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Chemometrical analyses of Zn distribution between water and soil of dams in Chirpan Municipality, Bulgaria

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Abstract. *This study was designed to evaluate the ecological status and to define probable liquid/solid correlations of Zn distribution between dam waters and adjacent soils in Chirpan Municipality, as a part of a continuous water and soil assessment project of Stara Zagora Region. Zinc concentrations in the investigated water and soil samples were determined by atomic absorption spectrometry (AAS) on AAnalyst 800 Atomic Absorption Spectrometer, Perkin Elmer, over the period December 2009 – November 2010. The analysis of Zn temporal distribution in the dam waters revealed maximum heavy metal loading of the three surface water bodies during June 2010. The ecological assessment of the studied soil samples showed that Zn content in some of them surpassed the maximum permissible concentrations (MPC), according to Bulgarian state standards. Basic statistical parameters (standard deviation values), liquid-to-solid metal correlations, linear regression analyses and Principal component analyses (PCA) were used to rationalize and interpret the analytical data for both media. Relationships between Zn contents in dissolved (dam water samples) and particulate (soil samples) phases in Chirpan Municipality, ascertained a high degree of water/soil correlation during December 2009 (R^2 0.9963) and July 2010 (R^2 0.9033) in the three investigated compartments. Distribution coefficients (K_d) of the trace metal between both phases presented as log K_d were in the range 1.98 - 3.26. The PCA confirmed the conclusions withdrawn on the bases of the chemical analyses and categorized the investigated sampling points into three classes – Class 1 (SP 216, SP 410A, SP 410-1A) with predominant Zn loading of the water bodies; Class 2 (SP 217, SP 418A, SP 418-1A) featured with excessive Zn soil contents; Class 3 (SP 215, SP 409A, SP 409-1A) with average levels of the heavy metal in both phases. The data of the present study provided a scientific basis for best-management practices of natural water and land use in the investigated municipality.*

Keywords: zinc, dam water, soil, ecological status, distribution coefficients

Introduction

Among the most significant water and soil contaminants resulting from both natural and anthropogenic sources, heavy metals are of prime importance due to their long-term toxicity effect and non-biodegradability (Chen et al., 2005; Yaylali-Abanuz, 2011). Many scientific activities have been devoted to the determination of the sources, types and degree of the heavy metal pollution in natural surface waters and soils (Christoforidis and Stamatis, 2009; Franco-Uría et al., 2009; Yaylali-Abanuz, 2011). Their content and the impact upon ecosystems are influenced by many factors such as parent material, climate and anthropogenic activities: industry, agriculture and transportation. Due to heterogeneity of the soil and often accidental nature of contaminating processes, concentrations of heavy metals can vary remarkably over short distances. Therefore, the high variability of the heavy metals soil concentrations obtained at various sampling sites requires a careful evaluation and interpretation to decide which of contributions, pedogenic or anthropogenic, are of crucial importance for the main distribution patterns found in the samples (Vasilev, 2009; Skrbic and Durisic-Mladenovic, 2010).

The anthropogenic sources of Zn are related to the non-ferric metal industry and agricultural practice (Kabata-Pendias, 2000). Most Zn fertilizers ($ZnSO_4.H_2O$; $ZnSO_4.7H_2O$; $ZnSO_4.xH_2O$; ZnO; $ZnCO_3$; $ZnCl_2$; $Zn(NO_3)_2.3H_2O$; chelates - $Na_2ZnEDTA$, NaZnHEDTA, NaZnNTA, $Zn_3(C_6H_5O_7)_2.2H_2O$; $Zn(NH_3)_4SO_4$) have a significant residual effect in soils. Reports have shown that crop response to applied Zn can be demonstrated at least five years after application. Because Zn has such a residual effect, levels of available Zn in soils may increase with annual applications of Zn fertilizers. Soil or plant tissue samples should be taken to monitor the available Zn status with time. Zn rates should decrease or no Zn should be applied if these levels increase to the adequate range (Robson, 1993).

