Multivariate Statistical Analysis of Trace Elements in Soil on Spoil Heap, South East Congo-Brazzaville

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Abstract: The aim of this research was to assess geochemical processes that govern trace elements migration in the soil near an abandoned ore treatment plant. For this purpose, the principal characteristics of soil and trace elements concentrations in a soil profile at the level of spoil heap have been determined. Trace metals content was determined for As, Cd, Co, Cr, Cu, Mo, Ni, Pb, Sb, Zn, Zr, V, U. Reference elements (Al, Fe, Mn and Ti) content were also determined. Trace elements with higher content were Pb, Zn, As, Cu and Sb. Their concentrations decreased in the soil profile and were from 11800 to 2000 mg/kg, 2400 to 1200 mg/kg, 1300 to 600 mg/kg, 270 to 90 mg/kg and 73.5 to 21.6 mg/kg, respectively for Pb, Zn, As, Cu and Sb. Multivariate statistical analysis which include factor analysis and hierarchical classification highlighted the role of soil texture, organic matter, Fe, Mn and Al in the vertical migration of trace elements. Among trace elements analyzed, lead showed a particular behaviour and was associated with sulphate and chloride. Its migration in the soil profile depends on the solubility of these two salts.

Key words: Metals · Soil · Migration · Statistical analysis

INTRODUCTION

The activities of a treatment plant of base metals (Pb, Zn and Cu) in Mfouati (South-East of Congo-Brazzaville) caused the rejection of great quantities of heavy metals in the environment. Between 1976 and 1988, the treatment plant treated at least 180,000 tons of ore of which lead content varied between 15 to 20%. The lead concentrate obtained was in the form of sulphide (PbS). At the present time, the treatment plant was abandoned. In the ore, metals are present in the form of sulphide (galena PbS, sphalerite ZnS, Chalcosine Cu_2S, Chalcopyrite $CuFeS_2$), carbonates ($PbCO_3$, $ZnCO_3$) and others more complex compounds such as hemimorphite $Zn_4Si_2O_7(OH)_2.H_2O$, lead chloroarseniate $Pb_4(PbCl)(AsO_4)_3$, vanadinite $(VO_4)_3Pb_4(PbCl_2)$, malachite $CuCO_3.Cu(OH)_2$. The environmental risks of trace elements in the soil are groundwater contamination, their biodisponibility and toxicity [1, 2]. Many geochemical studies on the soils highlighted the control of some characteristics such as pH, CEC and texture on the distribution of trace elements [3-7]. Other studies have related the retention of trace elements to clay and organic matter [8, 9] and sulphates [10].

Data analysis in geochemical studies is easier with statistical tools such as factor analysis and hierarchical classification which are exploratory techniques. This study was carried out in a mining area. Its purpose is to assess the concentration of trace elements such as As, Cd, Co, Cr, Cu, Mo, Ni, Pb, Sb, Zn, Zr, V, U and reference elements such as Al, Fe, Mn, Ti in a soil profile on the spoil heap, to determine by statistical analysis the soil components which control the vertical distribution and the geochemical processes associated to the migration of trace elements.

MATERIALS AND METHODS

The Study Area: The site retained for sampling was the spoil heap of a treatment plant which activity was stopped for more than twenty years. The ore treatment plant was built on the side of a hill located at 442 meters of altitude in Mfouati between 367178.59 - 370507.20 UTM south latitude and 9512957.94 - 9515800.06 UTM east longitude (Fig. 1). The annual rainfall varies from 1050 to 1650 mm. The soils are of ferralitic type, which have been derived from schisto-limestone.

