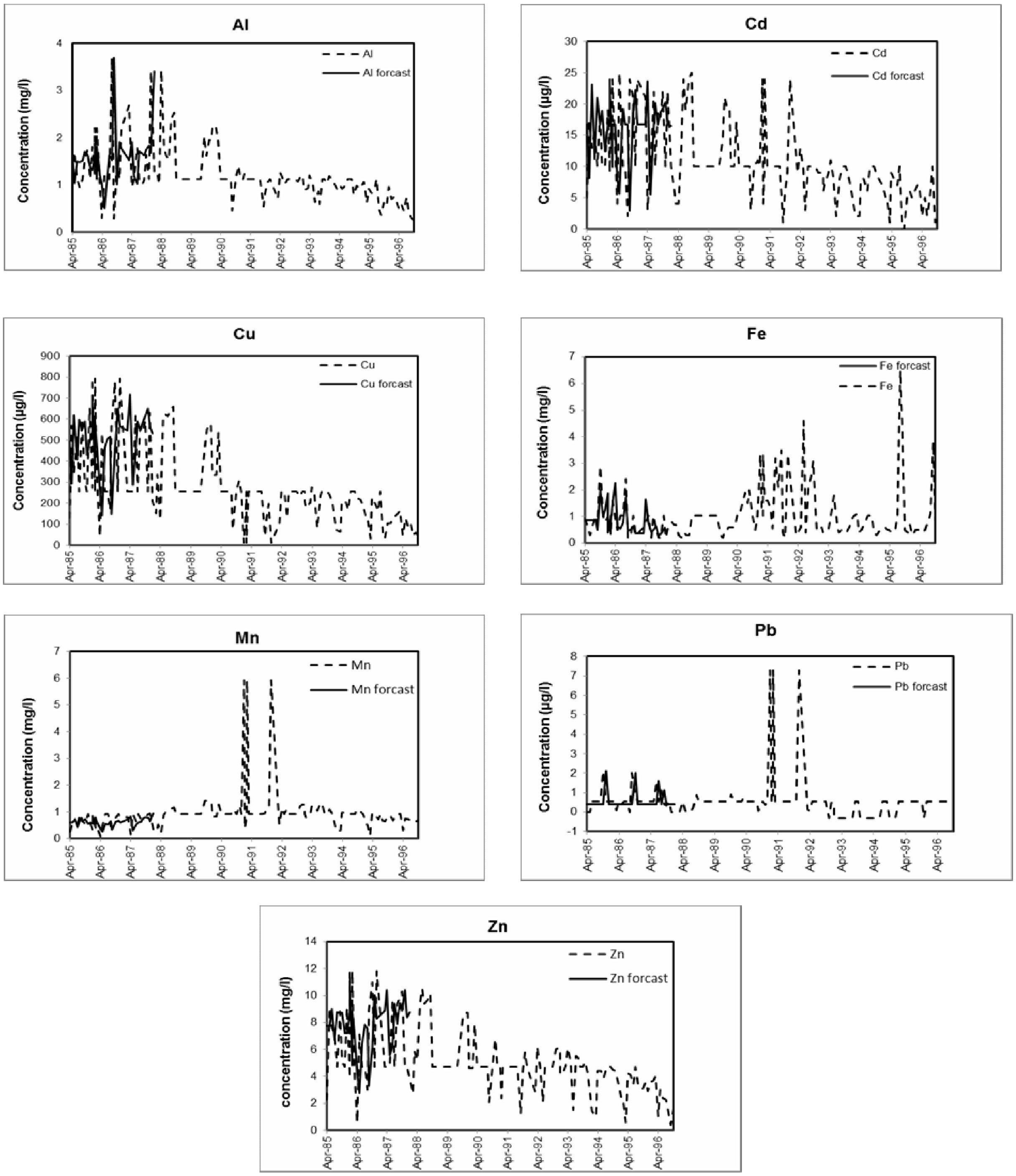

Figure 4. Time series analyses with measured and predicted concentration from 1985 to 1996. The solid line shows the predicted concentration for the period of 1985 to 1988.

Kuntebo

The samples from this creek were divided into two main clusters by Q- mode cluster analyses (Figure 5). Cluster one include the samples that were taken before 1990 and cluster two includes the samples were taken after this year. The analytical parameters were divided into two main groups by R- mode cluster analysis (Figure 6). Cluster one has two sub-clusters. Sub-cluster one includes Al, Cd, Cu and Pb; and second sub-cluster includes Fe, Mn, and Zn. Cluster two has only one member: pH.

