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Heavy metals assessment and identification of their sources in agricultural soils of Southern Tehran, Iran

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Abstract A detailed investigation was conducted to evaluate heavy metal sources and their spatial distribution in agricultural fields in the south of Tehran using statistics, geostatistics, and a geographic information system. The content of Cd, Cu, Co, Pb, Zn, Cr, and Ni were determined in 106 samples. The results showed that the primary inputs of Cr, Co, and Ni were due to pedogenic factors, while the inputs of Zn, Pb, and Cu were due to anthropogenic sources. Cd was associated with distinct sources, such as agricultural and industrial pollution. Ordinary kriging was carried out to map the spatial patters of heavy metals, and disjunctive kriging was used to quantify the probability of heavy metal concentrations higher than their recommended threshold values. The results show that Cd, Cu, Ni, and Zn exhibit pollution risk in the study area. The sources of the high pollution levels evaluated were related to the use of urban and industrial wastewater and agricultural practices. These results are useful for the development of proper management strategies for remediation practices in the polluted area.

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Introduction

Soil contamination by heavy metals due to nondecay over time and long biological half-lives has been intensely studied. The main sources of heavy metals in agricultural soils are due to activities such as irrigation using wastewater, agricultural fertilizers, pesticides, organic manure, disposal of urban and industrial wastes, mining, smelting processes, and atmospheric pollution from motor vehicles and the combustion of fossil fuels (Adriano 2001; Alloway 1990; Huang et al. 2007; Nicholson et al. 2003; Zhang 2006). Due to long-term irrigation with domestic sewage and industrial effluents containing heavy metals, contamination in agricultural soils has become significantly higher than background levels (Rodriguez et al. 2008). This wastewater contains organic compounds, macronutrients, micronutrients and nonessential trace elements, organic micro-pollutants, microorganisms, and the eggs of parasitic organisms (Alloway and Jackson 1991).

Because of the low precipitation and high evaporation rates in semi-arid climates, the use of urban and industrial wastewater in agricultural fields in these areas is unavoidable. Thus, heavy metals may accumulate in the food chain, leading to potential threats to human health. They may also leach out of soils to groundwater and decrease crop yield quality. Deterioration of environmental conditions and increasing reliance on agrochemicals have led to a growing public concern over the potential accumulation of heavy metals and other contaminants in agricultural soils (Nicholson et al. 2003). Some metals, such as Zn and Cu, are essential elements for plant growth. However, at high concentrations, these metals become toxic. Others, which are not essential for plant growth (e.g., Pb or Cd) may be tolerated by the ecosystem at low concentrations but are harmful at higher concentrations (Alloway and Ayres 1993).

A high concentration of heavy metals in agricultural soils affects the crop output and quality. It also deteriorates the growth, morphology, and metabolism of microorganisms in soils (Giller et al. 1998). These effects can be considered a real threat to human food safety (Okoronkwo et al. 2005). In recent years, rapid industrialization and urbanization have caused heavy metal contamination to become a serious concern in many countries (Chen et al. 2008; Liu et al. 2006; Rodrigues et al. 2006; Shi et al. 2007). One of the major direct environmental impacts of development is the degradation of water resources and water quality (USEPA 2001). Using wastewater for agricultural purposes occurs either indirectly, when partially untreated effluent is discharged into rivers that supply water for agricultural fields, or directly, in urban farms when partially treated sewage effluent is conveyed into some gardens. Iran and other countries located in the world's arid belt face severe water scarcity. To partially meet the demand for water by their large urban populations, the governments in these countries are compelled to reuse a significant volume of urban and industrial wastewater that is contaminated by heavy metals from agriculture (Yargholi et al. 2008). The metropolis of Tehran produces approximately 6 m^3/s of wastewater, which is subsequently used for the irrigation of crops and vegetables in more than 1,000 km^2 of agricultural fields located in the plains of southern Tehran. Sewage wastes from different areas of Tehran have been transferred to southern areas of the city along two rivers for 70 years. Because of this practice, several vegetable species, which represent the major portion of the daily diet of Tehran's population, have been produced by using domestic and industrial wastewater for many years. Thus, due to the rapid development of urbanization and industrialization, soil pollution by heavy metals in Southern Tehran has become an urgent problem. Long-term use of wastewater has resulted in the accumulation of heavy metals in the soil and their transfer to various crops under cultivation, with levels of contamination that exceed permissible limits. There is no legislation regarding metal concentrations in agricultural and urban soils in Iran. A few studies have been done on the metal levels in agricultural soils that have received wastewater (Salmasi and Tavassoli 2006; Yargholi 2007). Heavy metal pollution in soil is commonly estimated by interpolating concentrations of heavy metals sampled at point locations so that each heavy metal is represented in a separate map (Webster and Oliver 2001). The methods of geostatistics use the stochastic theory of spatial correlation for both interpolation and for apportioning uncertainty (Goovaerts 1997). In particular, geostatistics has been popularly applied in investigating and mapping soil pollution by heavy metals in recent years (Lin et al. 2001; Liu et al. 2006; McGrath et al. 2004; Romic and Romic 2003).