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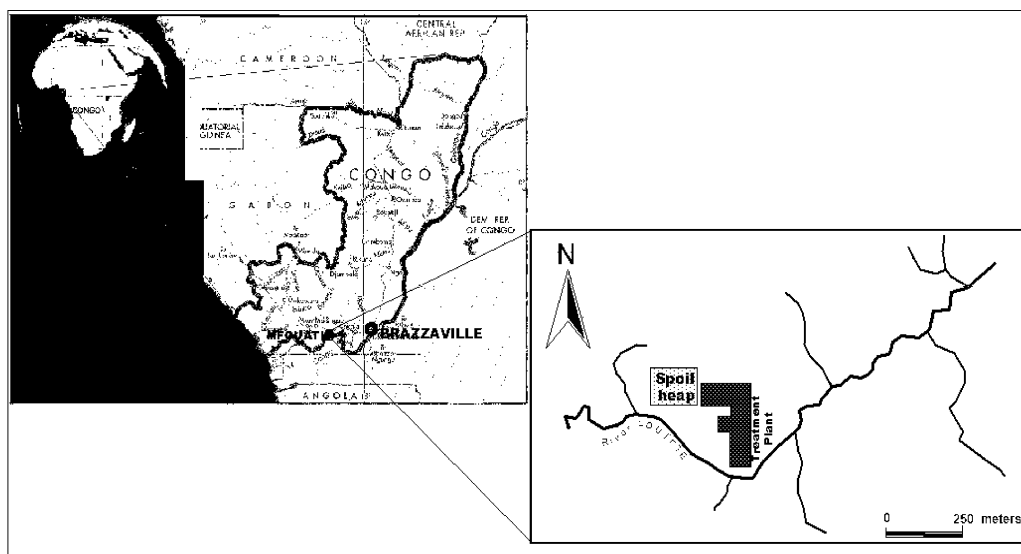
Fig. 1: Location of the soil heap in the study area

Sampling: A soil profile (1.50 meters of depth and 1 meter broad) was sampled on the spoil heap. The first 15 centimeters thickness road base present an accumulation of ore and was not sampled. A total of 54 samples from five soil depths were collected in August 2007 then divided into five composite samples E1 (12 samples), E2 (12 samples), E3 (12 samples), E4 (12 samples) and E5 (9 samples) at the depth 15 - 45 cm, 45 - 75 cm, 75 - 105 cm, 105 – 135 cm and 135 - 150 cm, respectively. The samples were collected with a polyethylene spatula to avoid the use of metal tools then stored in plastic bags.

Analytical Methods: In the laboratory, the soils samples were air-dried and passed through a 2-mm plastic siewe to move gravel and rocks and stored in new plastic bags. The samples were analyzed for heavy metals and major elements by ICP-MS (Thermo fisher) in the Service Central du CNRS, Vernaison, France. Briefly, 0.1g of each homogenized soil sample was digested by triacid attack (HF-HClO$_4$-HNO$_3$) in a Teflon vessel and heated in a microwave oven at 180°C for 10 min. The digested solution was diluted to a known volume with double distilled water and then it was analyzed for metals by ICP-MS. The content of anions such as F$^-$, Cl$^-$ and SO$_4^{2-}$ was determined by ionic chromatography and coulometric detection. Total sulfur was determined by LECO, the mass of the sample is 0.1 g. The principal properties of soil were determined, such as CEC (Metson method), TOC and organic matter (OM) (standard method NF ISO 10694), granulometry (five fractions without decarbonation; standard NF X 31 – 107). Soil pH was measured in the proportion 1:2 (soil/water) using a Hanna pH-meter with combined electrodes after calibration with two buffer solutions, pH 7 and 4. Statistical analysis was performed with STATISTICA 7.1 software [11] for the computation of correlation matrix, hierarchical cluster analysis (HCA) and factor analysis (FA), which represent a quantitative and independent approach of samples and variables classification in environmental studies. HCA is a powerful tool for identifying and selecting the homogeneous groups from the environmental data within a particular data set. This analysis encompassed a number of different algorithms and methods for grouping similar objects into respective categories. As a multivariate method, FA facilitates the reduction, transformation and organization of the original data by the use of intricate mathematical techniques. It created a new set of uncorrelated variables called factors, which are the linear combinations of the original ones with the same amount of information. The extracted factors reflect the main part of information of the data set. The first few factors included the largest part of the total variance and the interpretation of dominant factors was made by taking into account the highest factor loadings on chemical elements. Data were processed by means of R-mode factor analysis, applying the varimax-raw rotational technique. For the theoretical details of FA see for instance [12, 13].

