Assessment of Heavy Metals in Urban Areas of Al Hashmiyya City of Jordan

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Abstract

The present study aims to assess the behavior of heavy metals (Pb, Zn, Cd, Cr, Cu and Ni) in the topsoil of Al Hashmiyya city in Jordan, their contamination level and the possible contamination sources. Heavy metals analyses were assessed using Krigging map, Enrichment Factor (EF), and geo-accumulation index (Igeo). The findings indicate that all heavy metals, except Cu, were present in higher concentrations above the safe limit. Multivariate analyses indicate that for all the tested heavy metals, soil pH was the most significant factor affecting heavy metal loads except for Cu, which was significantly related to iron oxides only. Moreover, the results indicate the presence of three major clusters: Pb, Zn, and Cr; Zn, Pb and Cr; and Cu, which does not behave differently from the other heavy metals. Most of the sites in the present study area are contaminated by heavy metals above threshold levels, reaching 100% probability. The present study shows the urgent need to monitor and control industrial emissions and remediate the heavily contaminated urban soils found in the study area.

1. Introduction

Heavy metals in soils have severe impacts on the environment and the local communities. Heavy metals accumulate in the human body via absorption, inhalation, and ingestion (Lim et al., 2008), and children and the elderly are the most strongly affected (Olawoyin et al., 2012). Several studies showed that anthropogenic activities lead to accumulation of heavy metals in topsoil (Tume et al., 2011; Li et al., 2013). Both vehicle emissions and industrial discharges have been identified as sources of heavy metals (Guo et al., 2012), and urban soils are known to behave as a sink for heavy metals from these sources (Tiller, 1992). Studies concerning heavy metal contamination in urban soils are needed to develop strategies to protect urban environments and human health against long-term accumulation of heavy metals. The present study represents a first attempt to identify the behavior, distribution, contamination and sources of heavy metals in urban topsoil in Jordan's highly-industrialized Al Hashmiyya City in Zarqa Governorate.

2. Materials and Methods

Soil samples (0-20 cm) were collected randomly to cover the areas around the highways in Al Hashmiyya city in Zarqa Governorate (described previously in Mashal et al., 2015) (Figure 1).
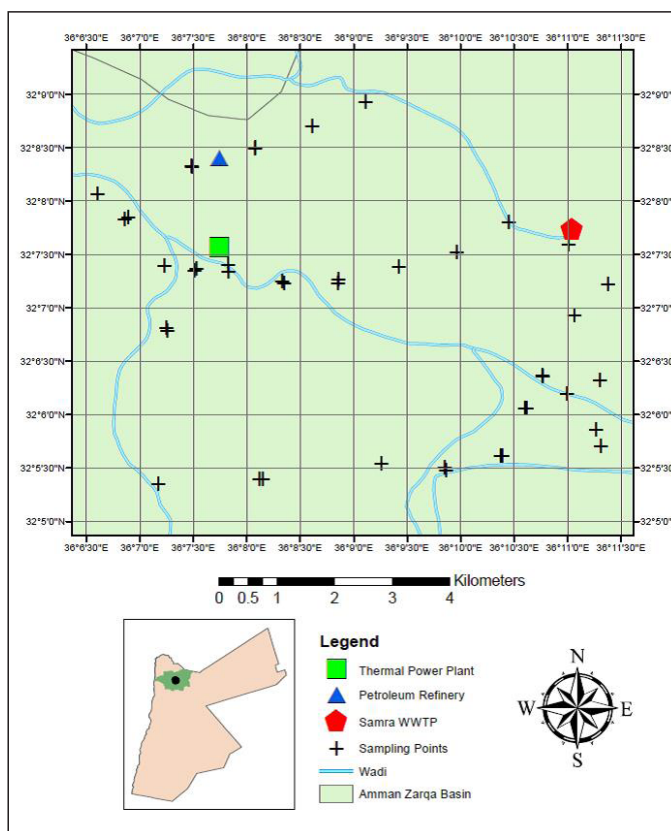