Geostatistics provides a set of statistical tools for incorporating spatial coordinates of observations in data processing (Goovaerts 1999). Recent studies have attempted to apply both multivariate analyses and geographic information system (GIS) techniques to agricultural soil studies (Facchinelli et al. 2001; Micó et al. 2006, 2007). There are few detailed studies that have been undertaken to determinate the heavy metal levels in agricultural soils in Iran. Thus, the aim of the present study was to determine: (1) metal distribution through geostatistical analysis to identify their spatial patterns in the region, (2) natural or anthropic sources of individual metals in the soils using geostatistical and multivariate statistical analyses, and (3) mapping of the environmental quality and risk assessment in agricultural soils in the south of Tehran.

Material and method

Study area

The study area is located in a vegetable planting area (35°33′39″∼35°24′15″ N, 51°35′29″∼51°25′03″ E) situated in southern Tehran with an area of 800 km (Fig. 1). Land use in the area has been intensely affected by human activities for agricultural production, such that 70% of the land has been used for agricultural farming; 4% of the land is occupied by residential settlements; and the rest is occupied by range. This area is characterized by gently sloping to very steep slopes with the gradient ranging from 0% to 20%. The altitude of the area varies from 920 to 1,112 m. This zone is characterized by mild-cold winters and a semiarid continental climate with an average annual rainfall of 232 mm (for the period 1993–2003, in Mehrabad Station) and a minimum and a maximum average annual temperature of −4°C and 42°C, respectively. The agricultural lands in south of Tehran are well known for vegetable production. The main soil types in the study area are Typic Calciorthids and Natric Camborthids with an average pH value of 7.8. Most of the agricultural land has been irrigated with wastewater and sewage from Tehran city. The large population (approximately 10 million) and dense industrial activity in Tehran have discharged a large amount of wastewater into the urban environment.

Sampling and chemical analysis

In July 2008, a total of 106 surface soil samples (0–25 cm in depth) were taken in agricultural areas based on a land use map at 1:100,000 scale (Fig. 1). Heavy metals from anthropogenic sources mainly accumulate at the surface of these areas, and most of the roots of vegetable crops are located at this depth (Ross 1994). Sampling sites were selected randomly. A composite soil sample consisted of five subsamples obtained using a stainless steel hand auger at regular distances from each other. Subsamples were mixed into one composite sample for each sampling site and analyzed in triplicate. The coordination of the sample locations

