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TRACE ELEMENTS IN ALLUVIAL SOILS AFFECTED BY WASTEWATER IRRIGATION

An industrial area at Shubra El-Khaima in the north of greater Cairo was selected to investigate the impact of industrial activities on soil trace element concentrations, as part of the assessment of the effect of wastewater irrigation on alluvial soils. The area studied was divided into four sectors according to its source of irrigation water and/or probability of pollution. Mineral content of clay and contamination of irrigation wastewater were the key parameters determining the trace element concentrations in these soils. Heavy metals, rare earth and lanthanide elements were determined using neutron activation analysis (NAA). Results, discussion, elemental correlations and conclusions are reported in detail.

1. INTRODUCTION

Trace elements have important effect on life processes. In nature, the concentrations of undesirable elements both in the soil solution and in natural waters are low. This situation can change drastically when the content of these undesirable elements in the soil is increased by several orders of magnitude in industrial areas.

The concept of soil pollution must be restricted to accumulation of heavy metals, etc., at a reactive level that is noxious for soil organisms and/or harms plant production [1]. Soil pollution may occur as a consequence of different types of input of unwanted substances such as disposal of sewage sludge, irrigation with polluted wastewater, using pesticides and intensive fertilization.

The distribution of trace elements in soils is influenced by several factors such as parent material, mineralogy, organic matter content, particle-size distribution, vegetation, and irrigation input. Element concentrations have been related to a few of these factors only. Most frequently, the effects of particle-size variations on element distribution have been neglected. Unfortunately, many studies are also limited to a few

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elements only. The distribution and behaviour of trace elements that are essential or toxic to plants and animals have been studied most intensively. Less information is available about the elements that are not as critical in agriculture or to human health. BROGOWSKI et al. [2] demonstrated significant effects of the particle-size distribution of several trace elements on their concentrations in soils. They found a general enrichment of the clay and silt fractions with trace elements. The determination of a number of trace elements in soils is difficult for several reasons, including very low concentrations of the elements of interest in the presence of other elements in large concentrations. It has been reported that nondestructive multi-element techniques suit perfectly to such purposes [3]. Among these techniques, NAA, X-ray fluorescence, and emission spectroscopic methods are noteworthy. NAA has also been successfully applied to a number of complex sample matrices [4]–[7].

The aims of this work are to establish a data-base for previously uninvestigated heavy metal and lanthanide concentrations and to investigate multi-elemental relationships resulting from different sources of irrigation wastewater on alluvial soils.

2. MATERIALS AND METHOD

2.1. PREPARATION OF THE SAMPLES

Four soils profiles were dug in industrial area in the north of greater Cairo at Shubra El-Khaima: soil sample (1) is a control soil irrigated with the Nile water, soil sample (2) from Bahteem was irrigated with sewage effluent, soil sample (3) from Mostorod and soil sample (4) from El-Marg were irrigated using industrial waste. Sixty soil samples from different soil layers (at 20, 40 and 60 cm depths) were collected, each in five replications representing each layer. The samples were air-dried and crushed to pass through a 200 mesh sieve.

2.2. NEUTRON IRRADIATION

About 0.1 g of homogeneous soil sample was packed in pure aluminum foil and prepared for irradiation, while an empty aluminum foil of known weight was subjected to irradiation in order to identify and subtract the background based on the γ-ray peaks of aluminum envelopes. A gold foil (0.004 g) was also prepared and rolled in separate aluminum sheet for flux monitoring. Also 0.1 g of standard reference material (soil-7) [8] was investigated to certify the accuracy of analysis. The irradiation time was 48 hours at the Nuclear Research Center First Reactor 2 MW (ET-RR-1). The neutron flux was 4.4×10^{12} n/cm$^2 \cdot$ s.

2.3. INSTRUMENTATION

After 72 hour-cooling time samples were analysed by gamma spectrometry using a high-resolution HPGe detector connected to 8192 multi-channel analyzer and PC analysis program through a suitable electronic system. The samples were positioned individually 10 cm in front of the detector and the accumulating time was 2 hours for good statistics. The HPGe detector had an efficiency of 30% and an energy resolution of 1.85 keV at 1332.5 keV. The multi-gamma-ray standard source MGS-4 was used to perform the energy and efficiency calibration of the detection system [9]. Selected γ-rays from ^{155}Eu, ^{57}Co, ^{113}Sn, ^{137}Cs, ^{54}Mn and ^{65}Zn were used for these measurements. Analytical measurements were repeated for each sample three times at a time interval of one month to follow the decay of short-lived nuclides.