Figure 1. Sampling points and study area

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Four sub-samples were collected at each site to obtain a representative composite sample. A sampling density of two samples per km² was adopted wherever possible with a total of 43 samples. Al Hashmiyya area is susceptible to pollution from several industrial sources, including a Petroleum Refinery (PR), a Thermal Power Plant (TPP), and a Waste Water Treatment Plant (WWTP). The study area consists of highly dissected rocks with very gently undulating limestone parent material of the WadiesSir Limestone Formation, which represents the upper most of the Ajlun Group Formation (Ministry of Agriculture, 1994). Soil samples were obtained by mixing four subsamples from each site. The soil samples were collected using a stainless steel spade and were stored in self-sealing plastic bags. The spade was washed several times with distilled water and finally with deionized water and wiped dry with paper towels between each use. Geographical coordinates of sampling locations were recorded at each sampling point with a handheld GPS with 3m accuracy. Soil samples were air-dried, ground and sieved through a-2 mm sieve. The chemical properties, including pH_{se}, EC_{se} , Organic Matter (OM), percentage of total carbonates ($CaCO_3$), the active iron oxides (amorphous), and Cation Exchange Capacity (CEC), of all soil samples were measured according to appropriate analytical procedures reported in *Methods of Soil Analysis, part 3* (Sparks *et al.*, 2001).The total concentration of metals in the soil was measured using aqua regia extracts. Total concentrations of Pb, Cd, Cu, Ni, Cr and Zn in the digestion solution were determined by PerkinElmer Analyst 300, with HGA 850 graphite furnace. The element standard solutions used for calibration were prepared by diluting stock solutions of 1000 mg/L of each element supplied by Sigma. Quality Assurance and Quality Control (QA/QC) procedures were assured through analyzing all samples in duplicate and the results were accepted when the relative standard deviation falls within 5%. Blank samples were also performed throughout all the experiments for correction of background and other sources of error.

Several ordinary (descriptive) and multivariate statistical analyses were performed for both soil chemical properties (pH_{se}, EC_{se}, OM, $CaCO_3$, CEC, and amorphous Fe oxide), and heavy metals contents (Cu, Zn, Pb, Cr, Cd, and Ni), using JMP statistical program (JMP 8, 2009). Descriptive statistical analyses included measures of central tendency, dispersion, and distribution, while multivariate analyses included correlation analysis, multivariate regression, stepwise regression, Principal Component Analysis (PCA), and cluster analyses. Stepwise Regressions (SWR) were selected according to Mallow's Cp (Cp) criterion (Mallow, 1973) and Akaike's Information (AIC) criterion (Akaike, 1974). Principal component analysis was achieved based on the correlation and the covariance matrix with a standardized original variable (Hawkins, 1974). Hierarchical cluster analysis (agglomerative clustering) was performed to investigate the presence of group factor effect on soil chemical properties and heavy metals contents (Milligan, 1980). In addition, the spatial distributions of soil properties and metal contents were investigated using the ArcMap program (ArcGIS 9.2, ESRI, 2006). The selection of the best empirical semivariance model was based on both cross-validation

test and provided smallest nugget value (Goovaerts, 1997). The third stage involved the interpolation of the variable at unknown locations using kriging techniques with a simple linear weighted-interpolation scheme.

3. Results and Discussion

The descriptive statistical analyses of the chemical properties of urban topsoil samples and heavy metals content are summarized in Table (1). The soil described as highly calcareous soils with 62.4% maximum content of $CaCO_3$, most likely derived from underlying limestone. The CEC ranged from 0.79 to 11.8 cmolkg⁻¹, with a mean value of 5.3 cmolkg⁻¹. These low values are consistent with the low clay content and high calcite and quartz content in these soils. The mean OM was 2.4% and ranged from 0.53% to 16.3%.