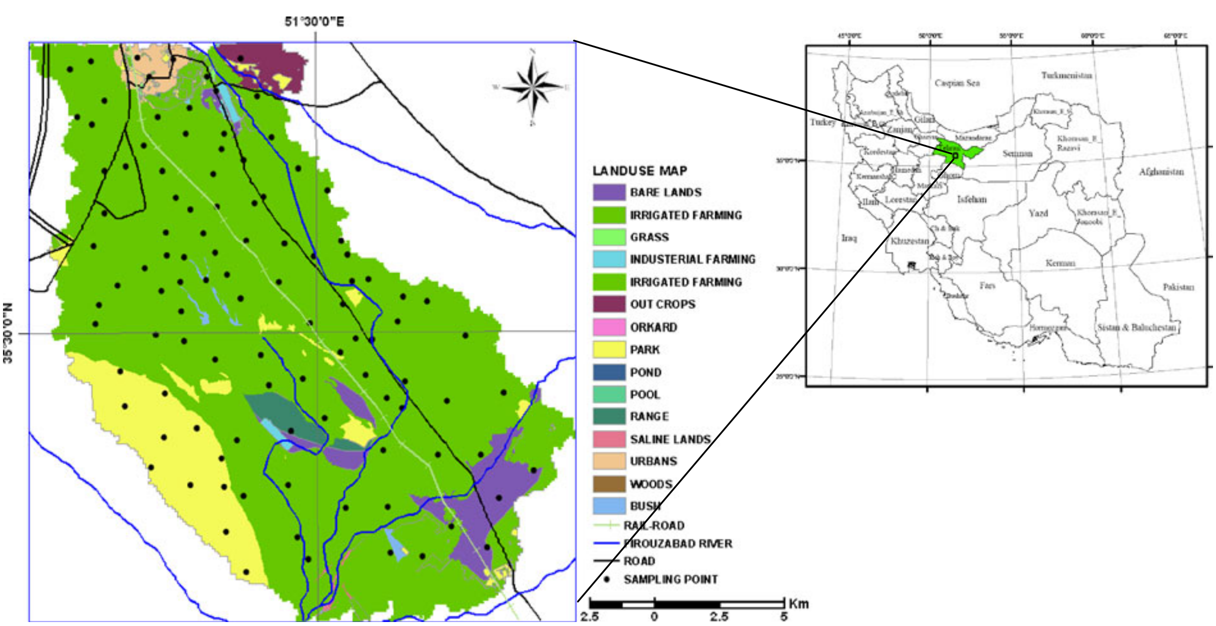


Fig. 1 Land use map and sampling sites of the study area

was conducted using a global positioning system receiver. Approximately 1 kg of each sample was stored in polyethylene packages and transported to the laboratory. The soil samples were air-dried for several days at room temperature (20–22°C) and then ground and sieved to a size of 2 mm for analysis of their properties. Soil samples were digested by aqua regia with a mixture of nitric and hydrochloric acids according to method 3050B of the United States Environmental Protection Agency (USEPA 1996). The heavy metal concentrations of Cd, Cu, Co, Pb, Zn, Cr, and Ni were determined using inductively coupled plasma atomic emission spectroscopy (ICP-ES; 138 Ultrace; Jobin Yvon).

Geostatistics

Geostatistics uses the variogram technique to measure the spatial variability of the recognized variable and provides the input parameters for the spatial interpolation of kriging (Krige 1951; Webster and Oliver 2001). Kriging has been widely used as an important interpolation method at different scales, especially in studies on soil pollution (Chen et al. 2008; Shi et al. 2007). The semi-variogram, $\gamma(h)$, measures the mean variability between two points, X and $X+h$, as a function of their distance, h, for data location at discrete sampling locations. The semivariogram is an autocorrelation statistic defined by the following equation (Webster and Oliver 2001):

$$\gamma(h) = \frac{1}{2N(h)} \sum_{i=1}^{N(h)} [Z(xi) - Z(xi+h)]^2$$

where, $Z\ (xi)$ is the value of the variable Z at point i; $Z(xi+h)$ is the value of the variable Z at point $i+h$; and $N(h)$ is the number of sample pair points separated by the lag distance, h. The experimental variogram measures the average degree of dissimilarity between unsampled values and nearby data values and can thus depict autocorrelation at various distances. The variogram model was chosen from a set of mathematical functions that describe spatial relationships and usually fitted by weighted lost squares, and range, nugget and sill are then used in the kriging procedure. In this study, to make distribution maps, several spatial interpolation techniques were evaluated for the best results including kriging, global/local polynomial interpolation, inverse distance weighting, and radial basis functions. We used kriging (ordinary kriging) as a spatial interpolation technique to make distribution maps because it is very flexible and allows users to investigate graphs of spatial autocorrelation and allows the production of prediction, prediction standard error, and probability maps and, at the same time, minimizes the error of the predicted values. The statistics of the differences between the measured and predicted values at sampled points are often used as an indicator of the performance of an inexact method (Burrough and McDonnell 1998).