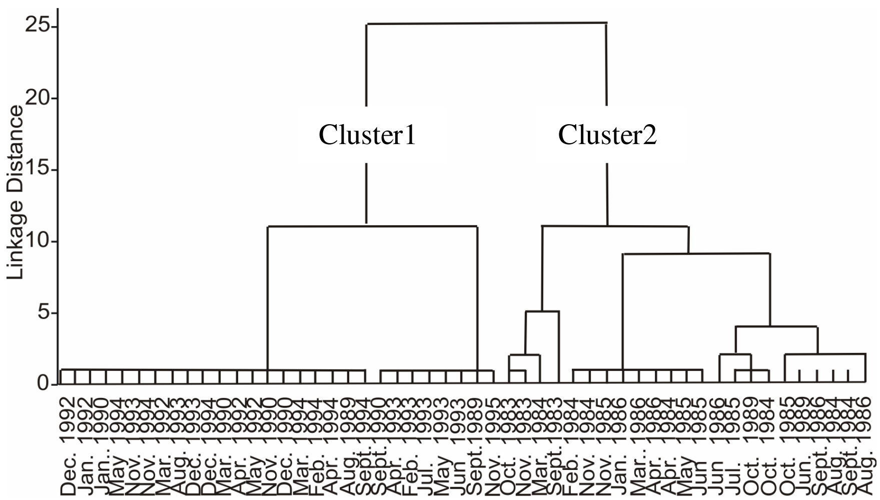

Figure 5. Q- mode cluster analysis of Kuntebo creek. The samples were divided into two main clusters. Cluster one include the samples that were taken before 1990 and cluster two includes the samples were taken after this year.

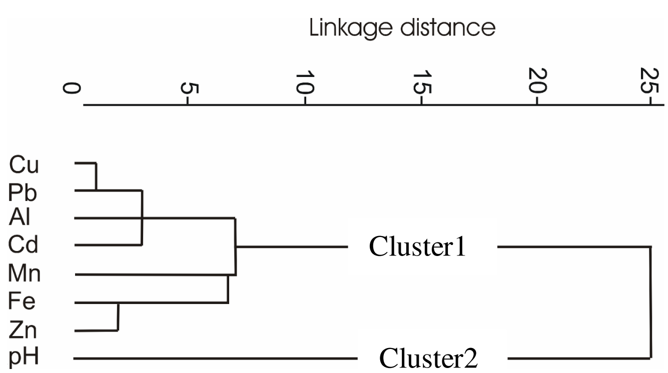

Figure 6**.** R- mode cluster analysis of Kuntebo creek**.** The analytical parameters are divided into two main clusters Cluster one has two sub- clusters. Sub-cluster one includes Al, Cd, Cu and Pb, and sub-cluster two includes Fe, Mn, and Zn. Cluster two has only one member: pH.

Similar to the behaviour of the elements from Lake Gruvsjön, increasing and decreasing the concentrations are visible for all elements from Kuntebo before the remediation period. A similar behaviour is observed for Al, Cd, Cu and Pb. The concentration of each of these elements, decreased noticeably after the remediation period, and the forecasted values are higher than the measured ones after remediation. However, the concentration of Zn does not change noticeably after the remediation period, and the forecasted values are less than the measured ones. The behaviour of Fe and Mn are similar. Regardless the wavy behavior, the concentration of each element decreased after the remediation period. However, two peaks of high concentrations were measured in 1993 and 1995. Figure 7 shows the described trends.

