Impact of cropping periods on micro and heavy metals availability in El-Gabal El-Asfar soils irrigated with sewage water

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Abstract

The current research aimed at investigating the consequences of irrigation with wastewater of El-Gabal El-Asfar drain on the bioavailability of heavy metals (HM) on the long run. To achieve such goal, seventeen farms were selected from El-Gabal El-Asfar area to represent soils of different cropping periods ranging from <5 up to 70 years. Soils were analyzed for DTPA extractable Fe, Mn, Zn, Cu (micronutrients) and Co, Pb, Ni and Cd for three successive seasons. The highest DTPA extractable heavy metals were in soils of long cropping period except for Co which did not follow a certain trend patterns of DTPA-extractable heavy metals (except for Co) as affected by the cropping periods were identical confirming that the applied sewage water was the main cause of heavy metal contamination heavy metals in soils. DTPA extractable micronutrients were calculated on a logarithmic basic and assumed to be log-normally distributed. The central tendency and variation of data, expressed as the geometric means (GM) and geometric deviations (GD), were determined. The range of concentrations for the calculated GM/GD² and GMxGD² ('baseline' values) were markedly wider than the observed concentrations for all heavy metals except for Ni. The results reflect the impact of the anthropic activity on Ni concentration in soil. Overall, DTPA- extractable heavy metals were significantly correlated with the cropping period, soil organic carbon, soil pH, soil and EC, but no significant correlation with CaCO₃. No significant correlations were detected between DTPA extractable Co and any of the studied parameters.

Keywords: heavy metals; El-Gabal El-Asfar; 'baseline' values; soil chemical characteristics

Introduction

Egypt is suffering from water scarcity (El Kharraz et al., 2012), which is estimated by less than 1000 m³/capita/year.(**Rijsberman**, 2006). limited available water resources (El-Sadek, 2010), water scarcity could have negative consequences on the current and future sustainability of the country (Iglesias et al., 2007). The growing fears of water crisis caused the Egyptian governments to use alternative resources of water for irrigation (Abdel-Kader and Abdel-Rassoul, 2010; Mustafa et al., 2013; Ali et al., 2016), to meet the intensified demands for water (Pereira et al., 2002; Bixio et al., 2006). Wastewater, which are produced in large quantities from different sources such as domestic, industrial and commercial ones (Qadir et al, 2010) can be used in irrigation purposes (Hamilton et al., 2007). They can increase available water budget and in many cases enriche soils with plant nutrients (Mohammad and Mazahreh, 2003), that can increase soil fertility and improve the growth of plants (Lubello et al., 2004; Selim, 2008). However, such wastewater can also bring many contaminants to soil i.e. polycyclic aromatic hydrocarbons (Khan et al., 2008) and microbial microorganisms (Petterson et al., 2001; Mara et al., 2007) beside of the heavy metals (Abbas et al., 2007; Li et al., 2009; Gupta et al., 2010), which can be taken up by the grown plants and find their way to the food cycle through the edible parts of the plant (Ibrahim et al., 2016; Hashim et al., 2017). This causes serious environmental and health risks on the long-term (Mapanda et al., 2005; Sridhara Chary et al., 2008; Abdelhafez et al., 2015; Abdel-Salam et al., 2015). Accordingly, proper management of wastewater would decrease such hazards when used for irrigation (Rusan et al., 2007).