For the evaluation of the degree simulation quality and the model–experiment comparison of different model approaches, cross-validation indicators and additional model parameters can be used. For comparing these models, we used cross-validation by the statistical parameters mean error (ME), root mean square error (RMSE),

Table 1 Summery statistics for heavy metals concentrations (mg kg^{-1}) in top soil

	Cd	Cu	Co	Pb	Zn	Cr	Ni
Mean	0.77	36.09	13.27	16.46	217.99	67.96	36.92
Minimum	0.16	11.25	6.04	1.53	21.36	9.66	10.06
Maximum	2.01	102.54	20.32	46.32	521.22	243.05	89.05
Std. Deviation	0.40	22.72	3.96	8.63	119.44	32.82	14.08
Skewness	1.28	1.31	0.26	0.91	0.26	1.98	0.71
Kurtosis	1.38	0.85	−1.15	0.98	−0.76	9.10	1.79
CV%	51.43	62.94	29.80	52.42	54.79	48.29	38.13
Background value[a]	0.46	48.78	16.13	75.52	125.86	61.04	35.17
Guide value[b]	0.60	100.00	40.00	350.00	300.00	350.00	60.00
Firuzabad River	76.00	54.00	n.d	71.00	56.00	n.d	1762.00
K–S p	0.00	0.00	0.010	0.28	0.050	0.14	0.17
K–S p log	0.64	0.49	0.050				

$P < 0.05$

CV coefficient of variation

[a] Alavi Naeini et al. (2005)

[b] Chinese Environmental quality standard for soil (pH > 7.5; GB15618-1995)

Table 2 Total variance explained

Component	Initial eigenvalues			Extraction sums of squared loadings			Rotation sums of squared loadings		
	Total	% of Variance	Cumulative %	Total	% of Variance	Cumulative %	Total	% of Variance	Cumulative %
1	2.237	31.953	31.953	2.237	31.953	31.953	2.232	31.887	31.887
2	1.753	25.046	56.998	1.753	25.046	56.998	1.749	24.982	56.869
3	1.084	15.487	72.486	1.084	15.487	72.486	1.093	15.617	72.486
4	0.765	10.935	83.421						
5	0.599	8.551	91.972						
6	0.361	5.152	97.124						
7	0.201	2.876	100.000						

Extraction method: principal components analysis; rotation method: Varimax with Kaiser normalization

average standard error (ASE), mean standard error (MSE), and root mean squared standardized error (Robinson and Metternicht 2006). ME is used for determining the degree of bias in the estimates, often referred to as the bias (Isaaks and Srivastava 1989), but it should be used cautiously as an indicator of accuracy because negative and positive estimates counteract each other, and the resultant ME tends to be lower than the actual error (Nalder and Wein 1998). RMSE provides a measure of the error size, but it is sensitive to outliers as it places a lot of weight on large errors (Hernandez-Stefanoni and Ponce-Hernandez 2006).

MSE suffers from the same drawbacks as RMSE, whereas MAE is less sensitive to extreme values (Willmott 1982; Vicente-Serrano et al. 2003) and indicates the extent to which the estimate may be in error (Nalder and Wein 1998). The difference between the known data and the predicted data is calculated using the mean error, and it should be a value near zero. The RMSE quantifies the error of the predicted surface. The variability of the prediction is evaluated by comparing the ASE and RMSE. If the ASE is greater than the RMSE, the variability of the prediction is overestimated. Because the ME is a function of the scale of the data, the ME is standardized to the MSE by dividing the prediction error by their prediction standard error. The MSE should be a value near zero. In ordinary kriging, the probability maps depend on the kriging standard errors. If the ASE is close to the root-mean squared prediction error, assessment of variability in the prediction error has been done correctly. If the average standard error is greater or less than the root-mean-squared prediction error, the prediction has been either over- or underestimated, respectively.