3. RESULTS AND DISCUSSION

Separation of soil fractions was carried out by a standard method [10]. Table 1 shows selected soil characteristics as indication of soil variability. The properties of the soils tested varied, depending on the irrigation water/wastewater applied. It is clear from table 1 that the control soil, Bahteem and El-Marg are alkaline, but Mostorod is acidic. We also noticed that Mostorod soil sample is characterized by higher values of electric conductivity (EC), organic matter content (OM) and cation-exchange capacity (CEC) than the other samples.

Table 1

Some selected soil characteristics

Soil layers (cm)	pH	Organic matter (%)	EC (ds/m.)	CEC (meq/100 g soil)	Sand (%)	Silt (%)	Clay (%)
Control							
20	7.50	3.45	4.49	27.20	28.68	22.41	48.91
40	7.95	2.72	2.33	25.20	24.53	27.25	48.22
60	7.90	0.73	2.26	25.20	29.60	25.60	44.80
Bahteem							
20	7.73	4.80	4.05	32.16	27.20	20.80	52.00
40	7.89	2.81	1.65	30.20	25.80	27.56	46.64
60	7.94	2.54	1.77	30.20	28.61	27.30	44.09
Mostorod							
20	6.74	7.99	8.43	37.44	31.49	24.31	44.20
40	6.94	6.99	6.01	31.20	29.87	25.05	45.08
60	6.99	6.90	5.72	29.74	27.68	28.93	43.39
El-Marg							
20	7.99	4.17	2.15	30.82	29.09	26.59	44.32
40	7.88	2.18	2.03	30.82	27.47	27.73	44.80
60	7.91	2.09	2.52	29.60	28.11	23.97	47.92

*EC – electric conductivity, CEC – cation exchange capacity.

Using X-ray diffraction we evaluated the internal structure of the different soil samples as shown in table 1.

The elemental constituents of the soil samples under investigation were estimated by means of the activities induced by (n, γ) reaction. Gamma-rays emitted were identified according to the energies of the well-resolved γ-ray lines taking into consideration that some of the activation products could omit more than one γ-ray line. To confirm the accuracy of the analytical system, the results for some elements in the present study, which appear to be well resolved, are compared with the certified values for soil-7 as shown in table 2. The errors represent the combined procedural and statistical uncertainties. An excellent agreement within limits of error has been found between the certified values and the data obtained by neutron activation analysis (NAA).

Table 2

A comparison between the present work and certified values for the standard soil-7

Element	Present work concentration (ppm)	Certified values (ppm)
Sc	8.50±0.42	8.3
Cr	57.56±2.88	60.0
Co	8.07 ±0.40	8.9
Cs	5.14±0.25	5.4
La	27.11±1.35	28.0
Sm	5.04±0.25	5.1
Eu	0.97±0.04	1.0
Hf	5.16±0.25	5.1

The concentrations of trace elements in the control soil are within the normal ranges for alluvial soils [11], [12]. Table 3 shows mean concentrations of heavy metals and their distribution in the soil profile. The content of heavy metals (Sc, Fe, Zn, Sb, Cs, Ba, Hf and Au) and rare earths (La, Ce, Eu, Tb, Yb, Ta and Pa) in the soils tested varied, depending on the source of origin of metals applied as contaminants in irrigation water/wastewater. Figure 1 shows a γ-ray spectrum of Mostorod soil.

The scandium (Sc) content is lower in the contaminated soil (especially in Mostorod and El-Marg soils) compared to iron (table 3). Normal concentrations of this element were reported worldwide to be 5–50 ppm with grand value of 7 ppm [13].