RESULTS AND DISCUSSION

Physico-Chemical Properties of the Soil Profile: The principal characteristics of soil in the five samples of the profile are given in Table 1. The data show that the total sulphur concentration, total organic carbon (TOC)

Table 1: Principal physico-chemical properties of the soil profile

| Depth | Total sulphur (mg/kg) | pH | Granulometry 5 fractions (g/kg) | | | | | TOC (g/kg) | O.M (g/kg) | CEC (cmol/kg) |
			CS	FS	CSilts	FSilts	Clay			
15 - 45 cm	374	5.69	150	156	157	212	325	1.590	2.76	2.90
45 - 75 cm	263	6.10	126	142	141	258	333	1.510	2.61	2.91
75 - 105 cm	227	6.04	136	122	131	268	343	1.150	1.99	3.11
105 - 135 cm	190	6.25	132	107	133	275	353	1.140	1.98	3.30
135 - 150 cm	214	6.15	103	132	139	282	344	0.934	1.62	3.54

TOC = Total organic carbon; OM = organic matter; CEC = cation exchange capacity

CS = Coarse-Sand (0.2-2 mm); FS = Fine-Sand (0.05-0.2 mm); CSilts = Coarse-Silts (0.02-0.05 mm); FSilts = Fine-Silts (0.02-0.002 mm); Clay minerals (< 2 µm)
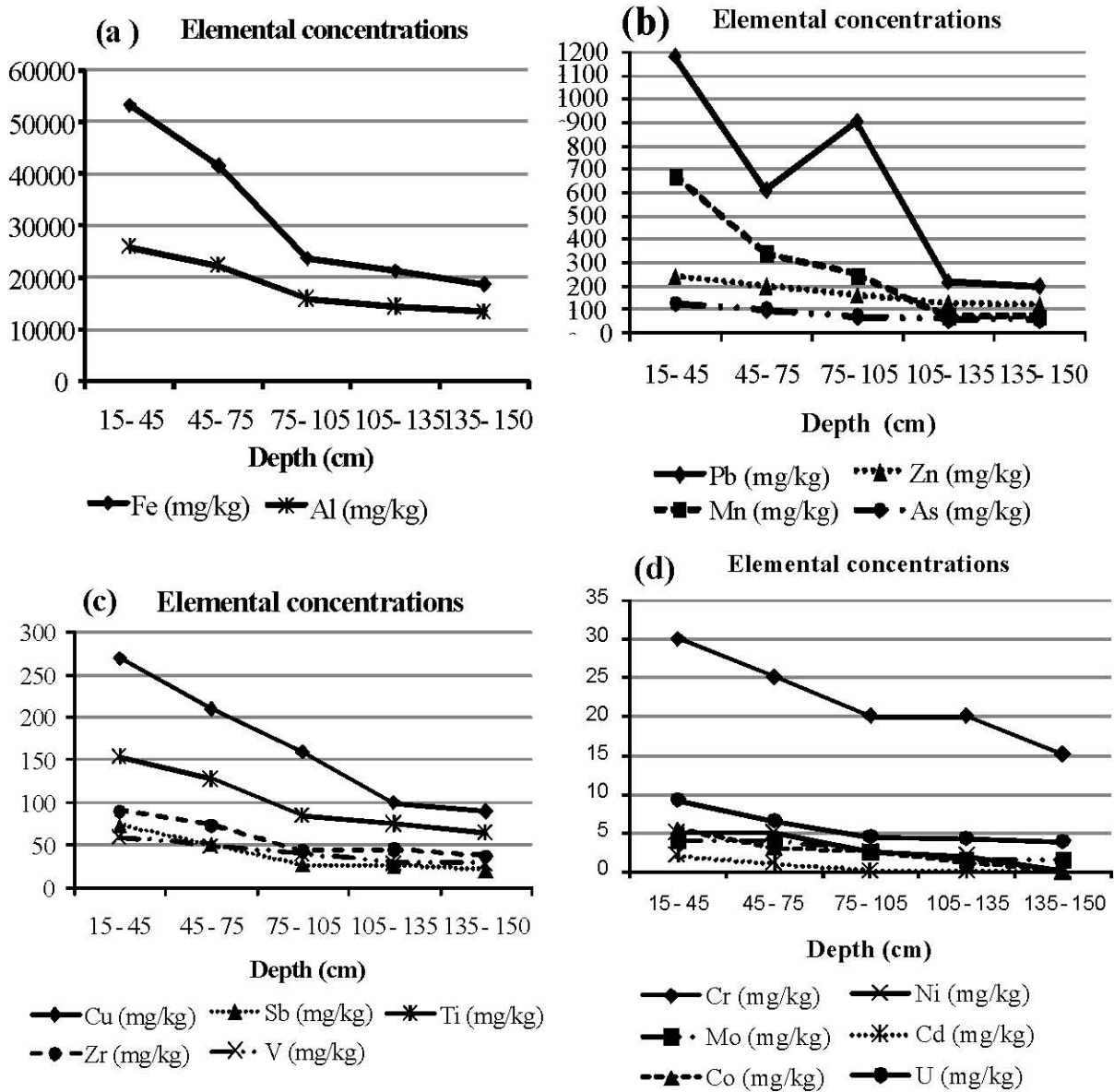