Table 1. Statistical summary of chemical properties of soil samples and heavy metals content of the study area

	Unit	Maximum	Minimum	Mean	StdDev	CV
pH		8.5	6.7	7.6	0.40	5.3
EC	dSm⁻¹	63.8	1.3	15.2	15.1	98.7
$CaCO_3$	%	62.4	26.6	46.6	11.1	23.7
OM	%	16.3	0.5	2.4	2.6	107.9
Fe-oxide	mgkg⁻¹	2515.3	410.3	1558.3	754.9	48.4
CEC	cmolkg⁻¹	11.8	0.8	5.3	2.9	54.2
Pb	mgkg⁻¹	469.0	22.4	108.8	98.5	90.6
Zn	mgkg⁻¹	854.8	49.3	172.3	169.2	98.2
Cd	mgkg⁻¹	17.9	2.0	5.9	3.2	53.5
Cr	mgkg⁻¹	96.6	13.1	41.3	18.8	45.7
Cu	mgkg⁻¹	267.6	2.96	41.1	52.8	128.4
Ni	mgkg⁻¹	68.2	14.1	39.4	11.5	29.31

The heavy metal concentration in the urban topsoil samples were found in the following order: Zn > Pb > Cr >Cu >Ni >Cd (Table 1). However, Pb, Zn, Cd, and Cu concentrations varied greatly in the studied soils as indicated by the coefficient of variation, which exceeded 50%. For comparison of observed concentrations with geological baseline concentrations, the ranges of the baseline data estimated with the median ± 2MAD were used (Reimann *et al.*, 2005). The observed concentration ranges of all trace metals (Table 2) were greater than their upper limits, suggesting the contamination of these soils with the studied heavy metals. The mean values of Cu, Cr and Ni in the analyzed soils are much lower than the critical mean concentrations (Table 2). Moreover, the contamination levels of soils with heavy metals and their sources in urban soils were assessed using two different pollution indices. The first is the geo-accumulation index (Igeo), an estimate of the enrichment of metal concentrations above geological baseline concentrations, according to the following equation (Muller, 1981):

$$I_{geo} = \log_2\left(\frac{[M]}{1.5[M_b]}\right)$$

where *M* is the measured concentration of the heavy metal in the study area, and M_b is the geochemical background value in a reference shale (Turekian and Wedepohl, 1961). Ranges of Igeo values are used to delimit levels of contamination. Igeo values with associated contamination levels for the study area appear in Table (2). Zinc, Cu and Cr appear to be of lowest concern, while Pb and Cd show the highest Igeo values.

The second pollution index is the Enrichment Factor (EF), using the following equation (Banat *et al*., 2005):

$$EF = \frac{[M]}{M_b}$$

EF values between 0.5 and 1.5 indicate the metal is entirely from crustal materials, whereas EF values greater than 1.5 suggest enrichment from anthropogenic sources (Zhang and Liu, 2002). Moreover, the degree of contamination is also classified based on ranges of EF values (Lacatuso, 1998).

The results in Table (3) show that the EF values ranged from 0.40 to 19.6. The EF values calculated for Cr, Cu and Ni suggest very limited input from man-made sources, while the elevated Cd and Pb values indicating anthropogenic sources of pollution for these two metals. The big difference in EF values between these two anthropogenicaly-sourced metals may be due to the difference in the magnitude of input for each metal in the urban topsoil and/or the difference in the removal rate of each metal from the soil.