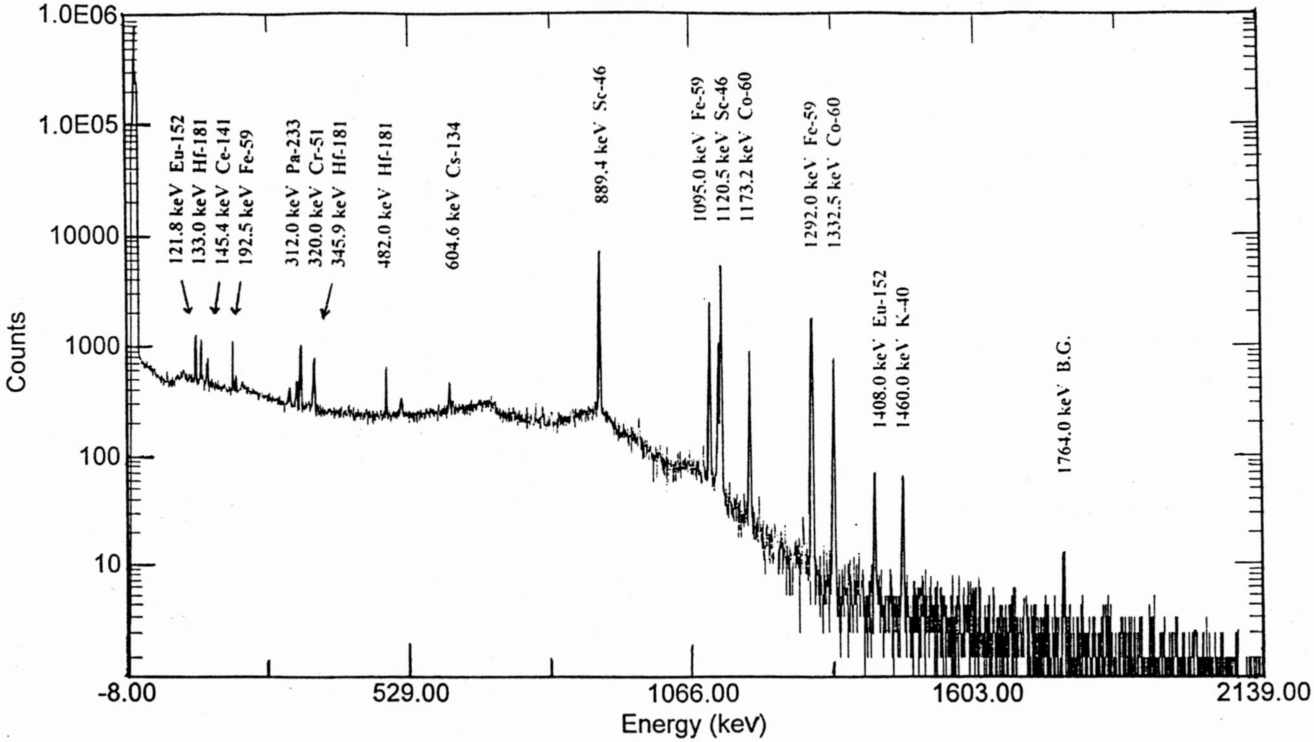

Fig. 1. A gamma-ray spectrum of Mostorod soil

Table 3

Distribution of heavy metals in soil profiles (ppm)

Element	Soil depth (cm)	Control	Bahteem	Mostorod	El-Marg
Sc	20	23.0	23.0	15.0	17.8
	40	21.0	27.0	8.0	16.9
	60	23.0	19.0	10.4	19.8
Fe	20	90632	92983	77941	74172
	40	82074	109532	42865	66150
	60	98010	69048	62308	80638
Co	20	41.5	40.3	29.2	31.9
	40	37.1	48.1	17.2	28.8
	60	42.3	32.6	23.9	34.7
Zn	20	249	400	733	265
	40	240	240	631	227
	60	330	250	560	237
Sb	20	1.7	1.9	6.8	1.1
	40	1.4	2.4	6.1	1.1
	60	1.7	0.7	8.0	1.3
Cs	20	6.9	9.2	11.5	2.4
	40	6.9	9.2	9.2	2.0
	60	4.6	6.9	6.9	2.1