Fig. 2(a-d): Distribution of the elements in the soil profile
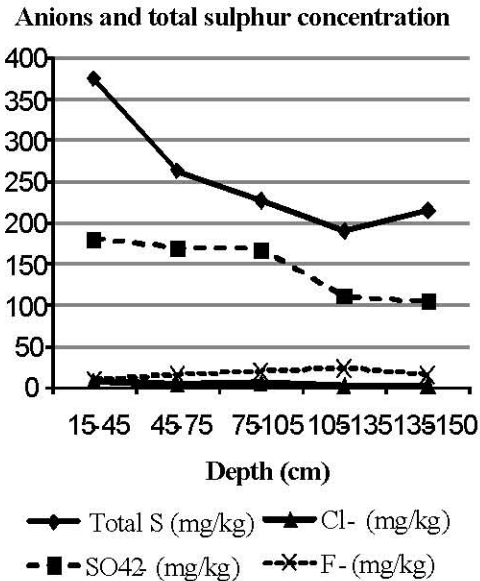
Anions and total sulphur concentration

Fig. 3: Concentration of some anions and total sulphur in the soil profile

and OM decreases when the depth increases. Soil pH and the particles of fine granulometry (fine silts and clay) increase with depth. In the profile, pH is acidic with a mean value of 6.04, ranging from 5.69 to 6.15. The increase in depth of pH which seemed to evolve to neutrality suggests that precipitation reaction and specific adsorption of trace elements on manganese and iron oxyhydroxides can become more important [14]. The total particulate organic carbon decreased from 1.59 to 0.934 g/kg and CEC increased from 2.90 to 3.54 cmol/kg with a mean value of 1.264 g/kg and 3.15 cmol/kg, respectively. Coarse sand (CS), fine sand (FS) and coarse silts (CSilts) fractions decreased in the soil profile from 150 to 103 g/kg, 156 to 132 g/kg and 157 to 139 g/kg, respectively. Fine silts (FSilts) and clay fractions increased in the profile from 212 to 282 g/kg and 325 to 344 g/kg, respectively. Composite soil sample E4 has the higher clay content (Table 1). A comparison of the five fractions showed that the percentage of coarse sand, fine sand and coarse silts decreased in the profile with range of 15 to 10% while the percentage of fine silts and clay increase from 21.2 to 28.2% and 32.5 to 34.4%, respectively.