Farm (Abdel-Shafy and Abdel-Sabour, 2006). For more than 80 years, the levels of heavy metals (HM) increased progressively in the soil of El-Gabal El-Asfar (Abdel-Shafy and Abdel-Sabour, 2006; El-Motaium et al., 2009; Holah et al., 2010; Singh et al., 2009). However, according to Waly et al., (1987), El-Hassanin et al. (1993) and El Sayed (2003), HM did not exceed the toxic levels in soil. A number of researchers, have referred to the soils of El-Gabal El-Asfar farm as polluted ones with heavy metals (HM) recording concentrations exceeding the permissible levels (Yassen et al., 2006; Abbas, 2007, Abdel-Rahman, 2014, Hashim, 2017). These contaminants persist in soil and do not undergo biodegradation (Greger, 2005). They their toxicity increases with time. Total contents of heavy metals in soil are poor indicators of their bioavailability (Alloway, 2013) which cause more conflicting conclusions about their toxicity (Abbas and Abdelhafez, 2013), especially in the light textured soils of El-Gabal El-Asfar. Ammonium bicarbonate diethylene triamine pentaacetic acid (AB-DTPA) is a recommended extractant for the available fractions of these heavy metals (Soltanpour, 1985). The AB-DTPA-extractable forms of HM in soils of El-Gabal El-Asfar were correlated positively significantly with their total contents (Abdel-Hameed et al., 2000).

Most researches indicated that the bioavailable forms of heavy metals are the most effective forms on the equilibrium among metal ions in soils that affect plant growth. Most chemical reactions in soils depend on the bioavailable forms of the elements. Therefore the current research is aimed at investigating the consequences of irrigation with wastewater of El-Gabal El-Asfar drain on the bioavailability of heavy metals rather than their total contents on the long run. Soils of different cropping periods with four the micronutrient of Fe, Mn, Zn and Cu and four non-nutrients of Co, Pb, Ni and Cd to fulfill the purpose of this study.

Materials and Methods

The study area is located at El Gabal El Asfar area, Egypt. at latitudes 30° 13 9" & 30° 16' 20" N and longitudes 31° 22' 10" & 31° 24' 10" E. This area was amended annually with sludge incorporated within the surface soil layer. The source of irrigation water for plants grown in this area was the sewage

effluent. Seventeen farm were selected from El Gabal El Asfar area to represent soils of different cropping histories ranged from <5 up to 70 years. Soil samples were collected from the upper (0-20 cm) surface layer of the investigated areas, during the winter season of (2014- 2015), the collected soil samples were air dried, crushed with wooden mallet, and sieved through a 2-mm sieve. Samples were analyzed for their chemical and physical properties as outlined by Page et al. (1982) and Gee and Bauder (1986). The results are presented in Table 1. Two other samples representing short time periods were collected from each of the investigated site with 6month time interval between each successive sampling i.e. during the summer season of 2015 and the winter season of 2015-2016. The extractable available presumably forms of Fe, Mn, Zn, Cu, Co, Pb, Ni and Cd were extracted (Lindsay and Norvell 1978). Then the concentration of Fe, Mn, Zn, Cu, Co, Pb, Ni and Cd were determined using atomic absorption spectrophotometer model: Atomic Absorption Spectrometer (AAS)- Hydride/Graphite system, Solaar MQZ_{environmental}, Thermo Electron Corporation England. The obtained data was statistically analyzed according to (Sparks et al. 1996)

Table 1. Chemical and physical characteristics of the investigated location soils of El-Gabal El-Asfar