The iron content (Fe) varied between soil layers and soil profiles in the range from 4.2 % up to 10.9%. Recently, the total content of iron in contaminated alluvial soils of Monofia Governorate and in the surface layer of alluvial soils in El-Saff region due to the irrigation with liquid industrial wastes were 4.7% [14] and 16.6% [15], respectively. We conclude that iron concentration in the samples under present investigation is in the normal range (the common range of 7000–550000 ppm) [12]. Stepwise regression analysis of the dependence of soil chemical properties on iron content in soil is represented by a significant linear model as follows:

$$\text{Fe content} = 1931 \times \text{sand\%} + 994 \times \text{clay\%}$$

$$- 3818 \times \text{OM\%} + 2291 \times \text{CEC} - 53855, \quad (R^2 = 84.58). \qquad (1)$$

The cobalt (Co) content in soils varies from 17.2 to 48.1 ppm depending mainly on the parent materials from which they derived, even though there are also differences with depth in the soil profile and between the soil types derived from a common parent material due to pedological processes. Within a given soil profile, cobalt is generally concentrated in the horizons rich in organic materials and clays. Usually higher cobalt contents in surface soils are observed in arid and semi-arid regions [16]. The normal level of cobalt in unpolluted alluvial soils of the Nile Delta ranged between 3.7 and 5.5 ppm with an average of 4.7 ppm for total content [17]. The cobalt concentrations in contaminated Egyptian alluvial soils are only roughly investigated. EL-LEITHI [18] studied the effect of industrial activities on the soils of the Nile Delta. He found that the total content of cobalt in these soils ranged between 11.2 and 36.1 ppm with an average of 23.7 ppm. EL-GAMAL [19] found that the total cobalt ranged from 30.36 to 41.40 ppm with an average of about 36.5 ppm in El-Gabal El-Asfar soil irrigated with sewage sludge for several years. The normal cobalt content in surface soils usually ranges from 1.0 to 40.0 ppm, with the highest frequency in the range of 3.0–15.0 ppm. Stepwise regression analysis of the dependence of soil OM% on cobalt content in soil and soil chemical properties is represented by a negative significant linear model as follows:

$$\text{Co content} = 43.37 - 2.38 \times \text{OM\%}, \quad (R^2 = 81.90). \tag{2}$$

Relatively high concentrations of zinc (Zn) in surface layers may be due to soil contamination by irrigation with water/wastewater. In control soil, zinc levels range between 330 and 250 ppm, while other soils showed values ranging from 227 to 733 ppm, with the highest values in all layers of the Mostorod profile (table 3). Recently, the total zinc content in Fayoum soils ranged from 16 to 216 ppm with an average of 91 ppm [20], while that of the alluvial soils collected from Giza, Kafr El-Shiekh and Moshtohor ranged from 10 to 120 ppm with an average of 95.0 ppm [21], and that in the unpolluted soils of the Nile Delta ranged between 81.0 and 101.0 ppm with an average of 92.0 ppm [17]. The concentrations of zinc in the samples under present investigation are higher than those in earlier investigations (common range from 10 to 300 ppm with an average content of 50 ppm) [12], which may reflect the industrial impact on the soil of the area tested. Stepwise regression analysis of the dependence of soil chemical properties and zinc content in soils is represented by a significant linear model as follows:

$$\text{Zn content} = 4.99 \times \text{sand\%} - 11.4 \times \text{clay\%} + 71.1 \times \text{OM\%} + 515.6, \quad (R^2 = 83.24). \tag{3}$$

Antimony (Sb) content followed a similar trend to zinc. Soil from Mostorod showed a remarkable increase in these metals through the soil profile. Stepwise regression analysis of the dependence of soil chemical properties on antimony content in soil is represented as follows:

$$\text{Sb content} = 0.059 \times \text{soil depth} + 1.14 \times \text{OM\%} - 4.02, \quad (R^2 = 88.43). \tag{4}$$

The concentration of caesium (Cs) ranges from 2 to 11.5 ppm, the highest values occur in Mostorod. Soil from El-Marg shows much lower concentration since it is usually irrigated with brackish water (saline drainage water) rich in sodium and potassium which may replace caesium. Stepwise regression analysis of the dependence of soil chemical properties on caesium content in soil is represented by a significant linear model as follows:

$$\text{Cs content} = 0.412 \times \text{sand\%} + 0.179 \times \text{clay\%} + 0.9 \times \text{OM\%} + 0.309 \times \text{CEC} - 22.42, \quad (R^2 = 81.29). \tag{5}$$

Table 4

Lanthanide total content in soil profiles as affected by soil contamination with industrial wastewater (ppm)