High doses of zinc show toxic and carcinogenic effects and result in neurologic complications, hypertension, kidney and liver function disorders (Yaylali-Abanuz, 2011). Extensive literature on the aquatic toxicity of Zn and especially its toxicity to fish has been reviewed. Zn is unusual in that it has low toxicity to man, but relatively high toxicity to fish. Accumulation of Zn and Cu in soils coupled with decrease in soil pH has a potential of aggravating the nutritional disorders associated with toxicity of these metals in the long run (Fatoki and Awofolu, 2003; Rattan et al., 2005).

Besides, Zn is a basic component of some primary and secondary minerals from which it could be released from weathering. Surface horizons of Bulgarian soils contain from 2.5 to 160.0 mg/kg Zn. The average content is approximately 60.0 mg/kg, which is slightly higher than the average value for soils worldwide - 50 mg/kg. (Gorbanov et al., 2005) Zinc content in natural and

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contaminated soils from mining and processing of lead-zinc ores can reach extremely high values - up to tens of grams per kilogram of soil (Vasilev, 2009).

The modern chemometrics is a branch of chemistry (very often related to analytical chemistry) which deals with the application of mathematical and statistical methods in order to evaluate, classify, model, and interpret chemical and analytical data, to optimize and model chemical and analytical processes and experiments and to extract a maximum of chemical and analytical information from experimental data (Mevic and Wehrens, 2007; Simeonov et al., 2010). The Principal Component Analyses (PCA) then provides a basis for interpreting different clusters of the metals based on their covariation (Simeonov et al., 2005; Tariq et al., 2006).

The aim of the present study was to evaluate the ecological status of natural water bodies (dams) and soils in Chirpan Municipality according to index Zn, and to assess liquid/solid correlations of Zn distribution between dam waters and adjacent soils by applying chemometrical techniques.

Material and methods

Water and Soil Sample collection

A total of 21 dam water samples and 6 soil samples were collected from 3 dams and the adjacent soils in Chirpan Municipality, Stara Zagora Region, Bulgaria during the period December 2009 - November 2010 (Table 1). Global positioning system (GPS) was used to determine the coordinates of the sampling points.

Spectrometric analyses

The water samples were collected and conserved according to the standard methods for the examination of water and wastewater (EN ISO 5667-1, EN ISO 5667-3) (American Public Health Association, 2008**)**. Zinc concentrations in the investigated real surface water samples were determined by atomic absorption spectrometry (AAS) (EN ISO 8288) on AAnalyst 800 Atomic Absorption Spectrometer, Perkin Elmer, at λ 213.9 nm during the period December 2009 - November 2010.

The soil samples from the specified sampling points (Table 1) were taken once, randomized from two depths: 0÷20 cm and 20÷40 cm. The sampling, transportation and preservation procedures: single manual sampling, composite sampling, were conducted following EN ISO 10381 requirements. pH of the soil samples was determined in aqueous extracts. The soil samples preparation and acid mineralization were conducted according to standard EN ISO 11466. The principle of the method is treatment of the soil sample with *aqua regia* (HNO_3:HCl = 1:3) for 16 hours at room temperature and subsequent two-hour treatment with heating, condensation and return of the escaping vapors with a reflux condenser. Zn concentrations in the investigated soil samples were determined by AAS on AAnalyst 800 Atomic Absorption Spectrometer, Perkin Elmer, according to EN ISO 11047.

All investigations were performed in triplicate. The comparative analysis of the investigated natural water bodies'/soil quality according to index Zn included evaluation of the coefficient of technogenic impact (K_T), expressed by the relationship:

$$K_T = \frac{Me_{exp.}}{Me_{st.}} \quad (1)$$

where $Me_{exp.}$ is the experimentally determined average annual heavy metal concentration in the water/soil samples (mg/L, mg/kg), and $Me_{st.}$ is the selected standard for surface water/soil quality (mg/L, mg/kg).

Potential leaching of metals and their bioavailability in soils were analyzed by using the relative partitioning of metal between soil and surface water (Watmough et al., 2005; Panichayapichet et al., 2007). The distribution coefficient, K_d (L/g) is the ratio of the total metal concentration in soil (mg/kg) over the concentration of dissolved metal in the corresponding surface water sample (mg/L).

$$K_d = \frac{Me_{soil}}{Me_{water}} \quad (2)$$

Statistical analyses

The statistical significance of the results was tested on the basis of the Standard Deviation (SD) values calculated by the Student's t-test. Principal component analysis (PCA) and was applied to the experimental data to assess relationship between Zn content in water and soil compartment and possible heavy metal distribution patterns. The multivariate statistical analysis was carried out by XLStat Pro.