The disjunctive kriging technique can be applied for the assessment of pollution risk to evaluate the probability that the true value of soil heavy metals at unsampled points exceeds the specified thresholds. It provides an estimate of the conditional probability that a random variable located at a point, or averaged over a block in two-dimensional space, exceeds certain thresholds. It is assumed that the concentration of a radionuclide is a realization of a random variable $Z(x)$, where x denotes the spatial coordinates in two dimensions. If a threshold concentration, z_c, is

Table 3 Component matrices for elements

Element	Component matrix			Rotate component matrix		
	PC1	PC2	PC3	PC1	PC2	PC3
Pb	0.008	0.821	0.172	−0.030	0.803	0.240
Zn	0.052	0.807	−0.018	0.026	0.807	0.052
Cr	0.781	0.175	−0.181	0.784	0.212	−0.117
Ni	0.882	−0.124	0.104	0.878	−0.107	0.148
LogCd	0.094	0.224	0.873	0.035	0.152	0.893
LogCu	0.004	0.566	−0.497	0.015	0.607	−0.447
LogCo	0.915	−.109	−0.034	0.919	−0.080	0.014

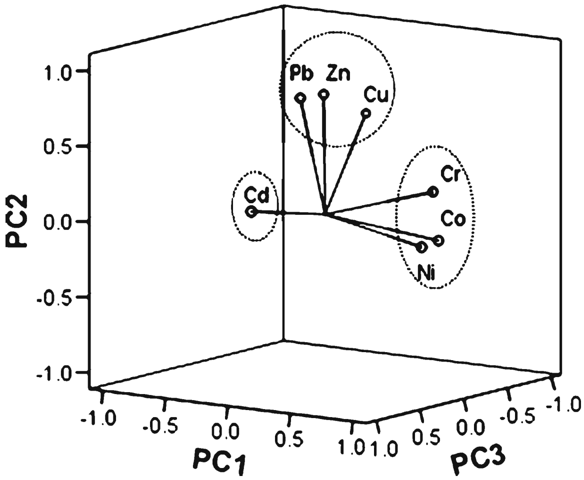


Fig. 2 Factor loading plots for three dominant factors

defined, marking the limit of what is acceptable, then the scale is dissected into two classes, which are less than and more than z_c, respectively. The soil must belong to one of these classes at any one place. The values 0 and 1, respectively, can be assigned to the two classes, thereby creating a new binary variable, or indicator, which is denoted by $Q[Z(x) \geq z_c]$ (Steiger et al. 1996; Lark and Ferguson 2004).

Multivariate statistics and data transformation

The test of normality of the dataset was performed with the Kolmogorov–Smirnov (K–S) test with the skewness and kurtosis parameter. It is necessary to normalize the data transformation for environmental variables because they have a lognormal pattern (Krige 1960). Logarithmic transformations were applied to normalize too highly skewed and outlier datasets because they can impair the variogram structure and the kriging results; however, normality may be strictly required in multivariate statistics and linear geostatistics (McGrath et al. 2004). Pearson correlation coefficients were applied to examine the relationships between soil heavy metals. Principal components analysis (PCA) was employed for identification of heavy metal sources. PCA converts the variables under investigation into factors, or principal components, and correlation among the original variables can be minimized and their elements divided into fewer groups. In addition, Varimax and Kaiser normalization rotation was applied to maximize the variance of factor loading across variances for each factor.

Multivariate statistical analyses and descriptive statistical parameters of the data were performed using the SPSS (V.13) software package (SPSS Inc., Chicago, USA) for Windows. Geostatistical analysis, semivariogram model fitting, and spatial distribution using ordinary kriging were performed with GIS software ArcGIS V.9.2 (ESRI Co, Redlands, USA).

Result and discussion

Descriptive statistics and normality test

The basic descriptive statistics for our raw data on heavy metals are summarized in Table 1. The raw datasets show positive skewness. The skewness and kurtosis for Cd, Cu, and Co are high, and the levels of these metals did not pass the K–S normality test (K–S p), whereas other variables, such as Pb, Zn, Cr, and Ni, did pass. The skewness and kurtosis values for Cd, Cu, and Co decreased after the raw datasets were logarithmically transformed. Because further statistics and geostatistics analyses require data to follow a normal distribution, the significance levels of the Kolmogorov–Smirnov normality test for the raw data and