	Period of Location			Soil	Soil	SOC,	CaCO ₃ ,	Particle size			Textural class
No	land use			pН	EC	g kg ⁻¹	$g kg^{-1}$	distribution, %			
	(year)	Latitude	Longitude	e				sand	silt	clay	_
1	5	30.25	31.36	7.55	2.37	10.10	2.30	97.28	1.95	0.77	sand
2	8	30.23	31.40	7.49	2.32	10.70	2.38	94.75	4.09	1.16	sand
3	10	30.28	31.37	7.35	2.26	12.10	3.20	98.08	0.22	1.70	sand
4	14	30.22	31.38	7.33	2.21	14.10	6.72	95.13	3.16	1.71	sand
5	16	30.26	31.37	7.31	2.16	14.40	1.67	92.18	5.83	1.99	sand
6	20	30.27	31.39	7.28	2.11	15.10	2.95	96.31	1.63	2.06	sand
7	20	30.20	31.35	7.25	2.06	16.30	3.53	93.49	4.14	2.37	sand
8	27	30.22	31.34	7.19	2.01	17.70	2.30	92.28	4.47	3.25	sand
9	30	30.26	31.41	7.18	1.96	18.10	2.95	83.26	12.53	4.21	Loamy sand
10	30	30.25	31.36	7.12	1.91	18.30	3.69	80.00	10.88	9.12	Loamy sand
11	37	30.24	31.34	7.03	1.86	19.10	4.35	76.77	7.61	15.62	sandy loam
12	40	30.21	31.32	6.95	1.81	19.50	4.10	73.93	8.64	17.43	sandy loam
13	40	30.24	31.41	6.92	1.78	20.10	2.30	62.5	20.45	17.05	sandy loam
14	40	30.24	31.38	6.87	1.75	21.10	10.33	58.03	24.55	17.42	sandy loam
15	50	30.27	31.33	6.81	1.72	22.30	11.48	49.82	35.73	14.45	Loam
16	70	30.21	31.40	6.70	1.69	25.70	2.79	49.64	30.55	19.81	Loam
17	80	30.23	31.39	6.66	1.66	28.30	8.61	38.77	37.97	23.26	Loam

SOC: soil organic carbon

Results and Discussion

3.1. Available contents of micronutrients (Fe, Mn, Zn and Cu) and non-nutritive elements (Co, Pb, Ni and Cd) in soil

Data presented in Fig 1 reveal that DTPA extractable micronutrients ranged between 3.38 - 14.51 mg kg⁻¹ for Fe, 4.02 - 6.26 mgkg⁻¹for Mn, 2.22 - 6.29 mgkg⁻¹for Zn and 1.66 - 3.86 mgkg⁻¹for Cu. Concentrations

of DTPA-extractable Co, Pb, Ni, and Cd within the surface layer ranged from 0.012 to 0.045 mgkg⁻¹ for Co, 0.938 to 2.926 mgkg⁻¹ for Pb, 0.064 to 0.185 mgkg⁻¹ for Ni, 0.018 to 0.098 mgkg⁻¹ for Cd (Fig 2). The highest concentrations of DTPA extractable heavy metals were recorded for the soils of long history in land cropping, while the lowest were recorded for the soils of the short cropping histories except for Co. On the short term cultivation period,

there were slight increases in the DTPA extractable HM observed from season to another. These results are in agreement with **Abdel-Aziz** (2015) who found significant increases in the micronutrient contents in soils irrigated with secondary treated wastewater. **Angin and Yaganoglu** (2011) recorded significant increases in DTPA-extractable Fe, Mn, Zn, and Cu concentrations in soils corresponding to application of sewage sludge at any of the rates of 40, 80 and 120 Mg ha⁻¹. This could be attributed to the chelation reactions of the investigated micronutrients with the organic compounds (**Herencia et al., 2008**) provided by wastewater application, which is one of the main mechanisms for enhancing solubility and availability of Fe and Mn in alkaline soils.

Data in Fig 2 representing the three seasons of DTPA-extractable HM as affected by cropping history seemed to be identical except for Co. The patterns representing each of the studied metal ions in the soils irrigated for longer periods are slightly higher than the corresponding ones irrigated for a shorter period. This means that increased application of the sewage water was associated with increased of

