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CHEMOMETRIC ASSESSMENT OF RIVER WATER DURING A FLOOD EVENT

CHEMOMETRYCZNA OCENA WODY RZEKI W CZASIE POWODZI

Summary: Heavy metals, arsenic, and physicochemical basic parameters were analyzed during a spring flood 2005. Daily samples were taken at a sampling site in the middle part of the river Elbe. The site is part of the network of the International Commission for the Protection of the Elbe (IKSE/MKOL). Cluster analysis, principal components analysis (PCA) and source apportionment were used to assess the flood-dependent matter transport. It was found that most of the metal components are in relation to suspended matter transport and can be explained by a sediment washout factor. In contrast, uranium, chloride, and dissolved organic matter were part of the water discharge factor. A third factor contains nickel, manganese, and zinc, reflecting a separate influence of former mining activities in the catchment area.

Keywords: heavy metals and arsenic, suspended matter transport, cluster analysis, PCA, source apportionment

The river Elbe is known as polluted large river in Europe [1, 2]. Substantial efforts to remove contaminants from the river led to a fundamental regeneration of the Elbe ecosystem within the last fifteen years [3–5]. Despite the significant decrease of the pollution situation of the Elbe and its tributaries [6–8], sediment contaminations with heavy metals and organic micropollutants still exist in the river basin [9]. During extreme events such as high floods and low water periods the sediments act as secondary sources for the pollution of the river [10–13]. Thereby trace elements originating from former mining areas in the tributaries Mulde and Saale are of quite importance [14, 15].

The assessment of the flood dependent matter transport can provide actual information of existing pollution potentials in the river basin. In the study chemometric methods are applied as an appropiate tool to identify different pollution sources. Moreover, the

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aim of the study is to improve the understanding of the different transport behaviour of contaminants in the middle part of the river Elbe.

Experimental

Sampling site

Daily samples were taken at Magdeburg sampling site (Fig. 1). The site is located in the middle part of the river Elbe, on the left bank at river kilometer 318 (German mileage). The sampling point is part of the monitoring program of the International Commission for the Protection of the Elbe (IKSE/MKOL). The water quality at this site depends on inputs of upper Elbe stretches (Czech part, Dresden industrial region) as well as inputs of the polluted tributaries Mulde and Saale. Their confluences are 59 km and 27 km upstream of the location, respectively. Therefore, under normal discharge conditions the water quality at this point represents the pollution situation of the Middle

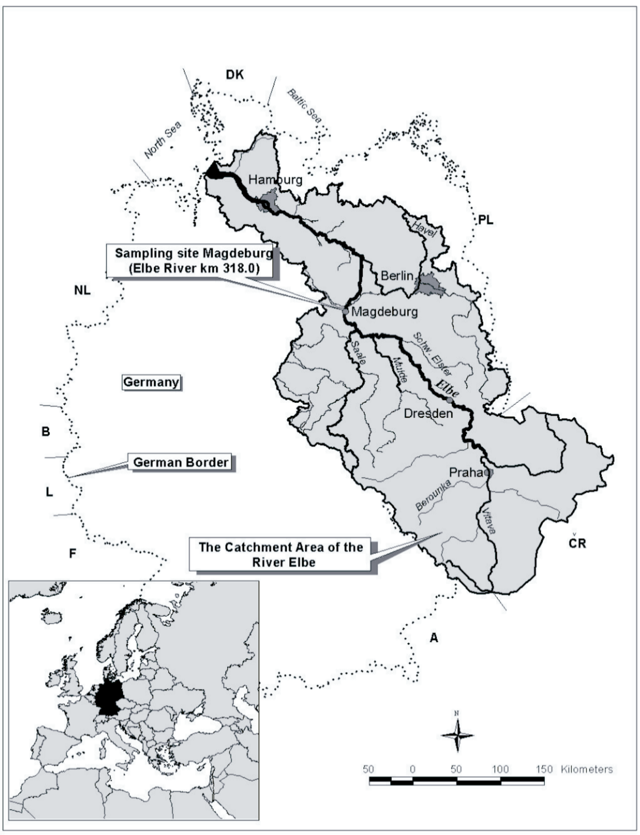

Fig. 1. Catchment area of the river Elbe with Magdeburg sampling site

Elbe. A detailed description of the sampling site with respect to suspended matter transport is given in Baborowski et al. [11].