Table 1. List of the investigated water and soil sampling points (SP)

SP	Code	Sample type	GPS coordinates
Chirpan Dam	SP 215	dam water	N 42.09753° E 25.18842°
	SP 409A	vertisol (0-20 cm)	N 42.09764° E 25.18977°
	SP 409-1A	vertisol (20-40 cm)	N 42.09764° E 25.18977°
Zetyovo Dam	SP 216	dam water	N 42.09112° E 25.21541°
	SP 410A	vertisol (0-20 cm)	N 42.09127° E 25.21712°
	SP 410-1A	vertisol (20-40 cm)	N 42.09127° E 25.21712°
Malak Yurt Dam	SP 217	dam water	N 42.19494° E 25.18706°
	SP 418A	fluvisol (0-20 cm)	N 42.19493° E 25.18687°
	SP418-1A	fluvisol (20-40 cm)	N 42.19493° E 25.18687°

Results and discussion

All experimental results were interpreted according to the national surface water and soil standards (Table 2).

Assessment of Zn contents in dam water

The chemical analyses data of the 21 dam water samples taken from Chirpan, Zetyovo and Malak Yurt Dams, Chirpan Municipality, during the period December 2009 – November 2010, allowed tracing out the temporal variations of Zn content. The experimental time-dependent curves presented in Figure 1 displayed analogous tendency and revealed highest heavy metal loading of all investigated samples during December 2009 and June 2010. Additionally, it was clearly observed that SP 216 was outlined as the "hot spot" regarding this index. The measured Zn concentration in June 2010 (2.037 mg/L) was up to 3.3 times higher compared to those registered in the other two dams during the same month. Moreover, the registered heavy metal content surpassed the quality standard for I category surface waters, as well as Category A1, A2 and A3 guide limits for surface water intended for drinking water abstraction and household supply, and practically coincided with the ACL for water intended for irrigation (Table 2, Figure 1). The observed tendency was probably due to two major factors: i) increased water vapourization due to the higher temperatures leading to reduction of the dam water volume; ii) favourable conditions for Zn mass transfer from the adjacent soils probably enriched with fertilizers during the spring. Besides, the present results and assumptions were sustained by the study of Georgieva et al. (2011) who established elevated Zn concentrations in the groundwaters of Chirpan Municipality during December 2009, attributed to more intensive penetration of Zn-rich infiltrates from the upper soil layers and the surface waters caused by gradual snow melting.

The concentration curves of SP 215 and 217 (Figure 1) characterized with smooth slopes expressing a decrease of the metal content in the direction June – July – September, while the plot for SP 216 was featured with quite a steep slope.Obviously, the waters of the three dams characterized with the lowest heavy metal concentration (0.002 - 0.044 mg/L) during the spring (March, May) and autumn (September, November) months (Figure 1). The latter could be attributed to intensive rain/snow falls, subsequent dilution and decrease of Zn content in the aqueous phase. The minimum, average annual and maximum Zn concentrations and total hardness (TH) values in all water samples are presented in Table 3. Additionally, the critical analyses of the experimental data outlined that the investigated dam waters were not suitable for pisciculture, especially for *Salmonid*. Zn contents surpassed the mandatory standard for *Salmonid* waters (0.5 mg/L) up to 4 times in 20 % of the water samples (Table 2, Figure 1). It should be emphasized that the ecological assessment of the dam water quality according to Regulation No. 4 was consistent with the requirements for total water hardness (Table 3).

The calculated distribution coefficients (K_d) of the trace metal between both phases presented as $log\ K_d$ were in the range 1.98 - 3.26 (Table 3). The SD_{Zn} values for all investigated sampling points were calculated and presented in Table 3. The range of their variation, from ±0.11 to ±0.29, proved the statistical significance of the experimental results.