Table 4 Semivariograms models and parameters of heavy metals

Metal	Semivariogram model	Nugget (C0)	Sill (C0 + C)	C0/(C0 + C)	Range	RMSE
Cd	Rational Quadratic	0.17023	0.27826	0.612	19000	1.098
Cu	J-Bessel	0.2612	0.35569	0.734	18635	1.002
Co	Pentaspherical	0.071947	0.1028	0.700	18632	0.999
Pb	J-Bessel	41.097	73.478	0.557	3541	0.952
Zn	Gaussian	12645	15401.3	0.821	14555	1.000
Cr	K-Bessel	266.19	1468.19	0.181	2857	1.002
Ni	Gaussian	150.66	247.748	0.608	19548	1.091

the logarithmically transformed significance are shown in Table 1. The mean concentrations of Cd, Zn, and Cr in the analyzed samples were higher than background level values, while the mean concentrations of Cu, Co, Pb, and Ni were lower than their background levels provided by the Ministry of Industries and Mines Geological Survey of Iran (Alavi Naeini et al. 2005). Large coefficients of variation (CV %) of Cd, Pb, Zn, and Cu in the soils, which reached 51.43, 52.42,

Fig. 3 Filled contours maps of soil Pb, Zn, Ni, Co, Cd, Cu, and Cr in agricultural soils