Table 2. Mean heavy metals concentrations and pollution indices of the sampled area

	Pb	Zn	Cd	Cr	Cu	Ni
Mean (mgKg^{-1})	108.8	172.3	5.9	41.3	41.1	39.4
Baseline data* mgkg^{-1}	12.1–27.3	29.2–115	0.18–0.46	14.8–35.2	7.1–33.5	11.6–35.6
Average shale (mgkg^{-1})	20	95	0.3	90	45	68
I$_{geo}$ values	1.85	0.26	3.70	<0	<0	<0
Igeo classification	moderately contaminated	uncontaminated to moderately contaminated	heavily contaminated	Uncontaminated	uncontaminated	uncontaminated
EF	5.4	1.8	19.6	0.46	0.91	0.58
Degree of contamination	Sever	slight	excessive	Uncontaminated	uncontaminated	uncontaminated

* Reimann et al. (2005)

Heavy metals correlation varied from weak to strong at 95% confidence (P<0.05) (Table 3). The Pb has a moderate positive correlation with Zn, and Cr. Strong correlations were found between Ni and Cr, suggesting that these heavy metals may originate from a common source. Cr and Ni have similar ionic radii, and previous research suggests that Cr and Ni are associated mostly with the mineral phase in soils (e.g., Zhang *et al*., 1999). Moderate correlation exists between Zn and Fe, Pb, Cd, and Cu. The significant correlations between these elements support the idea that anthropogenic activities, such as traffic movement, are the main source of heavy metals in soils. Cu showed only weak positive correlations with the other heavy metals, suggesting a different source than other studied metals. In assessing

the correlation between heavy metals and soil chemical properties, the results show that soil pH has a fairly moderate positive correlation with Pb, Zn, Cr and Ni (Table 3). Electrical Conductivity (EC) has no correlation with heavy metals. Soil OM showed a moderate positive correlation with Zn and Cu. Soil CaCO$_3$ showed only moderate positive correlation with Cd, and negative correlation with Cr, suggesting that Cd may be incorporated into calcite crystal structure by the formation of solid solutions (Shetye *et al*., 2009). The results show that CEC has a moderate positive correlation with Cr ad Ni while it has a moderate negative correlation with Pb, Zn and Cd. Zinc, Cd, and Cu elements were correlated positively with amorphous Fe oxide in the topsoil samples.

Table 3. Correlation between trace metal contents and soil properties in the urban topsoil samples (all heavy metals and amorphous Fe-oxide in mgkg^{-1}, EC in dSm^{-1}, and CEC in cmolkg^{-1})

	Pb	Zn	Cd	Cr	Cu	Ni	pH	EC	%OM	%CaCO$_3$	Fe-oxide	CEC
Pb	1.00											
Zn	0.51[b]	1.00										
Cd	0.14	0.35[a]	1.00									
Cr	0.37[a]	0.17	-0.06	1.00								
Cu	0.25	0.31[a]	0.12	-0.04	1.00							
Ni	0.28	0.08	0.02	0.73	0.09	1.00						
pH	0.23	0.28	-0.08	0.37	0.13	0.39[b]	1.00					
EC	0.12	-0.08	-0.16	-0.10	0.09	0.01	-0.46[b]	1.00				
% OM	0.21	0.58[b]	0.15	-0.08	0.34[a]	-0.03	0.06	0.21	1.00			
% CaCO$_3$	0.01	0.13	0.36[a]	-0.60[b]	0.26	-0.22	-0.22	0.20	0.18	1.00		
Fe-oxide	0.09	0.43[b]	0.56[b]	-0.38[a]	0.39[a]	-0.18	-0.26	0.07	0.37[a]	0.65[b]	1.00	
CEC	-0.34[a]	-0.33[a]	-0.33[a]	0.52[b]	-0.25	0.32[a]	0.25	-0.24	-0.28	-0.68[b]	-0.62[b]	1.00

[a] At the 0.05 level of significant correlation; [b] At the 0.01 level of significant correlation; p values in brackets