Table 2. National surface water and soil quality standards regarding index Zn

Water/soil category	Category/value			Ref.
Surface water	I category	II category	III category	Regulation No. 7
	1.0 mg/L	5.0 mg/L	10.0 mg/L	
Water intended for irrigation	Allowable limit concentration (ALC)			Regulation No.18
	2.00 mg/L			
Surface water intended for drinking water	Category A1	Category A2	Category A3	Regulation No.12
abstraction and household supply	0.5 (G)	1.0 (G)	1.0 (G)	
	3.0 (M)	5.0 (M)	5.0 (M)	
Waters supporting fish and shellfish	Salmonid water (M)		Cyprinid water (M)	Regulation No. 4
organisms' life				
TH ⩽ 10.0 mg/L $CaCO_3$	0.03 mg/L		0.3 mg/L	
10 ⩾ TH ⩽ 50 mg/L $CaCO_3$	0.2 mg/L		0.7 mg/L	
50 ⩾ TH ⩽ 100 mg/L $CaCO_3$	0.3 mg/L		1.0 mg/L	
100 ⩾ TH ⩽ 500 mg/L $CaCO_3$	0.5 mg/L		2.0 mg/L	
	Precautionary concentration (PC)			Regulation No.3
Clay-sandy and sandy soils	110 mg/kg			
Clay soils	180 mg/kg			
Permanent lawns/Uncultivated land	Allowable limit concentration (ALC)			Regulation No.3
- pH < 6.0	220 mg/kg			
- 6 < pH < 7.4	390 mg/kg			
- pH > 7.4	450 mg/kg			

G - guide value; M - mandatory value; TH - total hardness

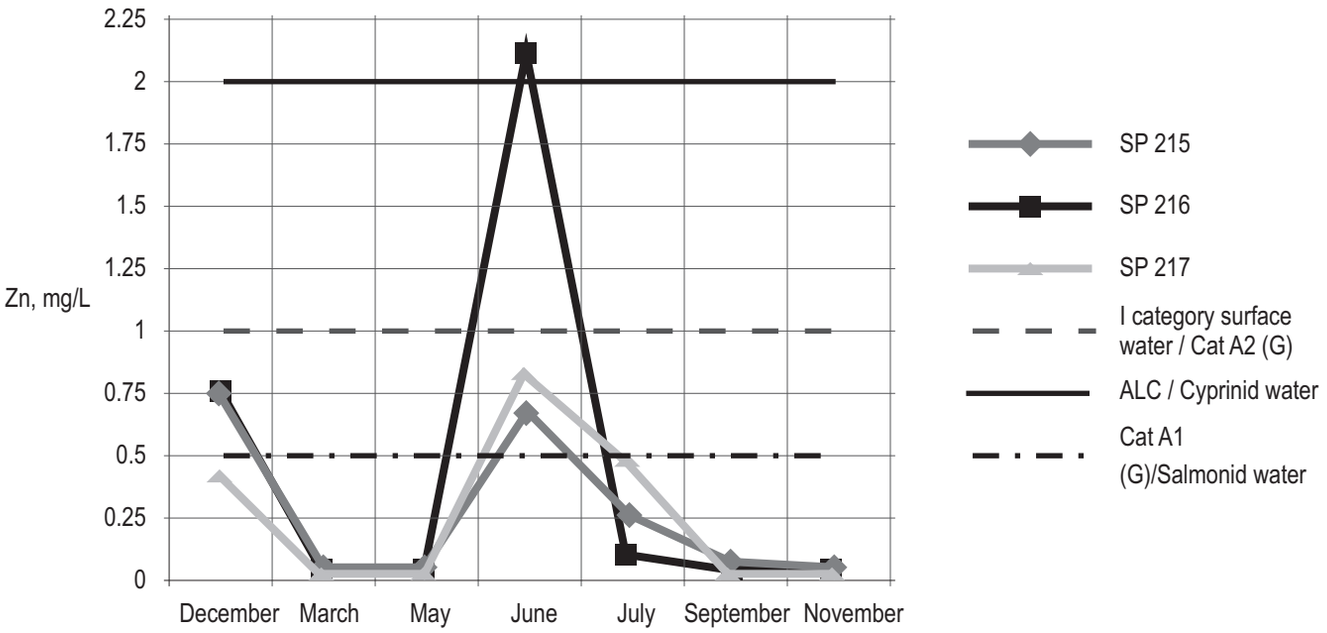

Figure 1. Temporal Zn distribution curves in the surface waters of Chirpan, Zetyovo and Malak Yurt Dams during the monitoring period