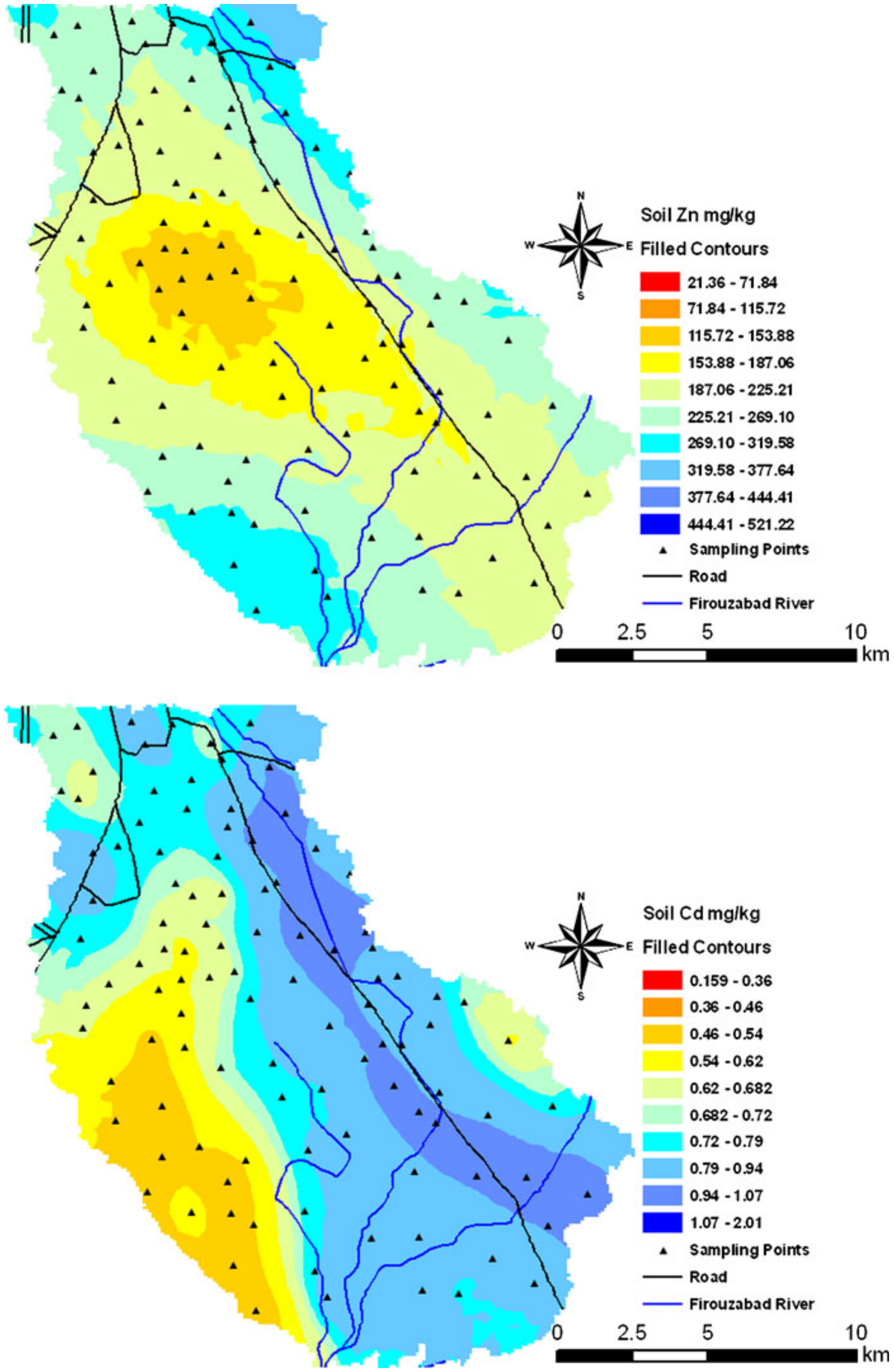
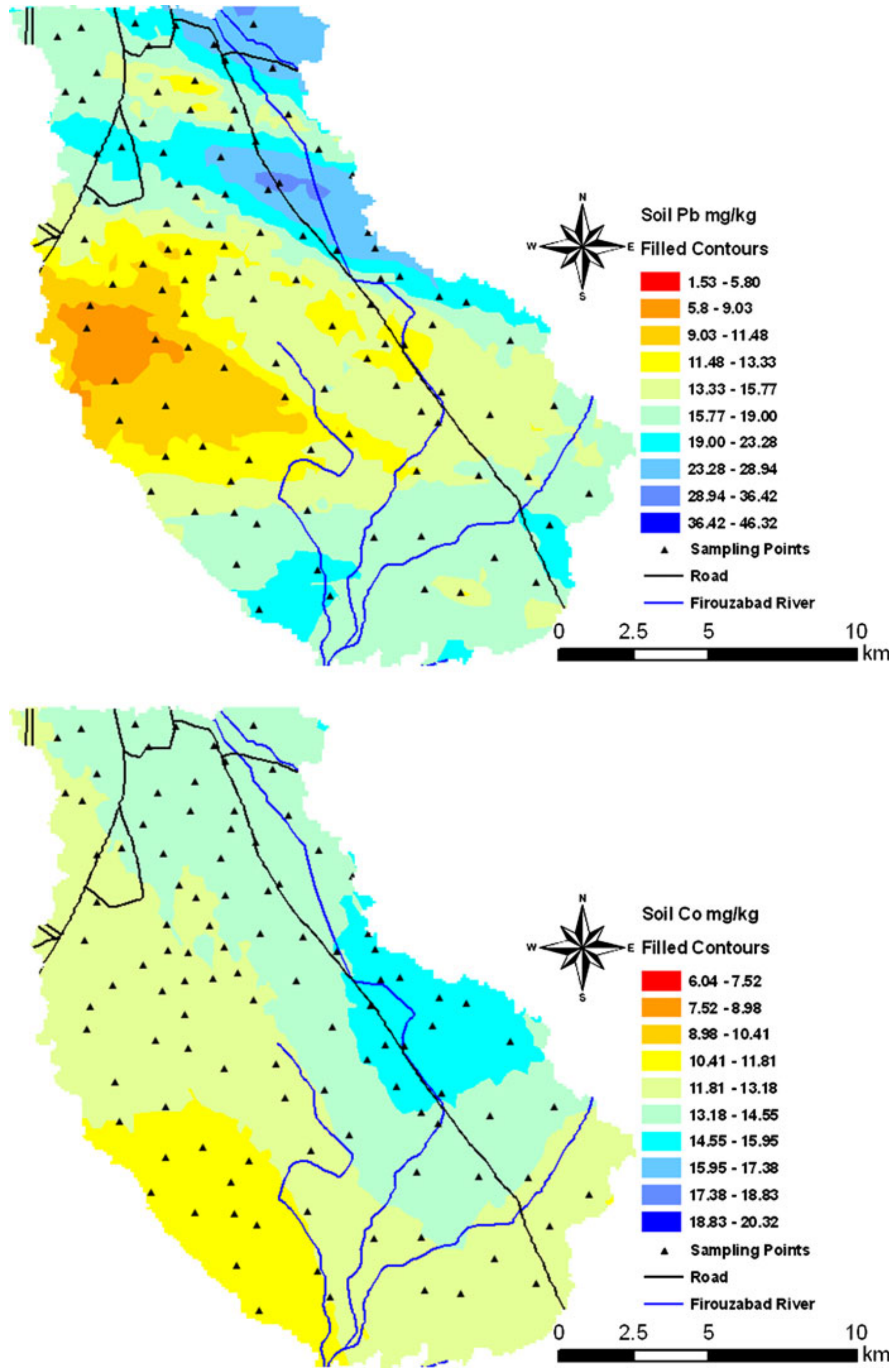