Sampling period

The investigations were performed during two waves of a spring flood, in February and March 2005. Daily water samples were taken, considering the local specific discharge threshold of the sediment erosion as starting point for the measurement campaign [16].

Physicochemical analysis

The suspended particulate matter (SPM) was characterized as dry substance (DS) and loss on ignition (LOI) according to German Industrial Standards (DIN 38409 part H2, filtration onto Whatman GF/F glass fibre filter). The particle size distribution of SPM was counted in the range of 2 to 200 μm using an optical instrument (AOCOTEAM, PARTmasterL, Germany) based on single particle evaluation [17].

The total P content (TP) was analyzed using the German Industrial Standards (DIN 38405–38406).

Heavy metals and arsenic (As) were measured for the homogeneous sample after digestion with HNO_3/H_2O_2 in a microwave equipment. Aluminium (Al), iron (Fe), manganese (Mn), and zinc (Zn) were determined by optical emission spectrometry with inductively coupled plasma (ICP-OES) (Perkin-Elmer, OPTIMA 3000, Germany), while arsenic, cadmium (Cd), chromium (Cr), cobalt (Co), copper (Cu), lead (Pb), nickel (Ni), uranium (U), and zinc (Zn) were measured using mass spectrometry also with inductively coupled plasma (ICP-MS) (Agilent Technologies).

Dissolved organic carbon (DOC) and the specific ultraviolet absorbance at $\lambda = 254$ nm (UV_{254}) were analyzed after filtration through 0.45 μm PVDF Millipore syringe filters. DOC was measured using a carbon analyser (Dimatec, Germany). UV absorption is calculated as extinction at $\lambda = 254$ nm (Lange, Germany).

The salt component chloride (Cl) was determined using ion chromatography (GAT Gamma Analysentechnik, ICA 5000 series, Germany).

All analytical measurements were done in cooperation with UFZ Department River Ecology Magdeburg, Germany.

Chemometrics

Cluster analysis, principal components analysis (PCA), and source apportionment (APCS apportioning) were carried out to assess the results of the flood investigations. The application of multivariate statistical methods in exploratory data analysis of monitoring data sets from different environmental compartments turns to be the best way of data classification, projection, modeling and interpretation [18–27].

The cluster analysis (Ward's method, squared Euclidean distances) was applied to 23 variables and 43 objects.

To identify the latent factors, which determine the monitoring data structure the PCA was performed with Varimax rotation of the normalized data. Loadings > 0.6 were discussed as statistically significant.

After the identification of the latent factors a multiple regression model was applied, which offers a quantitative relationship between the total mass (concentration) for each tracer species and each latent factor impact. The regression was performed according to the Thurston and Spengler [28] method of source apportioning (APCS apportioning).

Results and discussion

The cluster analysis carried out with respect to detect specific linkage between the chemical and physical variables measured as water quality tracers revealed a distinct clustering of all 23 variables into three separate clusters (Fig. 2).

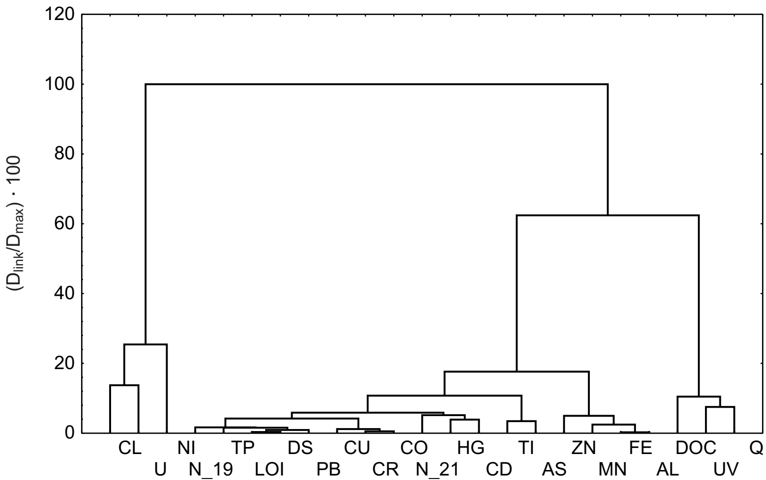

Fig. 2. Hierarchical dendrogram (Ward's method of linkage, squared Euclidean distance as similarity measure) for all 23 variables measured during the flood event