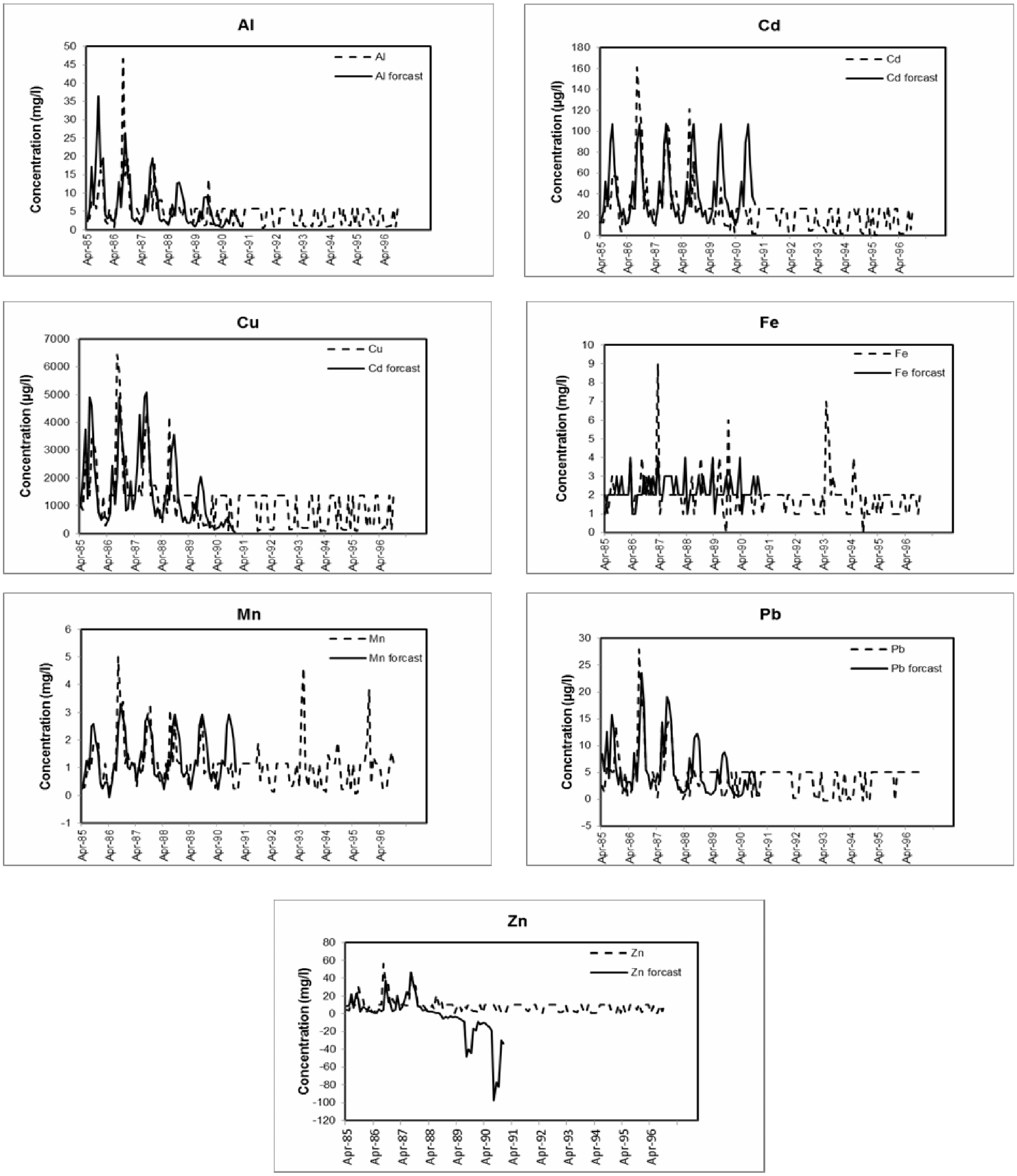

Figure 7. Time series analyses of Kuntebo data, measured and predicted concentration from 1985 to 1996. The solid line shows the predicted concentration for the period of 1985 to 1991.

Lake Risten

The samples from Lake Risten were divided into two clusters by Q-mode cluster analyses. Cluster one includes the samples that were taken before 1988, and cluster two contains samples that were taken after

this time (Figure 8). The R-mode cluster analysis was used to classify the analytical parameters. By means on this method, the analytical parameters were divided into four main groups.
The analytical parameters were divided into two main groups. Cluster one includes all elements; and cluster two has only pH (Figure 9).