Distribution of Trace Elements and Anions Content in the Soil Profile: The concentration of trace elements, along with Fe, Al and total sulphur, sulphate, chloride concentrations are presented in Fig. 2(a-d) and Fig. 3,

respectively. All the elements and anions concentrations decrease with depth. Descriptive statistics of trace elements in the soil profile are presented in Table 2. The lowest mean concentration among the trace elements belongs to Cd (0.90 mg/kg) and the highest to Pb (6220 mg/kg). Except the reference elements such as Al, Fe, Mn and Ti, the average abundance order of trace elements content in the soil profile is: Pb > Zn > As > Cu > Zr > V > Sb > Cr > U > Ni > Mo > Co > Cd. The comparison mean concentration of the analysed trace elements in the five sampled soil depths with mean worldwide values (Table 2) revealed higher Pb, Zn, As, Cu and Sb content and lower Mo, Ni, Cd, Co, Cr, V. The contents Pb, Zn, As, Cu and Sb in the soil profile largely exceeded the respective mean worldwide value. We concluded that the spoil heap presents a mineral contamination.

Correlation Analysis: The degree of association between two variables has been evaluated by calculating the coefficient of correlation [15]. Table 3 presents Pearson correlation coefficient for soil profile at the significant level of $p < 0.05$. For Cd which concentrations were lower than the detection limit of 1 mg/kg (samples E3, E4 and E5), a value equal to one-half of the detection limit was considered [16]. All trace elements present a strong positive correlation with Fe, Al and Mn, except lead. Ni is easily mobilized and precipitated in association with Fe and Mn in oxides and hydroxides [17]. The oxyhydroxides of Fe, Mn and Al also control the distribution and sorption of the trace elements in the soil profile [18]. Many studies have shown that fine particles in the soil control the distribution and the threshold of concentration for trace elements [19, 20]. Organic matter presents also a strong positive correlation with all trace elements except with lead and cadmium. A very marked negative correlation exists between the cation exchange capacity and Cu, Ni, Cr, V, Mo and Zn. Significant negative correlations were also observed between fine silts and clay with trace elements. On the other hand, coarse silts present a very marked positive correlation with Cd, As, Sb and U. Mineralogical study of the soil samples (not presented here) by XRD has shown that clay was mainly made up of talc which has not a great cation exchange capacity. Lead had rather a particular behaviour, a marked positive correlation with sulphate and chloride.

Table 2: Descriptive satatistics of soil profile trace elemets (mg/kg) with mean worldwide values

Element	Minimum	Maximum	Mean	SD	Mean worldwide data
Cu	90.00	270.00	166.00	75.70	14[a]
Zn	1200.00	2400.00	1700.00	500.00	62[a]
Ni	1.50	5.00	3.20	1.68	18[a]
Cd	0.50	2.00	0.90	0.65	3[a]
As	600.00	1300.00	840.00	304.96	4.7[a]
Co	0.50	5.50	2.50	1.97	6.9[a]
Cr	15.00	30.00	22.00	5.70	100[b]
Mn	700.00	6700.00	2810.00	2463.33	761[b]
V	30.00	60.00	42.00	13.04	135[b]
Mo	1.50	4.00	2.70	1.25	1.8[a]
Pb	2000.00	11800.00	6220.00	4267.55	25[a]
Zr	38.00	90.00	58.40	22.47	165[e]
Sb	21.60	73.50	40.10	21.85	2-10[f]
U	3.88	9.27	5.72	2.25	2.7[g]
Fe	18620.00	53130.00	31598.00	15008.00	32,000[c]
Al	13394.11	25835.3	18338.82	5426.75	67,000[c]
Ti	68.00	154.00	102.00	37.26	3,800[d]