accumulation metal ions. Thus, applied sewage water is the main source of increased heavy metals in soils. The obtained values are lower than those recorded by Abbas (2007) in El-Gabal El-Asfar soil which were 39.05 mg Fe kg-1, 6.787 mg Mn kg⁻¹, 21.75 mg Zn kg⁻¹, 11.03 mg Cu kg⁻¹, 1.967 mg Co kg⁻¹, 6.218 mg Pb kg⁻¹, 6.48 mg Ni kg⁻¹, 0.221 mg Cd kg⁻¹. In June 2009,the Egyptian government initiated Phase II of the second stage for treating the wastewater of El-Gabal El-Asfar darin to improve its quality with a total cost of 212.64 million \$ (94.1million \$ loan) (Africa Developing Bank, 2009). This probably minimized the accumulation of heavy metals added to soils through irrigation with the wastewater while, leached out some of the soluble heavy metals from the uppermost layer of the soil. Cultivated crops in these soils might contributed also to reducing concentration of available heavy metals through absorbing these heavy metals from soil by the grown plants. Their for the DTPA extractable elements recorded relatively lower concentrations. Cobalt did not follow a certain trend with cropping period, probably because its content in the used waste water.

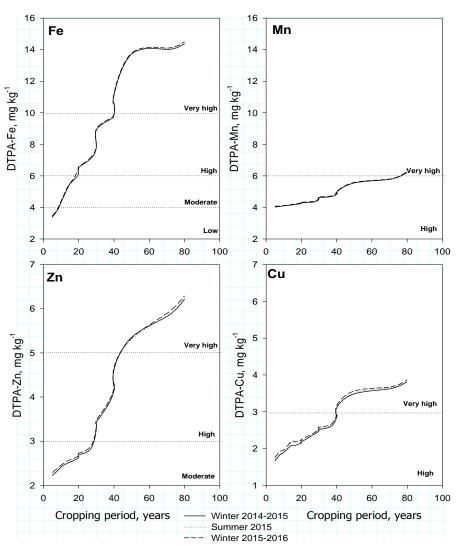


Fig 1. DTPA extractable Fe, Mn, Zn, and Cu from El-Gabal El-Asfar soils as affected by the cropping period

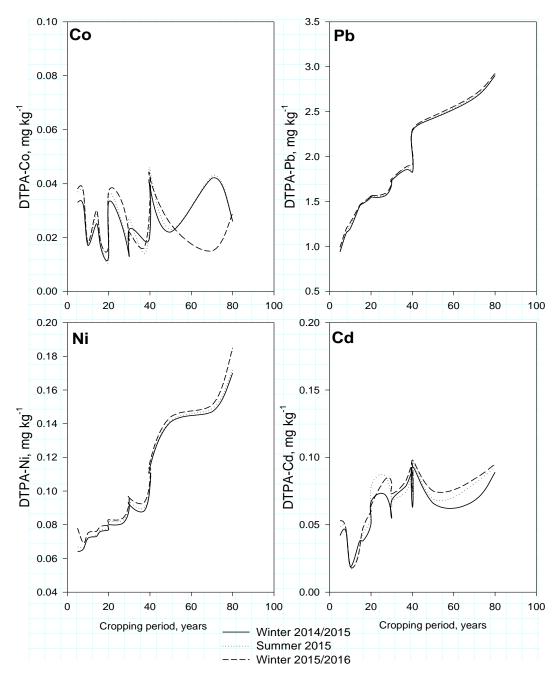


Fig 2. DTPA extractable Co, Pb, Ni and Cd from El-Gabal El-Asfar soils as affected by the cropping period

According to the critical levels of DTPA-available micronutrients which were recommended by **Lindsay and Norvell (1978)**, Fig. 2 and Table 2 reveal that 11.76 % of the tested soils were deficient in Fe, while the 17.65% of the tested soils were margin in their Fe content, 41.18% had adequate amounts and 29.41% of the tested soils recorded very high DTPA extractable Fe contents. Concerning DTPA- Mn, 94.12% of the tested soils were high in Mn content, while the other 5.88 % of the tested soils were very high in Mn content. In case of Zn, 47.06 %

of the tested soils were medium in their Zn content, while the other 52.94% soils were high in their Zn content. DTPA-available Cu levels were low in 76.47% of the tested soils, while medium, in the other 23.53% of the tested soils. These results indicate the significance of using such wastewater as a source of nutritive elements but on the other hand more attention should be paid towards using this water for irrigation for fear of exceeding their permissible limits.