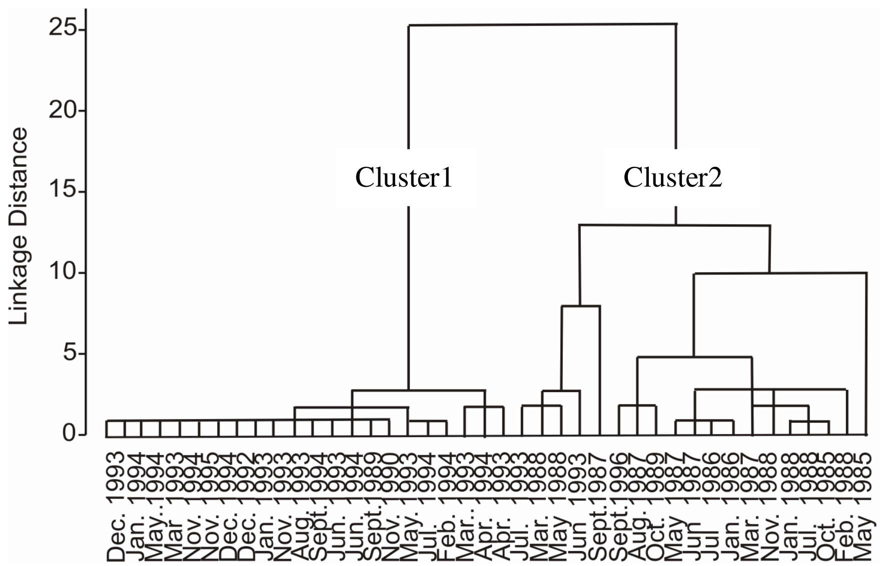

Figure 8. Q- mode cluster analysis of Lake Risten. The samples from this lake were divided into two main clusters. Cluster one includes the samples that were taken before 1988, and cluster two contains samples that were taken after this time.

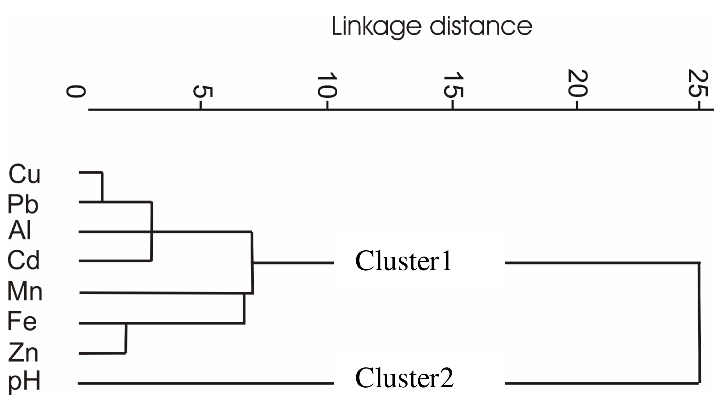

Figure 9. R- mode cluster analysis of Lake Risten. The analytical parameters were divided into two main groups. Cluster one includes all elements; and cluster two has only pH.

Based on the time series analyses, Cu, Pb and Zn exhibit a similar behaviour. A significant decrease occurred after the remediation period (mainly in 1990) for each of these elements, and the forecasted values are significantly higher than measured values. Three elements Al, Cd and Fe behave in a similar way as Al and Cd from Kuntebo and Lake Gruvsjön. The only exception is Mn, which shows a stable concentration before and after the remediation period. Figure 10 shows the described trends.

However, this exception will not influence the main outcome (the concentrations of the elements decreased after remediation period). The similarities of analytical parameters were investigated by R-mode clustering. The similar behaviour of the elements has a physicochemical explanation either by strong interactions with particulate/organic matter (Cu, Pb, Zn, Al, Cd, Fe) and their tendency to be the main element in stationary and mobile carrier phases (Al, Fe). In the Bersbo system Mn remains in an under-saturated state and has a very limited abundance in the particulate phase.