Table 3. Minimum (C_{min}, TH_{min}), average annual C*, TH*) and maximum (C_{max}, TH_{max}) Zn concentrations and TH values, K_T, SD and Log K_d values

Parameter	SP 215	SP 216	SP 217
C_{min}, mg/L	0.002	0.002	0.002
C*, mg/L	0.23	0.4	0.21
C_{max}, mg/L	0.7	2.04	0.8
SD_{Zn}	± 0.11	± 0.29	± 0.14
KT*	0.35	1.02	0.40
Log K_d (soil A/water)	2.498	1.984	3.291
Log K_d (soil-1A/water)	2.436	2.047	3.261
TH_{min}, mg/L CaCO3	265	277.5	161
TH*, mg/L CaCO3	417.5	393	199.5
TH_{max}, mg/L CaCO3	512.5	589.5	228.5
SD_{TH}	± 0.67	± 0.77	± 0.22

* according to ALC

Assessment of Zn contents in soil

The soil samples from the three sampling points were collected during April, 2010 from two depths: 0 – 20 cm and 20 – 40 cm. Zinc contents in all studied soil samples ranged from 38.6 to 410.5 mg/kg (Figures 2 and 3)). The comparative analyses between the experimental results and the national soil quality standards (Table 2) established that the samples around Chirpan and Zetyovo Dams corresponded to the quality requirements as the measured Zn concentrations were far below the PC for clay soils and the ALC for uncultivated land (Figure 2). Besides, the trace metal content in the soils around Chirpan Dam (SP 409A, 409-1A) were 1.8 times higher than that in the samples around Zetyovo Dam.

The present study revealed significant Zn loading of the soil samples taken from the area around Malak Yurt Dam (Figure 3). According to the measured heavy metal concentrations in both samples, they were approximately 10.5 times more polluted with Zn compared to the soils from SP 409A, 409-1A, 410A and 410-1A. In addition the registered metal levels surpassed with 3.5 times the PC for sandy soils (110 mg/kg, Table 2) and were very close to the ALC (450 mg/kg, Table 2) regulated by Regulation No. 3. As Zn toxicity regarding soil depends strongly on the soil pH, it should be noted that the results in the present study were interpreted with respect to the pH values of the investigated samples, which are as follows: pH 8.17 – SP 409A; pH 8.34 – SP 409-1A; pH 7.99 – SP 410A; pH 8.22 – SP 410-1A; pH 7.74 – SP 418A; pH 8.16 – SP 418–1A.

Statistical interpretation of the experimental data

To assess Zn distribution and probable mobility between aqueous and soil compartment, correlation analysis of the heavy metal concentrations in the water and soil samples was conducted (Figure 4). Relationships between Zn contents in dissolved (dam water samples) and particulate (soil samples) phases in Chirpan Municipality, ascertained a high degree of water/soil correlation during December 2009 (R^2 = 0.9963) (Figure 4a) and a satisfactory linear dependence during July 2010 (R^2 = 0.9033) (Figure 4b) in the three investigated compartments. The factor analysis was carried out with the Principal Component method which is, rather than the original data, based on the examination of dependency among the artificial variables which are computed from covariance and correlation coefficient matrixes. Principal Component Analysis (PCA) was conducted to investigate associations of different sampling sites and to assess the probable sources of Zn in both phases (Figure 5). For interpretation of the projected data both the score vectors were plotted. In the score plots, the grouping of objects can be recognized (Simeonov et al., 2010).