SI. GradeIron (Fe) Manganese (Mn) В \boldsymbol{C} \boldsymbol{A} В \boldsymbol{A} 1 Very low 0 - 2 -----0 - 0.5---0.5 - 1.2 2 low >2 -4 3.38 - 3.9111.76 3 Medium 4 - 6 4.32 - 5.6617.65 1.2 - 3.53.5 - 6.04.03- 5.798 6 - 10 6.14 - 9.91 41.18 94.12 High >10 Very High 10.54 - 14.5129.41 > 6.0 6.21 - 6.265.88 Zinc(Zn)Copper (Cu) Very low < 0.50 < 0.10 1 --2 low 0.50 - 1.00.10 - 0.30 1.66 - 2.9776.47 Medium 1.0 - 3.0 2.22 - 2.8347.06 0.30 - 0.803 3.11 - 3.87 23.53 4 High 3.0 - 5.03.35 - 6.2952.94 0.80 - 3.05 Very High > 5.0 > 3.0 ----

Table 2. Measured concentration of available Fe, Mn, Zn, and Cu in El-Gabal El Asfar soils

A: is the grade range (mg K^{-1}) after **Lindsayand Norvell (19978), B:** is the concentration range in soil (mg K^{-1}) **C:** is the percentage of soil samples

3.1.1. Baseline Data of the non-nutritive heavy metals in the investigated soils

The DTPA extractable HM were calculated on the logarithm bases (base 10) because they show positively -skewed frequency distributions. Since the data were assumed to be approximately log-normally distributed, the central tendency and variation of data were expressed as the geometric means (GM) and geometric deviations (GD), respectively. The GM and GD were used to estimate the range of variation expected for element content of the material being studied. From the normalized data the 95% expected ranges were calculated. About 95% of the samples in the randomly selected sites would be expected to fall within the limits defined by GM/GD² and GMxGD². The range of concentrations between the calculated GM/GD² and GMxGD² are 'baseline' values (Tidball and Ebens, 1976). These ranges are a more reliable measurement of the variation of results than the observed ranges since the distorting effects of the

few high values are minimized. In order to get a more informative insight into variability of the data. percentiles of element content calculated. The expected ranges of element contents of the studied soils were markedly narrower than the observed concentrations, particularly those of Co, Pb and Cd (Table 3). However, the observed concentrations of Ni were higher than the expected ranges, reflecting a significant anthropogenic impact on its concentration in surface soils. These ranges give a reliable estimate of the element levels in El-Gabal El-Asfar soils. The differences between observed and expected ranges of element concentrations (Co, Pb and Cd) reflect the presence of a small proportion of high values resulting from environmental contamination. The concentration of Co, Pb, Ni, and Cd in the EL-Gabal El-Asfar soils were similar to or even lower than the typical values in the uncontaminated soils of the world according to Berrow and Reaves (1984).

Table 3. DTPA extractable Co, Pb, Ni and Cd contents in soil samples (0-10 cm) presented as mean, geometric mean (GM) and geometric deviation (GD).

Metal	Mean	GM	GD	Observed range	95% Expected range *
Co	0.027	0.025	1.440	0.012 - 0.045	0.012 - 0.053
Pb	1.795	1.724	1.335	0.938 - 2.926	0.967 - 3.072
Ni	0.099	0.095	1.323	0.064 - 0.185	0.054 - 0.166
Cd	0.065	0.060	1.533	0.018 - 0.098	0.026 - 0.142

*Calculated as flows: $GM/GD^2 - GM \times GD^2$.