Multivariate analyses indicated that soil chemical properties significantly influence heavy metal loads. For all the tested heavy metals, soil pH was the most significant factor affecting their loads except Cu, which was significantly related to iron oxide only (Table 4). Soil EC had a negligible effect on heavy metal loads since it was omitted from all stepwise models, suggesting that soluble salts have no effect. On the other hand, soil CEC had a significant effect on Pb loads in urban soils, in which each one-unit increase in soil CEC reduced the pollution (load) of Pb in the soil by 14.4 mg kg^{-1}. Similarly, Fe oxide had a small but positive significant effect on the Zn, Cd, and Cu, suggesting that these elements are associated with the Fe (hydr)oxides in these soils. The Zn was highly related to OM content by which each one-unit increase in OM was subjected for increase in Zn load by 32.9 mg kg^{-1}, indicating that OM has a large sorption capacity towards these metals (Quenea *et al.*, 2009). This effect is probably due to the cation exchange capacity of organic material (Martin and Kaplan, 1998).

Table 4. Statistical summary of full and stepwise multi-linear modeling

Parameter	Full Regression Model	Stepwise Regression Model				
	Math Model	R^2	RMSE	Math Model	R^{2*}	RMSE*
Pb	-428.691 + 103.381 pH + 1.566 EC -3.574 CaCO$_3$ + 0.639 OM -21.181 CEC + 0.007 Fe	0.337	86.64	-435.946 + 82.350 pH- 14.419CEC	0.219	89.22
Zn	-817.703 +142.756 pH-0.463 EC -4.438 CaCO$_3$ + 31.190 OM -15.026 CEC + 0.086 Fe	0.568	120.13	-1126.785 + 146. 085 pH + 32.944 OM + 0.077 Fe	0.519	121.77
Cd	3.910+-0.163 pH -0.045 EC + 0.011 CaCO$_3$ -0.022 OM -0.017 CEC + 0.002 Fe	0.357	2.73	2.235 + 0.002 Fe	0.316	2.64
Cr	-66.845 + 17.086 pH + 0.286 EC- 0.881 CaCO$_3$ -0.440 OM + 1.641 CEC + 0.005 Fe	0.476	14.73	-7.008 + 12.068pH- 0.919CaCO$_3$	0.416	14.75
Cu	-332.889+ 40.543 pH + 0.6145 EC + 0.121 CaCO$_3$+ 3.210 OM + 0.429 CEC + 0.028 Fe	0.259	49.05	-1.054 + 0.027Fe	0.150	49.24
Ni	-87.959+ 15.397 pH + 0.276 EC -0.096 CaCO$_3$ -0.580 OM + 1.309 CEC + 0.004 Fe	0.298	10.45	-47.087 + 11.461pH	0.156	10.74

*where R^2 is the coefficient of determination, and RMSE is the root mean square error of the prediction model.

According to PCA, Table (5) summarizes the variation of the specified variables with principal components and how the principal components absorb the variation in the data. Based on correlation, the first nine principal components account for 96.146% of the variation in the sample. This indicates the degree of diversity in the data and the relation between parameters included. While based on covariance, the first principal component accounts for 94.146% of the variation in the sample. This indicates a directional influence of the variables by which some are positively related or negatively related. The first PC that represents the linear combination of the standardized original variables that has the greatest possible variance can be written as follows:

$$PC_1 = -0.42404 \text{ pH} + 0.00861 \text{ Ec} + 0.03748 \text{ CaCO}_3 + 0.11371 \text{ OM} - 0.15276 \text{ CEC} + 0.00059 \text{ Fe} + 0.00103 \text{ Pb} + 0.00140 \text{ Zn} + 0.08448 \text{ Cd} - 0.01639 \text{ Cr} + 0.00421 \text{ Cu} - 0.01718 \text{ Ni} + 1.28088$$

On the other hand, each subsequent PC is the linear combination of the standardized original variable that has the greatest possible variance and is uncorrelated with all previously defined components. The second PC can be written as:

$$PC_2 = 0.94936 \text{ pH} - 0.00671 \text{ Ec} - 0.00973 \text{ CaCO}_3 + 0.10844 \text{ OM} + 0.00491 \text{ CEC} + 0.00007 \text{ Fe} + 0.00428 \text{ Pb} + 0.00259 \text{ Zn} + 0.04698 \text{ Cd} + 0.02187 \text{ Cr} + 0.00455 \text{ Cu} + 0.03351 \text{ Ni} -10.60050$$

The slope of the parameters included in the PC indicates the importance of the parameter in explaining the variability within the data, and this coincides with regression models in which soil pH is the most dominant variable in all stepwise models affecting the variability of heavy metal loads.