The results of the PCA, presented as a scatter plot in Figure 5, proved the observations derived on the bases of the chemical

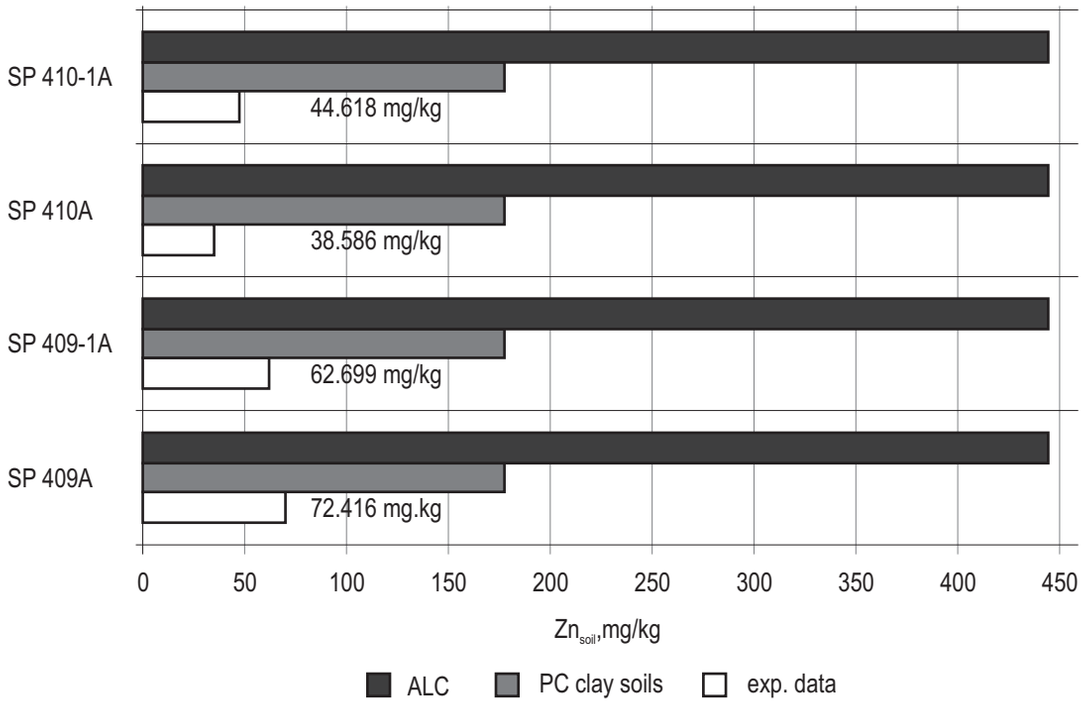

Figure 2. Zinc concentrations in the studied clay soil samples (Chirpan and Zetyovo Dams)