3.2. The relationship between the DTPA-extractable HM and both of the soil chemical characteristics and cropping period

Results shown in Table 3 reveal that the concentrations of DTPA- extractable nutritive elements were significantly correlated with the period of land use in cropping. These concentrations were significantly correlated with the total content of

soil organic carbon, soil pH, and soil EC. The cropping period recorded the highest significant relation affecting DTPA extractable Mn, Zn and Cu which themselves were significantly and highly correlated with SOC. It seems that the dissolved poly functional organics of low molecular weight (Harter and Naidu, 1995) complexed with Zn (Kalbitz and Wennrich, 1998; Chatzistathis,

2014),Cu (Gerringa, 1990; Mcbride et al., 1997; Wells et al., 1998; Bolan and Duraisamy, 2003; Qin et al., 2004; Beesley et al., 2010), Cd and Pb (Weng et al., 2002; Schwab et al., 2007). Such organics many act as ligand-assisted the dissolution of Mn (Wang and Stone, 2006). The solubility of these organic fractions could be negatively affected by soil pH (Weng et al., 2002; Ashworth and Alloway, 2008). These soluble organics could be adsorbed on soil minerals (Jardine et al., 1989; Lalonde et al., 2012) or complexed to more recalcitrant organic matter (Martínez et al., 2003). On the other hand, soil pH and EC seemed to be the significant factors affecting extractable Fe. This probably indicates that the organic amendments increased Fe (III)reduction and solubility (Hall and Silver, 2013) beside of the possibility of complexation of Fe with soil organic matter to form soluble complexes (Jones et al., 2011).

Concerning DTPA extractable non-nutrients significant correlations were detected between DTPA extractable Pb and the cropping period of the land. The highest significant correlation of DTPA

extractable Pb was detected with soil organic carbon followed by soil pH then soil EC. The correlation results of DTPA extractable Ni and Cd with the studied soil parameters seemed to be similar, to some extent, with those attained for DTPA extractable Pb. The dissolved organic carbon many have increased the mobility of Ni and Cd (Antoniadis and Alloway, **2002; Molas, 2002**). Up to 80 - 99% of the dissolved Pb could exist in the form of organic Pb complexes in soils amended with organic matter at near-neutral pH (Sauvé et al., 1998; Abollino et al., 2003). These soluble organic fractions were affected significantly by soil pH (Sauvé et al., 1998). Their accumulation in soil increased with ageing probably through adsorption on the mineral soil constituents at certain ligand/metal molar ratio besides stimulating the formation of ternary complexes (McBride, 1994) or through complexation to form more recalcitrant organic matter (Martínez et al., 2003). significant correlations were detected between DTPA extractable Co and any of the studied parameters. This might be due to the applications of various mineral fertilizers that contained heavy metals including Co (Abdelhafez et al., 2012).

Table 4. Correlations between the DTPA- extractable HV and both of the soil chemical characteristics and its cropping history

	FF8		xtractable		DTPA-extractable				
		nutritive elen	nents, mg kg	1	non-nutritive elements, mg kg ⁻¹				
	Fe	Mn	Zn	Cu	Co	Pb	Ni	Cd	
Time of land use	0.964**	0.982**	0.976**	0.974**	0.279	0.974**	0.970**	0.714**	
Soil pH	-0.979**	-0.905**	-0.938**	-0.962**	-0.275	-0.978**	-0.950**	-0.713**	
Soil EC	-0.970**	-0.886**	-0.922**	-0.949**	-0.243	-0.939**	-0.880**	-0.819**	
CaCO ₃ content	-0.120	-0.052	-0.060	-0.066	0.44	-0.560	-0.037	-0.137	
Soil organic carbon	0.965**	0.931**	0.949**	0.961**	0.251	0.982**	0.948**	0.762**	

Conclusion

The results obtained in this study highlighted the positive effect of using the wastewater of El-Gabal El-Asfar drain on increasing the DTPA extractable micronutrients in soil. Such type of irrigation is thought to be potential fertigation (Vasudevan et al., 2010). This might decrease chemical inputs of fertilizers to soil and, of course, makes crop production profitable. Levels of DTPA extractable non-nutritive elements increased in soil with increasing the cropping period. Although their levels were within or lower than the typical values in the uncontaminated soils of the world, more attention should be paid towards using this water for irrigation for fear of exceeding their permissible limits. The DTPA extractable Co did not follow a certain trend with irrigation ageing. Such a conclusion agrees with that of (Ali et al., 2016) who pointed out to the unmanaged agricultural practices as negative aspects of soil pollution.