Table 5. Statistical summary of principal component analyses based on correlation and covariance

Number	PC on Correlation		PC on Covariances			
	Eigenvalue	Percent	Cum Percent	Eigenvalue	Percent	Cum Percent
1	3.6956	30.797	30.797	576025.2	94.146	94.146
2	2.6413	22.011	52.808	26859.89	4.390	98.536
3	1.3587	11.322	64.130	6117.315	1.000	99.536
4	1.0282	8.568	72.698	2199.585	0.360	99.896
5	0.8591	7.159	79.858	323.9635	0.053	99.949
6	0.8180	6.817	86.674	208.3557	0.034	99.983
7	0.5673	4.727	91.402	69.0239	0.011	99.994
8	0.3236	2.697	94.098	26.1275	0.004	99.998
9	0.2457	2.048	96.146	5.7964	0.001	99.999
10	0.1949	1.625	97.770	2.7073	0.000	100.000
11	0.1631	1.359	99.129	2.1678	0.000	100.000
12	0.1045	0.871	100.000	0.0679	0.000	100.000

According to cluster analysis, the generated dendrogram and the scree plot indicates the presence of three major clusters at this study (Table 6). The three clusters actually represent three heavy metal loads (low, moderate, and high). The high metal loads of Pb, Zn, and Cr are grouped together and associated with soil pH above 8, iron oxides above 2000 and low soil EC. While Zn, Pb and Cr are grouped together and are associated with high OM above 5% with soil pH ranging between 7.5 and 8.3 and iron oxides above 2000. On the other hand, Cu is not grouped with any other heavy metal and thus acts differently. The high Cu loads are associated with soils with high $CaCO_3$ and low OM content. The mean of the generated clusters are presented in Table (6).

Table 6. Cluster Means as generated by Hierarchical cluster analysis

Cluster	pH	Ec	CaCO$_3$	OM	CEC	Fe	Pb	Zn	Cd	Cr	Cu	Ni
1	7.3	18.2	55.6	2.2	3.6	1935.8	76.6	143.7	6.2	25.3	26.8	30.2
2	7.6	13.8	34.9	1.3	8.2	731.9	94.1	84.2	3.5	53.8	18.7	44.0
3	7.8	12.6	48.5	3.8	4.1	2101.9	178.4	336.5	8.7	48.8	93.7	47.3

The spatial descriptions of soil variables are presented in Table (7). Generally, almost all soil chemical properties were characterized by anisotropic behavior indicating the oriented spatial dependence at variant associated angles as presented in the Table (8). Similarly, most soil heavy metals were having identical spatial trend of exponential semivariogrm with major independence ranges varied from 0.0561 to 0.0786 km and minor independence ranges varied from 0.0148 to 0.0298 km except for Ni that had isotropic behavior with independence spatial range of 0.0365 km. The similarity between soil chemical properties and heavy metals distributions suggests that the spatial distribution is controlled by pollution source and the transfer mechanism. Many contamination hotspots were identified in the krig maps (Figure 1a-e), some with potential contaminations of heavy metals up to 100 percent above threshold levels. Cadmium contamination is the most pronounced in the study area, followed by Pb. Lead is well known to be one of the less mobile elements in calcareous soils due to the precipitation of Pb carbonates as well as Pb adsorption to Fe oxides (Freyssinet *et al.*, 2002). This could explain the high values still found in urban soils, even after the recent adoption of unleaded fuel in this area. Zinc, Cr, and Cu are the elements presenting the lowest degree of contamination.