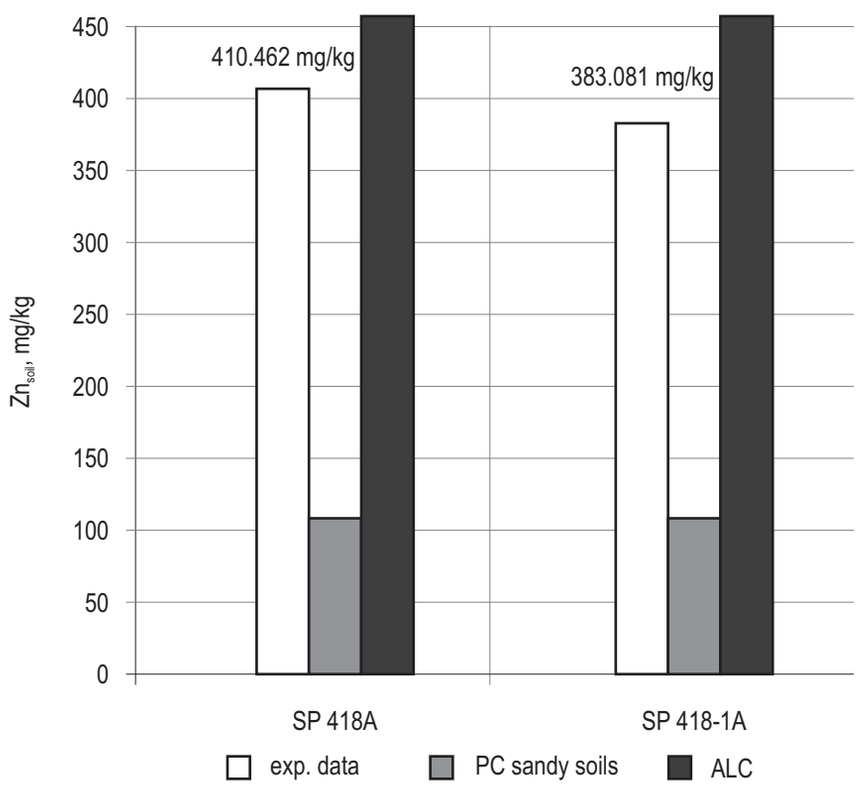

Figure 3. Zinc concentrations in the studied sandy soil samples (Malak Yurt Dam)

analyses and categorized the investigated sampling points into three classes – Class 1 (SP 216, SP 410A, SP 410-1A) with predominant Zn loading of the water bodies; Class 2 (SP 217, SP 418A, SP 418-1A) featured with excessive Zn soil contents; Class 3 (SP 215, SP 409A, SP 409-1A) with average levels of the heavy metal in both phases. The analysis of the experimental data and the PCA, however, revealed a contradictory issue, represented by Class 2, namely lowest Zn loading of Malak Yurt dam waters

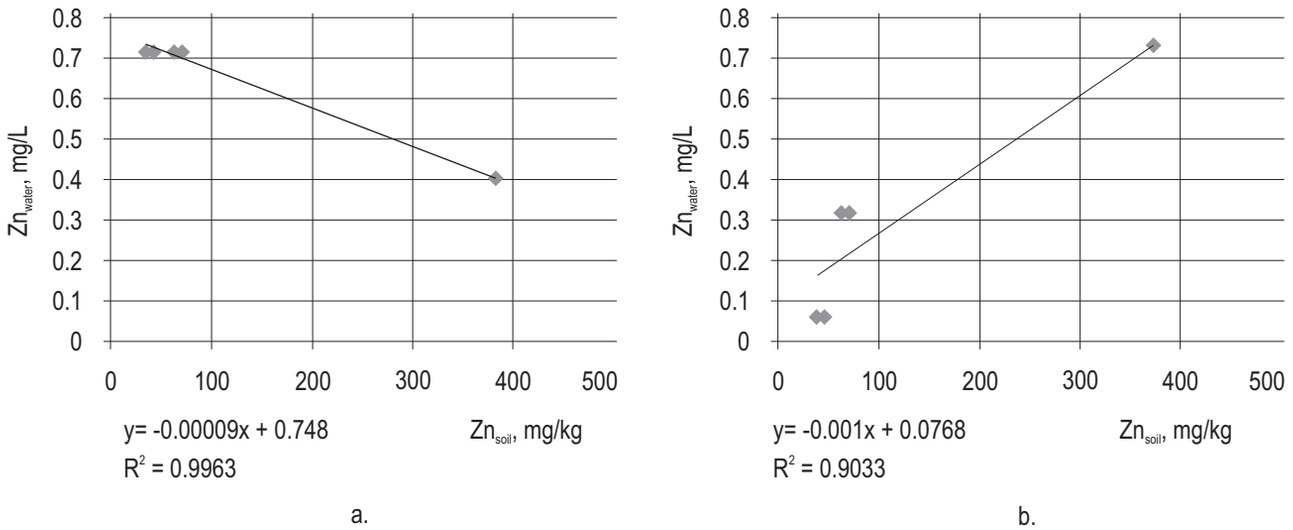

Figure 4. Liquid/solid correlations of Zn distribution between dam waters and adjacent soils in: a. December 2009; b. July 2010