Lanthanide	Soil depth (cm)	Control	Bahteem	Mostorod	El-Marg
Ce	20	44.0	43.1	18.8	33.4
	40	36.0	46.3	11.9	27.0
	60	23.0	19.1	13.9	4.2
Eu	20	1.7	1.6	1.0	1.3
	40	1.5	2.1	0.5	1.2
	60	1.6	1.3	0.9	1.4
Tb	20	1.5	2.0	1.6	1.1
	40	1.2	1.1	0.7	0.8
	60	0.9	2.2	1.1	0.9
Yb	20	5.0	4.1	4.9	4.4
	40	5.0	5.7	2.2	3.0
	60	4.9	3.3	2.9	2.8
Hf	20	9.6	10.4	8.6	8.7
	40	9.4	11.6	5.2	7.8
	60	9.9	6.8	6.2	11.6
Ta	20	3.9	2.7	3.0	2.3
	40	2.9	3.5	1.4	2.5
	60	3.4	2.3	1.6	2.2
Pa	20	4.9	6.8	7.0	6.2
	40	6.4	8.6	4.4	5.3
	60	7.6	6.2	3.6	7.4

Cerium (Ce) content varied between 4.2 and 46.3 ppm with an average of 26.7 ± 3.8 ppm. Surface layers of soil tend to have higher cerium levels (table 4). The proportion of cerium in the lanthanides being analyzed is high (42–61%) (table 5). ESSER et al. [22] have indicated that cerium appears in high amounts in clay minerals. Stepwise regression analysis of the dependence of soil chemical properties on cerium content in soil is represented by significant linear model as follows:

$$\text{Ce content} = 1.38 \times \text{silt\%} + 4.47 \times \text{clay\%} - 2.56 \times \text{OM\%} + 1.15 \times \text{CEC} - 236, \quad (R^2 = 74.8). \tag{6}$$

Table 5

Lanthanides proportions in soil profiles as affected by soil contamination with industrial wastewater

Lanthanides	Soil depth (cm)	Control	Bahteem	Mostorod	El-Marg
Ce	20	0.62	0.61	0.42	0.58
	40	0.58	0.59	0.45	0.57
	60	0.45	0.46	0.46	0.61
Eu	20	0.02	0.02	0.02	0.02
	40	0.02	0.03	0.02	0.03
	60	0.03	0.03	0.03	0.02
Tb	20	0.02	0.03	0.04	0.02
	40	0.02	0.02	0.03	0.02
	60	0.02	0.05	0.04	0.01
Yb	20	0.07	0.06	0.11	0.08
	40	0.08	0.07	0.08	0.06
	60	0.10	0.08	0.10	0.04
Hf	20	0.14	0.15	0.19	0.15
	40	0.15	0.15	0.20	0.16
	60	0.19	0.17	0.21	0.17
Ta	20	0.06	0.04	0.07	0.04
	40	0.05	0.05	0.05	0.05
	60	0.07	0.06	0.05	0.03
Pa	20	0.07	0.10	0.16	0.11
	40	0.10	0.11	0.17	0.11
	60	0.15	0.15	0.12	0.11

Regression analysis and stepwise regression analysis of the dependence of soil chemical properties on the content of various elements in soil led to these significant linear empirical equations (1)–(6) obtained using statistical methods. The equations obtained are useful only under the same conditions of the study. The content of organic matter (OM) and the cation exchange capacity (CEC) of the soil are significantly affected by water/wastewater irrigation. R^2 is the significance of each equation allowing us to predict the value of the variable tested.

Figure 2 presents the total content of heavy elements in soil profiles as affected by contamination by water/wastewater irrigation for different soil depths.

Although the lanthanides constitute a group of chemically very similar elements, their concentrations varied, depending on type of soil and soil layer tested (table 4).