Table 3: Pearson correlation coefficient matrix for soil profile parameters

	pH	CS	FS	CSilts	FSilts	Clay	O.M	CEC	SO_4^{2-}	Cl	Cu	Ni	Cd	As	Co	Cr	Mn	V	Mo	Pb	Zr	Sb	U	Zn	Fe	Al	Ti
pH	1.00																										
CS	-0.67	1.00																									
FS	-0.82	0.26	1.00																								
CSilts	-0.85	0.42	0.89	1.00																							
FSilts	0.94	-0.77	-0.78	-0.89	1.00																						
Clay	0.87	-0.45	-0.97	-0.87	0.87	1.00																					
O.M	-0.69	0.72	0.70	0.71	-0.86	-0.83	1.00																				
CEC	0.63	-0.77	-0.58	-0.50	0.76	0.75	-0.94	1.00																			
SO_4^{2-}	-0.74	0.72	0.64	0.47	-0.74	-0.79	0.81	-0.93	1.00																		
Cl	-0.97	0.80	0.68	0.71	-0.91	-0.78	0.68	-0.70	0.81	1.00																	
Cu	-0.87	0.73	0.82	0.77	-0.93	-0.93	0.94	-0.92	0.91	0.86	1.00																
Ni	-0.67	0.59	0.78	0.71	-0.81	-0.88	0.98	-0.93	0.83	0.64	0.94	1.00															
Cd	-0.91	0.65	0.85	0.96	-0.98	-0.89	0.85	-0.70	0.66	0.84	0.90	0.82	1.00														
As	-0.88	0.65	0.88	0.89	-0.96	-0.95	0.94	-0.84	0.79	0.82	0.97	0.93	0.97	1.00													
Co	-0.93	0.80	0.78	0.79	-0.97	-0.89	0.90	-0.87	0.88	0.98	0.98	0.87	0.93	0.96	1.00												
Cr	-0.78	0.82	0.68	0.76	-0.93	-0.82	0.98	-0.91	0.81	0.79	0.95	0.93	0.91	0.95	0.95	1.00											
Mn	-0.95	0.74	0.84	0.85	-0.98	-0.93	0.88	-0.83	0.85	0.93	0.98	0.86	0.95	0.97	0.99	0.93	1.00										
V	-0.87	0.69	0.85	0.80	-0.93	-0.95	0.94	-0.90	0.90	0.85	1.00	0.95	0.91	0.98	0.97	0.94	0.98	1.00									
Mo	-0.71	0.57	0.81	0.68	-0.80	-0.91	0.94	-0.93	0.90	0.69	0.96	0.98	0.79	0.92	0.89	0.89	0.88	0.96	1.00								
Pb	-0.90	0.80	0.64	0.58	-0.84	-0.78	0.72	-0.80	0.92	0.96	0.89	0.69	0.75	0.79	0.93	0.78	0.90	0.87	0.77	1.00							
Zr	-0.78	0.65	0.81	0.86	-0.92	-0.89	0.97	-0.85	0.75	0.73	0.94	0.96	0.94	0.98	0.92	0.97	0.93	0.95	0.92	0.70	1.00						
Sb	-0.85	0.68	0.84	0.89	-0.96	-0.91	0.95	-0.83	0.75	0.80	0.96	0.93	0.97	0.99	0.95	0.97	0.96	0.96	0.90	0.76	0.99	1.00					
U	-0.88	0.69	0.84	0.91	-0.98	-0.92	0.93	-0.81	0.75	0.83	0.96	0.90	0.98	0.99	0.96	0.96	0.97	0.96	0.88	0.78	0.98	1.00	1.00				
Zn	-0.86	0.72	0.82	0.80	-0.94	-0.93	0.96	-0.91	0.88	0.84	1.00	0.95	0.92	0.98	0.98	0.96	0.98	1.00	0.96	0.86	0.97	0.97	0.97	1.00			
Fe	-0.82	0.65	0.85	0.87	-0.94	-0.93	0.96	-0.86	0.78	0.77	0.97	0.96	0.95	0.99	0.94	0.96	0.95	0.97	0.93	0.75	1.00	0.99	0.99	0.98	1.00		
Al	-0.81	0.66	0.84	0.84	-0.92	-0.93	0.97	-0.89	0.82	0.77	0.98	0.97	0.93	0.99	0.94	0.96	0.95	0.98	0.96	0.77	0.99	0.99	0.98	0.99	1.00	1.00	
Ti	-0.82	0.66	0.84	0.84	-0.93	-0.93	0.97	-0.88	0.82	0.77	0.98	0.97	0.94	0.99	0.95	0.97	0.95	0.98	0.95	0.77	0.99	0.99	0.98	0.99	1.00	1.00	1,00