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تأثير فترات الاستزراع على تيسر العناصر الصغري والثقيلة بأراضى الجبل الأصفر التي تروي بمياه الصرف الصحى.

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تعانى مصر من ندرة المياه, ومن المتوقع أن تزداد تلك الأزمة مع مرور الوقت. ولذا دعت الضرورة إلى إعادة إستخدام مياه الصرف الصحى كمصدر إضافي للري ، ولكن تلك المياه قد تجلب العديد من الملوثات إلى التربة وينتج عن ذلك مخاطر بيئية كبيرة على المدي الطويل، خاصة مع تراكم المعادن الثقيلة في التربة والتي تمثل الصور الميسرة لها الجزء الأهم لما له من تأثير كبير علي نمو النبات، وبالتالي يهدف البحث إلى دراسة تيسر العناصر الثقيلة في منطقة الجبل الأصفر التي تروى بالمياه العادمة لفترات زمنية طويلة ،وقد تم اختيار 17 مزرعة تروى بتلك المياه لفترات زمنية تراوح ما بين أقل من 5 سنوات حتى 70 سنة. تم تحليل المحتوى الميسر من العناصر المغذية (الحديد ، المنجنيز ، الزنك ، النحاس) ، غير الغذائية بها (الكوبالت ، الرصاص ، النيكل والكادميوم) على مدار ثلاثة مواسم متتالية. حيث أظهرت النتائج أن التركيزات الأعلى من جميع العناصر الثقيلة القابلة موضع الدراسة والتي تم استخلاصها بواسطة الـ DTPA كانت في التي رويت بالمياه العادمه لفترات أطول باستثناء الكوبالت التي لم يظهر تغيرات محدده صعودا أو هبوطا مع الزمن ، كما تماثلت منحنيات الفصول الثلاثة الممثلة للمستخلص من العناصر الثقيلة بواسطة الـ DTPA (باستثناء الكوبالت)، مع الفترة الزمنية لزراعه الأرض ربما تشير تلك النتيجة إلى أن مياه الصرف هي المصدر الرئيسي لإثراء التربة بالمعادن الثقيلة موضع الدراسة ، كما تم حساب المستخلص بال DTPA من المعادن الثقيلة على أساس قواعد اللوغاريتم ويفترض أن تكون منحنى التوزيم الطبيعي. وقد تم تقدير الاتجاه المركزي وتغير البيانات، المعبر عنها بالمتوسط الهندسي (GM) والانحراف الهندسي (GD). وكان نطاق التركيز بين القيم المحسوبة GM/GD² و GM/GD² (قاعدة أساس البيانات) أكبر بكثير من التركيزات المقدرة لجميع العناصر الثقيلة باستثناء النيكل، وتعكس هذه النتائج أهمية تأثير النشاط البشري على تركيز النيكل في التربة السطحية. وبصفة عامة، كانت العناصر الثقيلة القابلة للاستخلاص ب DTPA - مرتبطة ارتباطا وثيقاً بفترة الاستزراع ، بجانب الكربون العضوي للتربة، ودرجة الحموضة للتربة وملوحة التربة ولكن ليس مع محتوى CaCO3 في التربة مما يعني أن هذه المتغيرات تسهم إلى حد ما في قابلية استخلاص العناصر . من ناحية أخري، لم يكن هناك ارتباط معنوي بين استخلاص الكوبالت بال DTPA مع أي من صفات التربة المدروسة.