Lanthanide proportions P_i were calculated from the equation:

$$P_i = C_i/\Sigma\text{REE}\,, \tag{7}$$

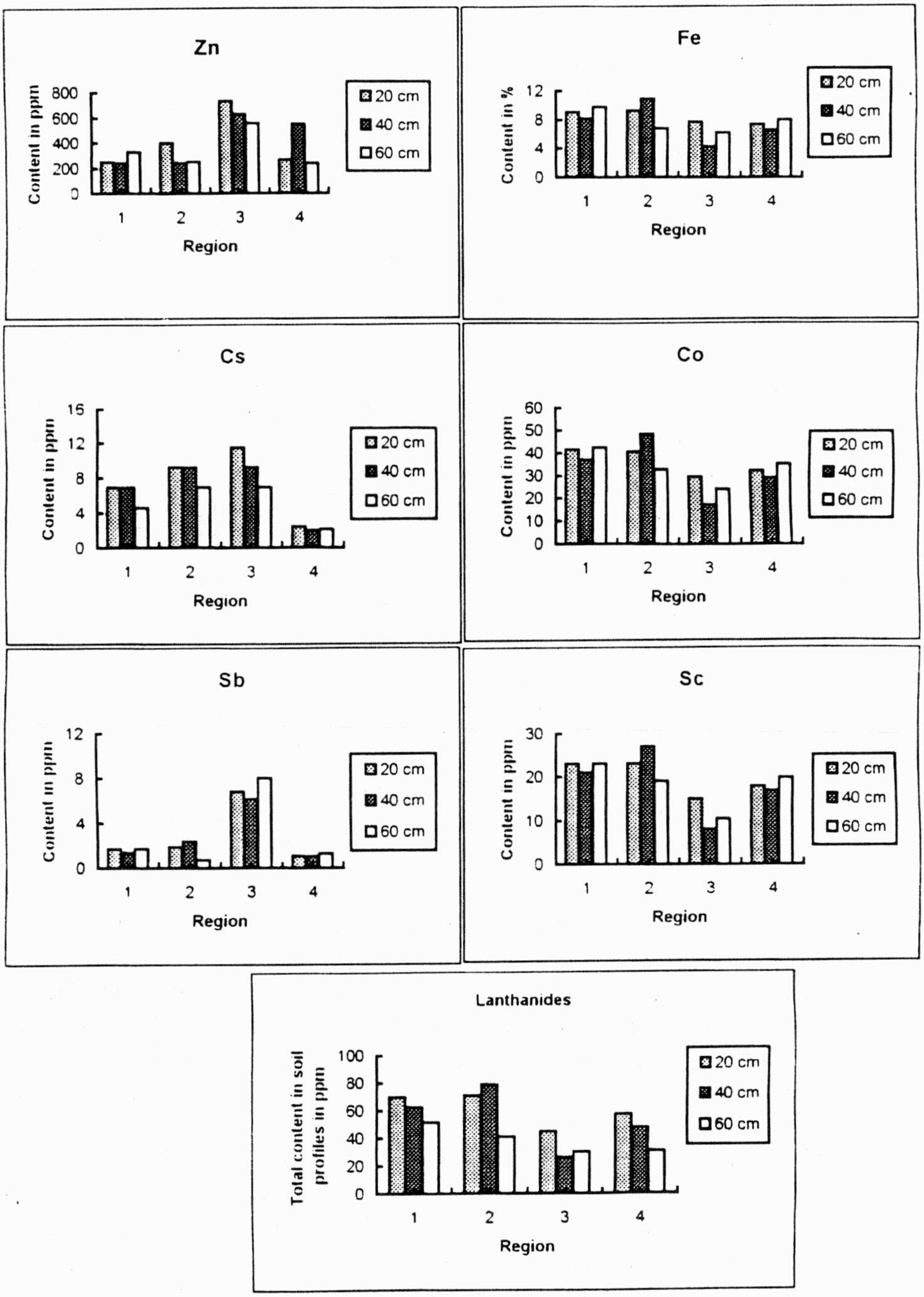

Fig. 2. Total contents of heavy metals and lanthanides in soil profiles (control, Bahteem, Mostorod and El-Marg in regions 1, 2, 3 and 4, respectively) as affected by contamination for different soil depths

where C_i is the concentration of the lanthanide i and ΣREE is the sum of the lanthanide concentrations in rare earth elements (REE). The proportions of lanthanides (equation (7)) vary considerably, depending on the elements and soil tested (table 5).