The biggest cluster located in the middle of the dendrogram contains almost all metal components along with the total phosphorus (TP), loss on ignition (LOI) and dry substance (DS) of suspended matter as well as the particle size parameters (n < 20 and n > 20 μm). The big cluster could be separated into two smaller sub-clusters: the left one, which indicates the relationship between smaller particles of the suspensions containing also Cu, Cr, Pb and the right one, indicating links between the bigger particles containing Fe, Mn, Al, Zn, As, Ti, Hg, Cd, *ie* many major components of the river sediments eroded by the inundating stream. The bottom of Elbe River contains coarse sediment (sand, gravel) and this interpretation is very reliable. The two smaller sub-clusters contain characteristics of the Elbe tributaries Mulde and Saale, reflecting former industrial activities (Cd, Cr, Hg) as well as former mining activities (As, Co, Cu, Ni, U, Pb, Zn). For the considered flood event they can be further separated in:

– the clay reference compounds (Al, Fe, Mn) and Zn: mainly transported in particulate form, with the highest sedimentation potential, as compared with other particle-bound elements [17],

– Ti and As: with As reflecting especially inputs of the river Mulde,

– Hg, Cd and n > 20 μm: with Hg as shown as strongly correlated to particles > 20 μm in [17],

– Cu, Co, Cr, Pb, N < 20 μm, DS, LOI: reflecting especially inputs of the river Saale.

Special attention is due to the linkage between Ni, U, and chloride. It reflects different transport way and behaviour as compared with the other chemical species. Here chloride is representative for the influence of the high salt polluted river Saale on the water quality of the river Elbe at Magdeburg monitoring site.

The discharge parameter is related to the dissolved organic carbon (DOC) and the parameter determining the humic substances (UV_{254}), which may mean that the flood washed out carbon of the peat lands and soils of the mountains into the main stream.

It was of interest to detect similarities within the period of monitoring, which coincides with the flood event. In Fig. 3 the hierarchical dendrogram for linkage of the objects (dates of monitoring) is presented.

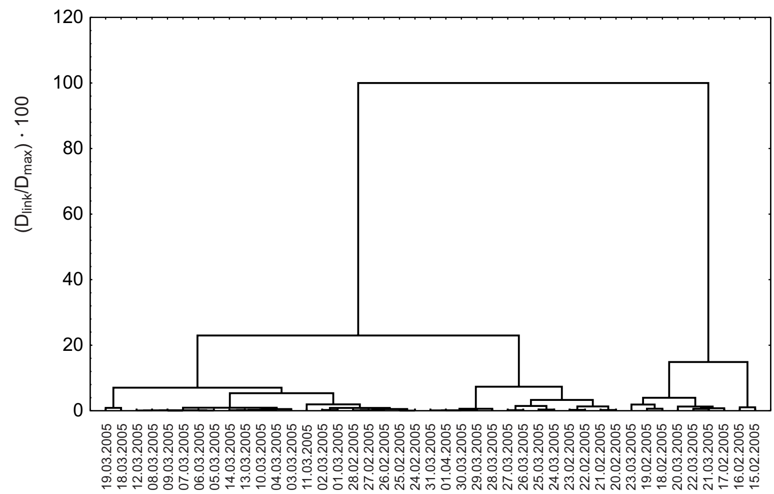

Fig. 3. Hierarchical dendrogram for clustering of the monitoring days

It is readily seen that two major clusters are formed. In the right corner of the dendrogram the dates characterizing the early stage of both flood waves are clustered since they represent a quite extreme situation with respect to the discharge and concentration levels of all chemical parameters. Thereby the two monitoring days in the right part of the first cluster (15./16.02.06) are in relation with the beginning of sediment erosion of the groyne fields above the sampling site. The monitoring days in the left part of the first cluster (17.2–19.02.06 and 20.03–23.03.06) are in relation with the input of suspended matter from the tributaries Mulde and Saale, which occur directly after erosion of the groyne field sediments.