Hierarchical Cluster Analysis (HCA): Classification of the samples according to the soil depth obtained by K-means clustering (Ward's method, Euclidean distance) gave two clusters represented by Fig. 4. Cluster I consisted of samples of depths 15-45 cm and 45-75 cm (composite samples E1 and E2) and cluster II is represented by two subdivisions which are II_A (E3: depth 75 – 105 cm) and II_B (E4 and E5: depths 105 – 135 cm and 135 – 150 cm). Cluster I is characterized by higher trace elements content in the soil profile. Class II_A of cluster II contains the soil samples whose trace elements content is lower than that of the samples of cluster I, but higher than that of the samples in class II_B. Class II_B contains the lower trace elements content in the soil profile. HCA of trace elements and principal soil characteristics are presented in Fig. 5 and highlighted
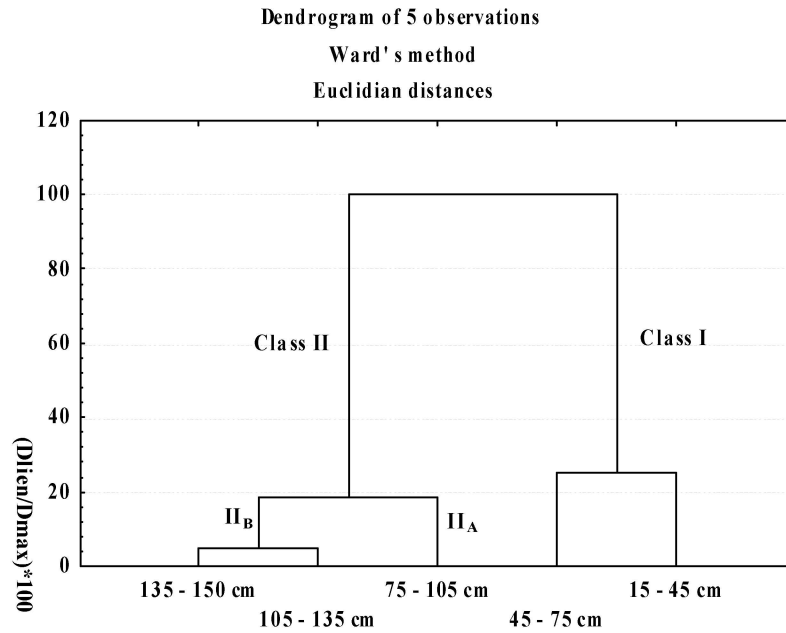
Dendrogram of 5 observations

Ward's method

Euclidian distances

Fig. 4: Dendrogram showing the relationship between composite samples at the different depths

Dendrogram of 30 variables

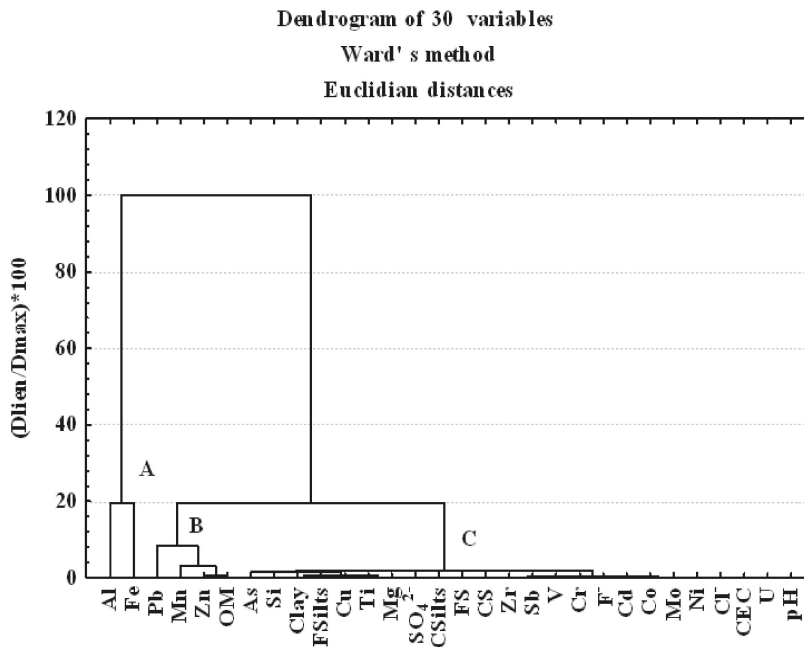**Ward's method**

Euclidian distances

Fig. 5: Dendrogram of soil properties, major elements, anions and trace elements In the soil profile