Table 7. Descriptive analysis of soil variables spatial distributions

	Transformation	Isotropy	Model	Range (km)		Partial Sill	Nugget	
				Major	Minor			
pH	Normal	Aniso	Penta-spherical	0.0002	0.0001	297.0	0.248	0
EC	Lognormal	Aniso	Exponential	0.0104	0.0016	81.0	1.020	0.176
CaCO$_3$	Cox-Box (1.83)	Aniso	Circular	0.0786	0.0223	306.0	22958	50043
OM	Lognormal	Isotropy	Exponential	0.0786		0.168	0.356	
CEC	Cox-Box (0.24)	Aniso	Exponential	0.0786	0.0439	297.0	0.303	0.417
*Fe (mg/kg)	Normal	Isotropy	Spherical	0.0753		263858	570790	
Pb (mg/kg)	Lognormal	Aniso	Exponential	0.0786	0.0298	36.0	0.502	0.276
Zn (mg/kg)	Lognormal	Aniso	Exponential	0.0786	0.0121	288.0	0.274	0.184
**Cd (mg/kg)	Lognormal	Aniso	Exponential	0.0652	0.0214	36.0	0.118	0.109
Cr (mg/kg)	Lognormal	Aniso	Exponential	0.0561	0.0148	297.0	0.122	0.108
Cu (mg/kg)	Lognormal	Aniso	Exponential	0.0786	0.0123	18.0	0.470	0.235
Ni (mg/kg)	Normal	Isotropy	Exponential	0.0365		99.371	47.944	