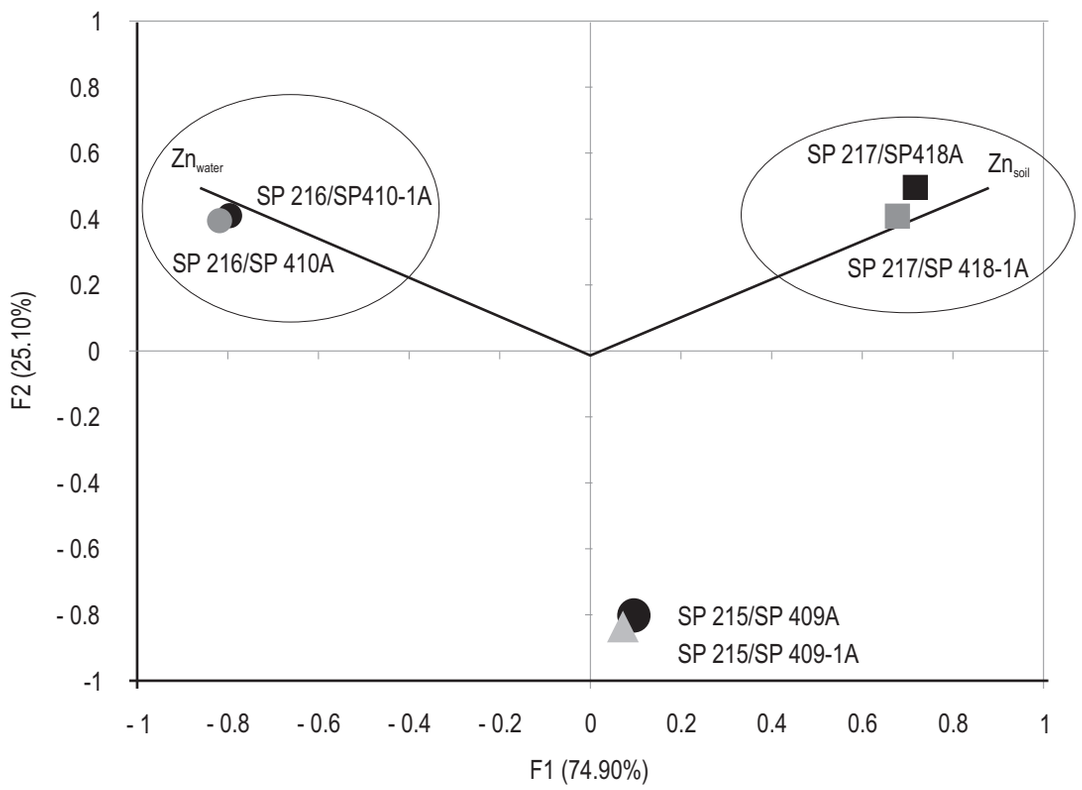

Figure 5. Scatterplot of the PCA case-wise factor scores for the surface water and soil samples

corresponding to the highest metal content in the adjacent soil samples. Considering the sandy nature of these soil samples, an assumption of probable heavy metal saturation is not substantiated, as heavy metal adsorption in sandy soils is negligible compared to clay soil types. (Srivastava et al, 1999) Thus, the registered elevated and excessive Zn content in SP 418A and 418-1A could be due to dissolution of Zn-enriched primary minerals during soil weathering. According to Jacquat et al. (2009), Zn in soils developed from the weathering of Zn-rich rocks is either linked to the inheritance of resistant Zn-bearing minerals or to dissolution of primary Zn minerals and subsequent redistribution of Zn into pedogenic phases.

Conclusion

A significant correlation between Zn contents in the analyzed dam water and soil samples from Chirpan Municipality was approved. The values of the corresponding regression coefficients

during December 2009 and July 2010 were R^2= 0.9963 and R^2= 0.9033, respectively. The PCA categorized the investigated sampling points into three classes. The deductions of the present study demonstrate the useful application of the statistical analysis of trace metal data towards providing evidence of soil and water contamination of the municipality under study. Further detailed chemometric analysis of the distribution of other heavy metals, as well as comparative assessment of the complex heavy metals loading of the surface water bodies and soils in other municipalities of Stara Zagora region are impending.

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Instruction for authors

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The manuscript should be structured as follows: Title, Names of authors and affiliation address, Abstract, List of keywords, Introduction, Material and methods,Results, Discussion, Conclusion, Acknowledgements (if any), References, Tables, Figures.

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Todorov N and Mitev J, 1995. Effect of level of feeding during dry period, and body condition score on reproductive performance in dairy cows,IXth International Conference on Production Diseases in Farm Animals, Sept.11 – 14, Berlin, Germany, p. 302 (Abstr.).

Thesis:

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