As shown in table 4, the other lanthanide being tested showed low levels with less variability in soils at any soil layer. It is noticed that the total content of the lanthanides (sum of the metals tested) was relatively similar in the different soil samples (figure 2). The Bahteem surface and subsurface layers accumulated the highest lanthanide amount, while the Mostorod soil samples showed the lowest concentrations of lanthanides. The lowest relative proportions were noticed for europium, terbium and tantalum (table 5). This may suggest that lanthanides are accumulated in different layers of soil due to soil pollution. Generally, the soils have been contaminated by trace elements, because they are irrigated with water/wastewater [23], which complicates the interpretation of the natural distribution of the elements.

4. ELEMENTAL CORRELATION

The occurrence of some metals is correlated with the occurrence of other specific metals which may be due to their origin and geochemistry. The correlation between metals is a diagnostic tool used in geochemistry to find relation between metals and factors affecting their levels. The analysis is done using computer software (MSTAT).

Table 6

Simple correlation coefficients for heavy metals and REE in soils

	Zn	Co	Cs	Sc	Sb	Ce	Eu	Tb	Yb	Hf	Ta	Pa
Fe	–0.45	0.97	IS	0.93	–0.45	0.64	0.94	IS	0.83	0.88	0.85	0.76
Zn		–0.61	0.64	–0.69	0.91	–0.41	–0.70	IS	IS	–0.51	IS	IS
Co			IS*	0.98	–0.60	0.69	0.99	IS	0.79	0.86	0.85	0.72
Cs				IS	0.56	IS	IS	0.43	IS	IS	IS	IS
Sc					–0.72	0.69	0.98	IS	0.73	0.86	0.82	0.74
Sb						–0.41	–0.66	IS	IS	–0.52	–0.42	–0.46
Ce							0.71	IS	0.74	0.44	0.69	IS
Eu								IS	0.74	0.85	0.81	0.68
Tb									IS	IS	IS	IS
Yb										0.61	0.88	0.59
Hf											0.67	0.80
Ta												0.54

* IS – insignificant.

High correlations between the heavy metals tested and lanthanides were obtained (table 6). Iron showed positive significant relations with cobalt, scandium, cerium, europium, ytterbium, hafnium, tantalum and protactinium. It is a well known fact that Fe-oxides and other Fe-compounds in soil have a sorptive capacity allowing them to retain other cations in the soil solution. However, zinc was correlated positively with caesium and antimony, and negatively with cobalt, scandium, cerium, europium and hafnium. The correlation between cobalt and scandium, cerium, europium, ytterbium, hafnium, tantalum, protactinium was the highest. Caesium was significantly correlated only with antimony, and a similar behaviour was exhibited by terbium. The highest significant correlation between scandium and lanthanides can be attributed to their common enrichment in heavy minerals in the very fine sand fraction. The high correlations between the light and intermediate lanthanides (cobalt, scandium and hafnium) are primarily due to their common enrichment in clay.

5. CONCLUSIONS

In conclusion, it is clear that the NAA technique is very useful in detecting the trace elements in soils for environmental assessment as well as the amount of heavy metals in the surface soil layers in the area studied. This amount is markedly increased due to the continuous irrigation of soil with water/wastewater. Soil irrigated with industrial water/wastewater accumulates more heavy metals than soil irrigated with sewage effluent. In the Mostorod region, the concentrations of zinc, caesium and antimony are higher than in the other regions due to parent material, mineralogy, organic matter content and water/wastewater irrigation. The transfer of such heavy metals as zinc, caesium and antimony is of particular importance because of the suspected toxic carcinogenic properties of these elements. Heavy metals and rare earth elements are related to soil chemical properties.

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MIKROELEMENTY W GLEBACH ALUWIALNYCH NAWADNIANYCH ŚCIEKAMI

Przemysłowy obszar Shubra El-Khaima w północnym Kairze został wybrany do badania wpływu działalności przemysłowej na stężenie mikroelementów w glebie. Badania te m.in. pozwalają określić wpływ, jaki mają ścieki służące do nawadniania na gleby aluwialne. Badany obszar podzielono na cztery

sektory w zależności od źródła wody do nawadniania i/lub możliwości skażenia gleby. Podstawowymi parametrami, które decydowały o stężeniu mikroelementów w glebie, były: substancja nieorganiczna iłu i zanieczyszczenie spowodowane nawadnianiem. Metale ciężkie i lantanowce oznaczono metodą aktywacyjnej analizy neutronowej.