Pb, Zn and Cu seem to follow the same pattern; where the highest concentrations are close to the highways and the lowest concentrations are in residential areas (Figures 2a, b and d). The Pb enrichment near highways most likely results from the burning of leaded fuel, Cu is likely derived from brakes, and Zn from worn out tires (Van Bohemen and Janssen Van, 2003). High Cd concentrations are found throughout the study area (Figure not shown). However, the lowest values were close to the residential areas, and the highest were near the Samra WWTP. The distribution of Cr and Ni were much more heterogeneous than the other metals (Figures 2c and e), suggesting that the concentration of Cr and Ni may not come from point pollution, such as industrial activities, and that natural factors, such as the soil parent materials, were also an important source of these two metals.
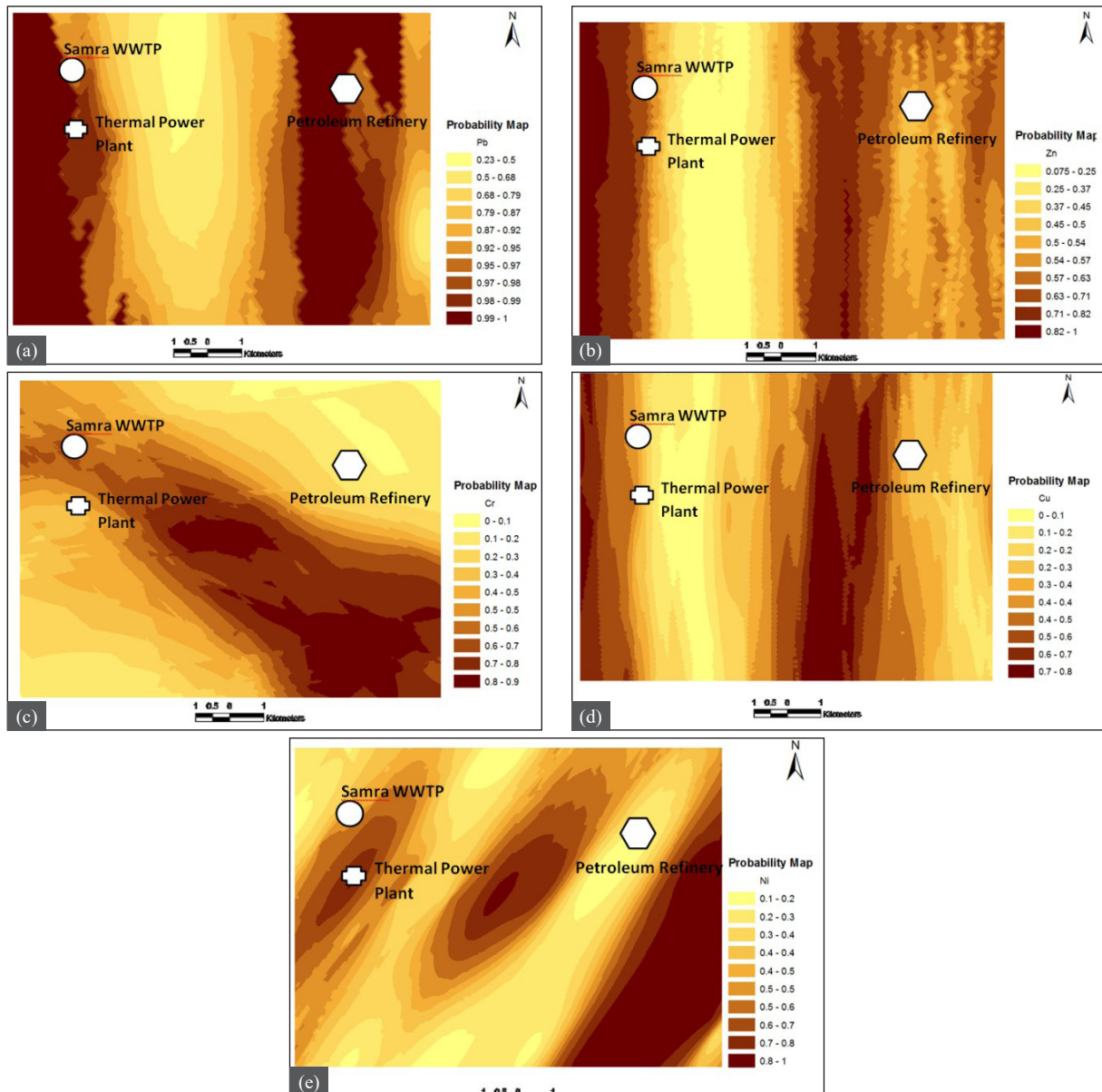
Figure 2. Probability maps of (a)Pb, (b) Zn, (c) Cr, (d) Cu, (e) Ni

Conclusion

Most of the sites in the present study area are contaminated by heavy metals reaching 100% probability above safe threshold levels. However, the Enrichment Factors (EFs) of the heavy metals showed that Cr, Cu and Ni are depleted by 0.46, 0.91, and 0.58, respectively, whereas Pb, Zn, and Cd are enriched by 5.4, 1.8 and 19.6, respectively. The calculation of Igeo index showed that the urban topsoil of Al-Hashemiyya is uncontaminated with Cr, Cu, and Ni, slightly contaminated with Zn, severely contaminated with Pb, and excessively contaminated with Cd. The contamination levels of Pb and Cd are higher than that for the other metals. Multivariate analyses indicate that soil pH is the most significant factor affecting the loads for the tested heavy metals except for Cu. Moreover, the results indicate the presence of three major clusters: Pb, Zn, and Cr; Zn, Pb and Cr; while Cu is not grouped with any other heavy metal and, thus, acts differently. The present study recommends adoption of monitoring, reporting and validation for the possible contamination so as to set appropriate strategies for controlling contaminants status and transports. Some of these strategies may include reduction in industrial emissions through improving their efficiencies, conducting periodical environmental impact assessments, creating zones and vulnerability maps for contaminated sites, and other related reclamation and rehabilitation programs.

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