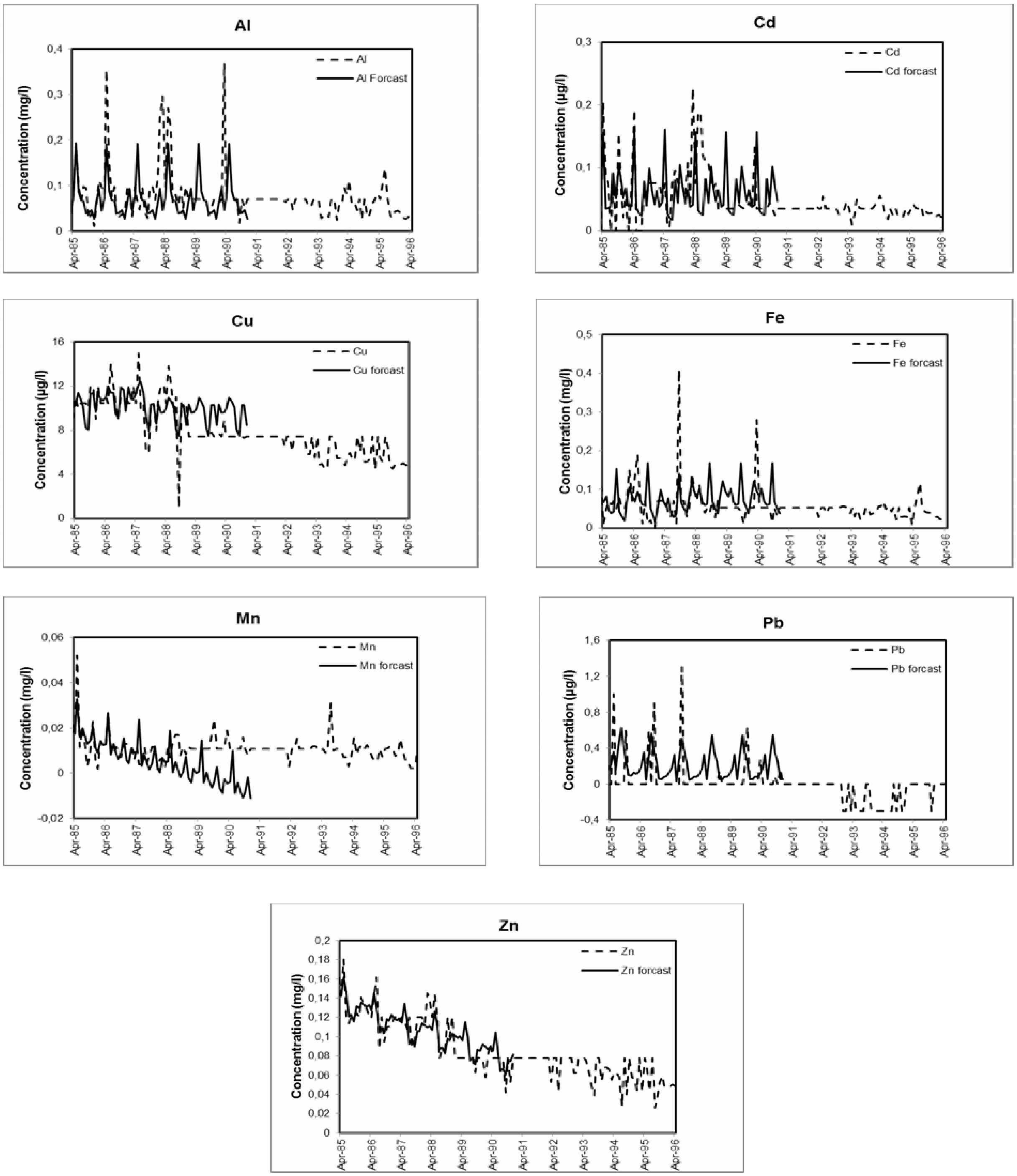

Figure 10. Time series analyses of Lake Risten data, measured and predicted concentration from the period 1985-1996. The solid line shows the predicted concentration for the period of 1985 to 1988.

Discussion and conclusion

The behaviour of the elements is different before and after the remediation period, based in the statistical analysis. These differences can be seen in the Q-mode cluster analyses as well as in the time series analyses. The analysed data are concentrations of single elements, and the results represent concentration changes, not mass fluxes. The Q-mode clusters were generally good representative of the samples that were taken before or after the remediation period. An exception is the Q-mode dendrogram of Lake Gruvsjön, where the samples from the remediation period are clustered into two separate groups.

The rapid increase in pH in combination with annual water flow changes from 0 to 150 l/s at the Kuntebo site are the main reasons for the waviness of the data. In addition, previous studies have identified the quantitative impact of metal redistribution between stationary and mobile carrier phases, respectively, in relation to the transport in the dissolved phase. The single most important factor that control the redistribution processes is pH, as identified by the statistical analysis and previous mechanistic interpretations (Karlsson and Bäckström, 2003). This also highlights the relevance of the pH dependent control for formation of carrier phases through precipitation (hydrous oxides of Al and Fe) as well as adsorption to stationary phases. In addition, the adsorption of trace elements to the solids is a highly pH dependent process. The slightly different outcome for Lake Gruvsjön is attributed to the continued weathering of sulphides in the covered waste where precipitated Fe(III) serves as the oxidizing agent in the absence of air. When groundwater that has passed through the deposit enters into Lake Gruvsjön there is a massive precipitation of ferric hydrous oxides, which is a highly acid generating process. This negative effect is illustrated by the time series analysis.

A general conclusion from the statistical analysis is that it supports the previously suggested mechanistic approach concerning the impact of the remediation on hydrogeochemical conditions. The remediation strategy significantly lowered the concentrations of the toxic metals, particularly at the Kuntebo sampling site. It is also clear that weathering of the waste could not be stopped by covering only, with respect to the increase of iron concentrations at the inlet of Lake Gruvsjön. Consequently it had the largest impact on the water balance. The statistical analysis also suggested the possibility of other mechanisms of mobility control and/or metal sources after the remediation. These circumstances must be fully investigated in order to complete a performance assessment. Hence, the overall success of the remediation should be evaluated in a long term perspective in order to include physicochemical aspects of the system as well as political ambitions.

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