three classes: A, B and C. Class A is formed by Al and Fe which have a greater sequestering ability for trace elements. Class B contains Pb, Zn, Mn and organic matter. Mn steps in the same processes with Fe and Al. Organic matter has the capacity to complex trace elements [21]. Class C includes As, Cu, Cr, V, Sb, Zr, Ti, sulphate, chloride and the soil texture (five fractions).

Factor Analysis (FA): The data reduction was performed by factor analysis (extraction method: principal components). Kaiser criterion [22] which retains only factors with eigenvalues > 1 was used for determining how many factors to take in account or to ignore. Three factors have been retained which the eigenvalues were respectively 26.23, 1.71 and 1.26. The three factors

Table 4: Factors loadings (rotated) for major, trace elements and principal soil components

Variable	Factor 1	Factor 2	Factor 3
SO_4^{2-}	0.23	0.64	0.63
Cl^-	0.44	0.87	0.22
F^-	-0.91	-0.23	-0.27
Mg	-0.57	-0.48	-0.66
Si	-0.50	-0.61	-0.61
Cu	0.51	0.52	0.68
Ni	0.44	0.24	0.86
Cd	0.73	0.42	0.48
As	0.65	0.40	0.64
Co	0.52	0.64	0.56
Mn	0.62	0.58	0.52
V	0.56	0.48	0.67
Mo	0.45	0.31	0.80
Pb	0.37	0.83	0.37
Zr	0.58	0.30	0.75
Sb	0.63	0.38	0.66
U	0.65	0.43	0.61
Zn	0.53	0.48	0.70
Fe	0.61	0.35	0.71
Al	0.57	0.36	0.74
Ti	0.58	0.36	0.73
pH	-0.71	-0.67	-0.23
SG	0.01	0.83	0.48
SF	0.88	0.16	0.39
LG	0.89	0.23	0.33
LF	-0.63	-0.58	-0.48
A	-0.76	-0.32	-0.53
M.O	0.38	0.34	0.86
CEC	-0.16	-0.49	-0.85

account each for 87.43 % (Factor 1), 5.71 % (Factor 2) and 4.22 % (Factor 3) of the total variance. Factor loadings for the three retained eigenvalues are shown in Table 4. The distribution of factor loadings shows that the fractions of soil (fine sand and coarse silts) and Cd are strongly associated with Factor 1. Clay and pH load negatively on the Factor 1. Factor 2 incorporates lead, chloride and coarse sand. Factor 3 was associated with trace elements such as Ni, Cr, Mo, Zr, lithogenic elements (Fe, Al, Ti) and organic matter.

The first factor shows the importance of fine-sands and coarse-silts in the vertical migration of trace elements. Factor 2 characterizes in particular the vertical migration of lead which depends on the solubility of $PbCl_2$ and $PbSO_4$. Factor 3 was related to the importance of organic matter, Fe, Al on the distribution of trace elements in the soil profile. The strong positive correlation between Ti and trace elements on Factor 3 could suggest an adsorption process in the retention of metals.

These characteristics which govern the distribution of trace elements in the soil profile have been highlighted in many studies on soils [9, 13, 23] and sediments [24, 25].

Trace elements released from ores, the solubilization of sulphates of trace elements resulting from the oxidation of their sulphides [15], specific adsorption of trace elements on minerals and organic matter depend on the pH and soil composition [6, 20, 26]. These factors control the chemical speciation of trace elements.

In conclusion, in this study, the acidic nature of the soil, organic matter, Fe, Mn contents and soil texture controlled the vertical migration of trace elements in the soil profile, except for lead which the distribution was governed by the solubility of chloride and sulphate compounds.

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