The other cluster, which is separated into two sub-clusters, indicates the intermediate levels of the water discharge and chemical concentrations between the two big waves of the flood event (left) and the decreasing concentration level of particulate pollutants after the flood crest is reached (right). The two days separated in the outside left corner (19./18.03.06) of the dendrogram represent the erosion of groyne field sediments at the beginning of the second wave. They should be part of the first cluster but probably they are shifted into the second cluster, due to the different slope of the discharge curve of the second wave, which led to earlier sediment erosion. It is important to notice that the first cluster together with the two days of the second cluster covers only 26.8 % of the objects, but is representative for the main important processes of the flood-related matter transport.

Next step in the chemometric analysis was to identify the latent factors, which determine the data structure. The analysis was performed by principal components analysis (PCA) with Varimax rotation of the normalized data. PCA was carried out in three ways – using all variables, using only chemical variables, and using only physical variables related to determination of the river water quality.

Table 1

Factor loadings for three principal components (statistically significant values are marked)

Variables	PC 1	PC 2	PC 3
Q	0.270	**0.912**	–0.216
Al	**0.802**	0.344	0.427
As	**0.843**	0.449	–0.069
Cd	**0.930**	–0.035	0.201
Co	**0.899**	0.122	0.379
Cr	**0.901**	0.216	0.325
Cu	**0.927**	0.054	0.291
Fe	**0.834**	0.313	0.415
Hg	**0.854**	0.223	0.308
Ni	0.270	–0.210	**0.801**
Mn	**0.709**	0.179	**0.629**
Pb	**0.952**	0.225	0.167
Zn	**0.667**	0.096	**0.611**
Ti	**0.838**	0.406	0.091
U	0.539	**–0.750**	–0.019
DS	**0.955**	0.193	0.180
LOI	**0.951**	0.146	0.227
n < 20	**0.892**	0.334	0.196
n > 20	**0.906**	–0.030	0.188
Cl	–0.249	**–0.906**	0.214
UV_{254nm}	0.273	**0.847**	0.243
DOC	0.324	**0.669**	0.402
TP	**0.939**	0.126	0.204
Explained variance (%)	59.4	19.1	12.1

Three latent factors are responsible for the data structure and this model coincides with the cluster analysis outcome. They explain over 90 % of the total variance. The first latent factor explains 59.4 % of the total variance and indicates high correlation between most of the metal components along with some physical parameters like particle size, loss on ignition, and dry substance of suspended particulate matter. It could be conditionally named “sediment washout” factor. Obviously, it is the main reason for the water chemical content during the flood event. Due to the specific characteristic of the sampling site Magdeburg the “washout factor” contains both, a washout factor of the remobilisation of groyne field sediments above the sampling site (related to “transport capacity”) as well as a washout factor of tributaries (related to “supply”). Whereas the “transport capacity” depends on the sheare stress and erosion stability of Elbe groyne field sediments, the “supply” depends on the depots of erodable sediments in the reservoirs and lock-and-weir-systems of the main Elbe tributaries Mulde and Saale.

The second latent factor is related dominantly with the increase of the total water quantity and explains nearly 20 % of the total variance of the system. It shows high loading for the discharge parameter but also high and statistically significant figures are observed for dissolved organic carbon (DOC), chloride, uranium, and $UV_{254\ nm}$ (humic substances factor). The conditional name “water discharge” factor seems to be appropriate since it resembles the typical water volume increase during the flood event leading to increased organic material flow into the river. The negative correlation to chloride and uranium variables is clearly an indication for dilution exactly of these species for the period of flooding.

The third latent factor explains also a significant part of the total variance (12.1 %) and needs interpretation. The highest factor loadings in PC 3 are for Ni, but also Mn and Zn have high values. This is a substantial difference to the linkage found in the cluster analysis where Ni was again an exceptional case but its relation was closer to chloride and uranium rather than to Mn and Zn. The relation of Ni to chloride and uranium in the cluster analysis can be explained by similar sources. In the mining affected, high salt polluted river Saale Ni is geochemically correlated and, hence, transported together with U. In contrast, the high factor load of Ni, Mn, and Zn in a separate latent factor (PC 3) of the principal components analysis can be explained by similar sources, but different transport behaviour. Whereas U is transported similar to chloride mainly in dissolved form, Ni, Mn, and Zn are transported partially as particle-bound species. Investigations of the ratio dissolved/particulate of these elements during the flood 2005 along a river stretch in the middle part of the Elbe [29] showed a dissolved part of approximately 20 % for Mn, 25 % for Zn and 55 % for Ni, in contrast to 80 % for U. Different from the particle-bound elements in PC 1, which contains elements of the groyne field sediments as well as of the suspended matter supply by the tributaries, PC 3 can be explained as pure supply component. Probably, this situation reflects a special geogenic “mining supply” effect of former mining activities in the catchment area of the river Saale and it explains the conditional name of this “mining supply” factor. The low loaded factor of As in PC 3 underlines the hypothesis of the river Saale as main source of variables containing in this component. The presence of Mn in PC 3 can be interpreted as indication of ground water influence.

In the next chemometric data interpretation only chemical variables were used for latent factors identification. Again, three latent factors were found responsible for the data structure and they explain over 90 % of the total variance. The case resembles entirely that of the interpretation by all variables. In the PC 1 (explanation of the total variance over 60 %) high factor loadings are found for Al, As, Cd, Co, Cr, Cu, Fe, Hg, Mn, Pb, Ti, Zn, and TP; in PC 2 (over 17 % explained variance) Ni has the highest loading but, again, Mn and Zn have relatively high loadings values (it may mean again that they originate from the discussed sources during the flooding period); in PC 3 (with

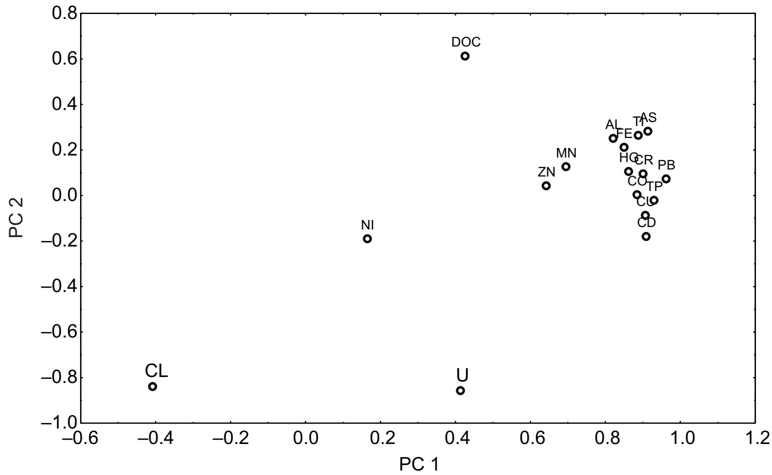

Fig. 4. Bivariate plot PC 2 *vs* PC 1 for only chemical parameters

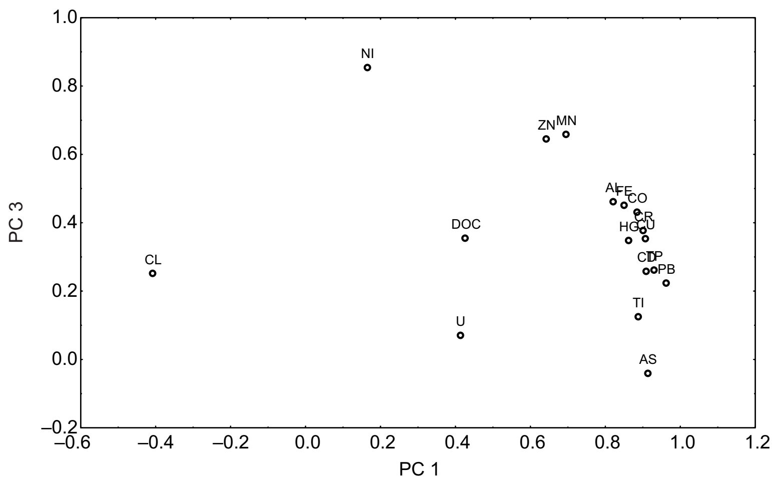

Fig. 5. Bivariate plot PC 3 *vs* PC 1 for only chemical parameters

nearly 13 % explained variance) chloride and uranium are correlated, on the one hand, and DOC, on the other. The reasons for the relationships are the same as before – impact of peat lands and soil washout effects and of enhanced water level. In Figs. 4 and 5 the bivariate plots for the factor loadings (PC 2 *vs* PC 1 and PC 3 *vs* PC 1) are presented.

Finally, only physical parameters describing the flood event were involved in PCA. They are grouped into two latent factors explaining over 90 % of the total variance. The first one (with over 60 % explained variance) is conditionally named "dry substance" factor since it includes high loadings for parameters characterizing the particle size of the suspended matter in the water bulk: dry substance, particle number concentration for n < 20 μm and n > 20 μm and loss of ignition.

The second latent factor, with over 30 % explained variance, shows the relationship between the water discharge and the specific ultraviolet absorbance and its conditional name could be "humic substances" factor. In Fig. 6 the bivariate plot for the distribution of the factor loadings for the physical variables (PC 2 *vs* PC 1) is given.

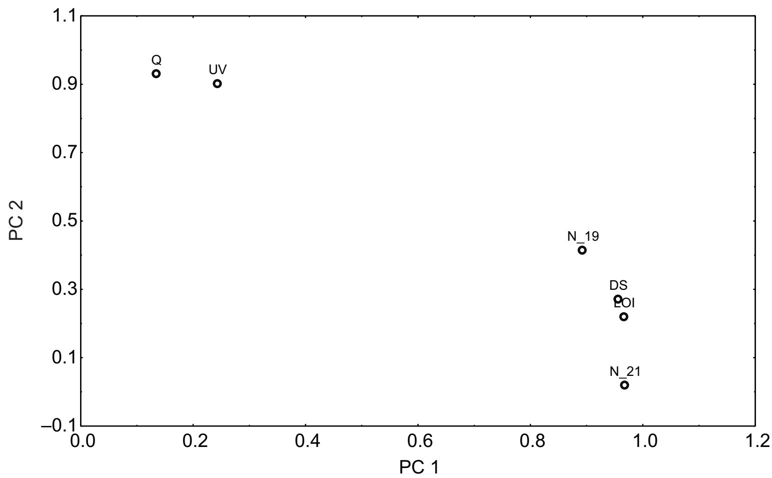

Fig. 6. Bivariate plot PC 2 *vs* PC 1 for only physical parameters

The identification of the latent factors responsible for the monitoring data structure made it possible to create multiple regression models, which offer a quantitative relationship between the total mass (concentration) for each tracer species and the each latent factor impact. The regression is performed according to the Thurston and Spengler method of source apportioning (APCS apportioning) [28] and informs on the contribution of each identified factor on the total concentration (or total mass) of the species involved in the monitoring procedure. In Table 3 the source apportionment models are shown.

In this table the column "Intercept" represents the free term of the regression and its physical meaning is the percentage of unexplained mass (concentration). The square of the multiple regression coefficient r^2 is a measure for the model fit (relation between measured and calculated by the model concentrations or masses).

Table 2

Source apportionment (in %) for the flood event

Species	Intercept	Sediment washout	Water discharge	Mining supply	r^2
Q	8.8	—	91.2	—	0.88
Al	7.5	75.2	7.2	10.1	0.89
As	15.2	71.1	13.7	—	0.81
Cd	10.1	80.6	—	9.3	0.79
Co	9.2	75.5	6.1	9.2	0.84
Cr	10.3	74.2	5.5	10.0	0.82
Cu	8.6	82.8	—	8.6	0.78
Fe	7.7	77.1	6.3	8.9	0.85
Hg	8.3	73.2	8.2	10.3	0.81
Ni	9.8	6.1	—	84.1	0.77
Mn	13.9	42.6	5.1	39.4	0.79
Pb	20.4	60.8	10.2	8.6	0.81
Zn	9.7	44.2	—	46.1	0.82
Ti	7.7	72.2	20.1	—	0.79
U	12.1	21.2	66.7	—	0.82
DS	16.0	67.1	8.3	8.6	0.81
LOI	24.8	63.2	3.9	8.1	0.72
$n < 20$	15.0	70.2	9.0	5.8	0.78
$n > 20$	9.3	80.3	—	10.4	0.79
Cl	13.3	—	74.5	12.2	0.75
UV_{254nm}	8.7	5.5	81.3	4.5	0.81
DOC	2.0	10.0	75.5	12.5	0.82
TP	9.4	79.0	3.6	8.0	0.77

The regression models obtained indicate quite a good fit and give an idea about the contribution of each identified latent factor on the total mass or concentration. For instance, for most of the metal components the sediment washout process is of substantial importance for their total concentration during the flood event. The water discharge factor is for them of lesser importance but the mining supply impact should not be neglected. On the contrary, for Ni, Mn, and Zn this is a very important contributor.

The specific ultraviolet absorbance (related to humic substances) is strongly influenced by the increased water discharge, while particle tracers (size, suspended solids, loss of ignition) are depending mainly on sediment washout.

Conclusion

The paper presents a combination of

(I) cluster analysis of physicochemical variables and sampling days to detect water quality tracers characteristic for the course of the flood,

(II) PCA to identify the latent factors determining the data structure, and
(III) APCS apportioning to inform on the contribution of each identified factor on the total concentration of the analyzed variables.

The results show that the described assessment method can improve the understanding of the flood-dependent matter transport in the sub-catchment areas above the sampling site. Its application to the middle part of the river Elbe allows the identification of three main pollution sources:
(i) sediment washout,
(ii) water discharge, and
(iii) mining supply.

In this connection metals linked to the clay fraction of sediments were shown as tracers for the sediment washout, whereas dissolved parameters (DOC, UV_{254}, chloride, U) were characteristic for water discharge and Ni, Zn, and Mn for mining supply. Moreover, it could be shown that the main important processes of the flood-related matter transport occur at the early stage of the flood.

Beside others, in sediment management of river basins, the evaluation of source--control programs is essential to perform a cost-benefit analysis of risk reduction through source control [30]. With respect to the development of tailor-made flood measurement programs, the results underline the importance of the right starting time of a sampling campaign. With the aim to optimize such programs for the river Elbe, further investigations are necessary to reduce the number of water quality tracers containing in the sediment washout source.

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CHEMOMETRYCZNA OCENA WODY RZEKI W CZASIE POWODZI

Streszczenie

Podczas wiosennej powodzi w 2005 roku analizowano w wodzie rzeki stężenie metali ciężkich, arsenu i parametry fizykochemiczne wody. Próbki pobierano w środkowej części biegu rzeki Łaby. Ten teren jest częścią większego obszaru będącego pod ochroną Międzynarodowej Komisji Ochrony Łaby (IKSE/MKOL). Zastosowano analizę klastrów, analizę czynników głównych (PCA) i udziału źródeł pochodzenia zanieczyszczeń do oceny wpływu powodzi na transport zanieczyszczeń wody. Stwierdzono, że największe stężenia metali pochodzą z materii zawieszonej, pochodzącej z wymywania osadów dennych. Natomiast uran, chlorki i rozpuszczone substancje organiczne pochodziły ze zrzutów ścieków do rzeki. Obecność w wodzie niklu, manganu i cynku wskazywała na wpływ, już zaprzestanej, działalności górniczej w tym rejonie dorzecza.

Słowa kluczowe: metale ciężkie i arsen, pyły zawieszone emitowane przez transport, analiza klastrów, PCA, udział źródeł