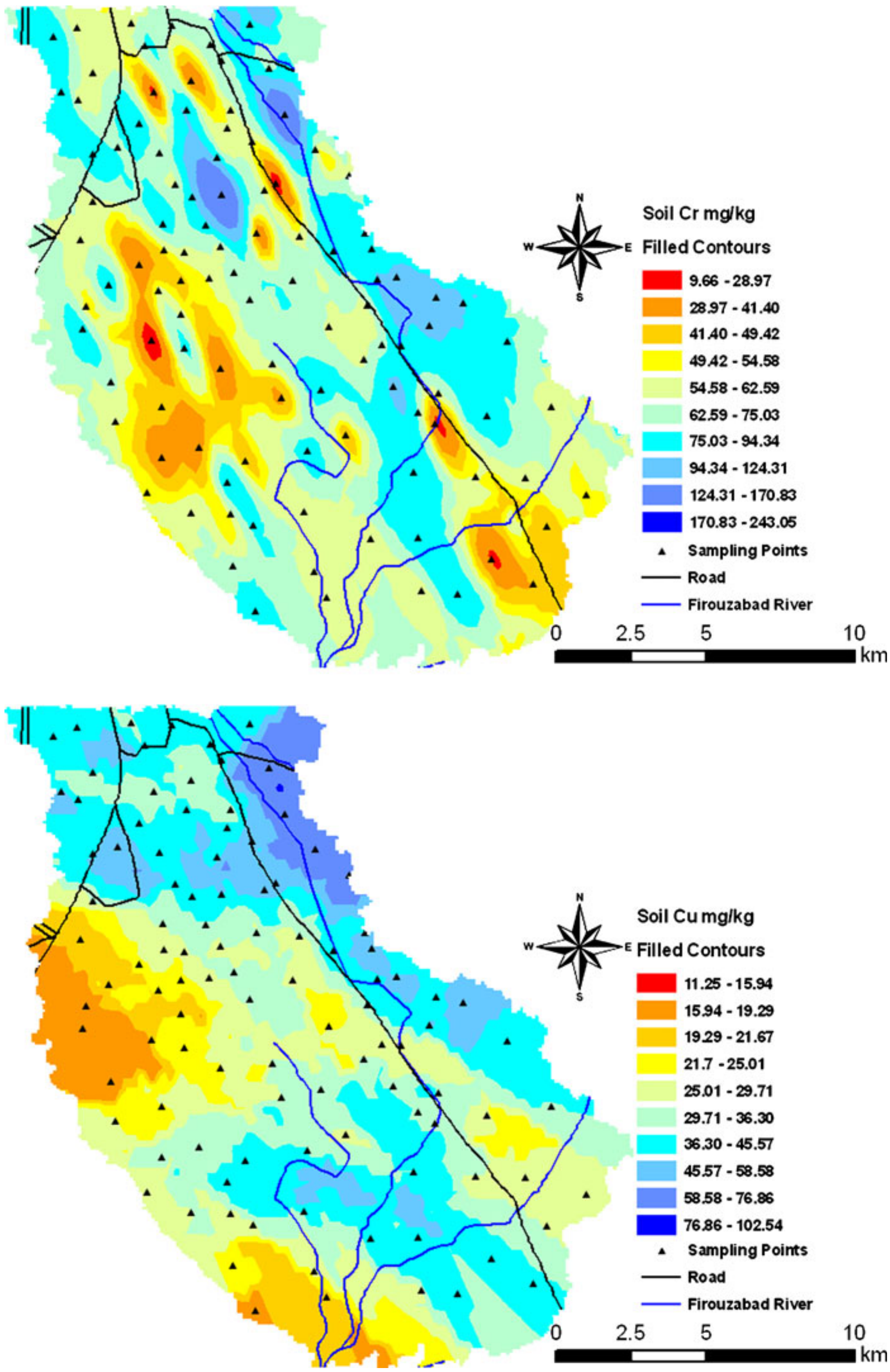


Fig. 3 (continued)



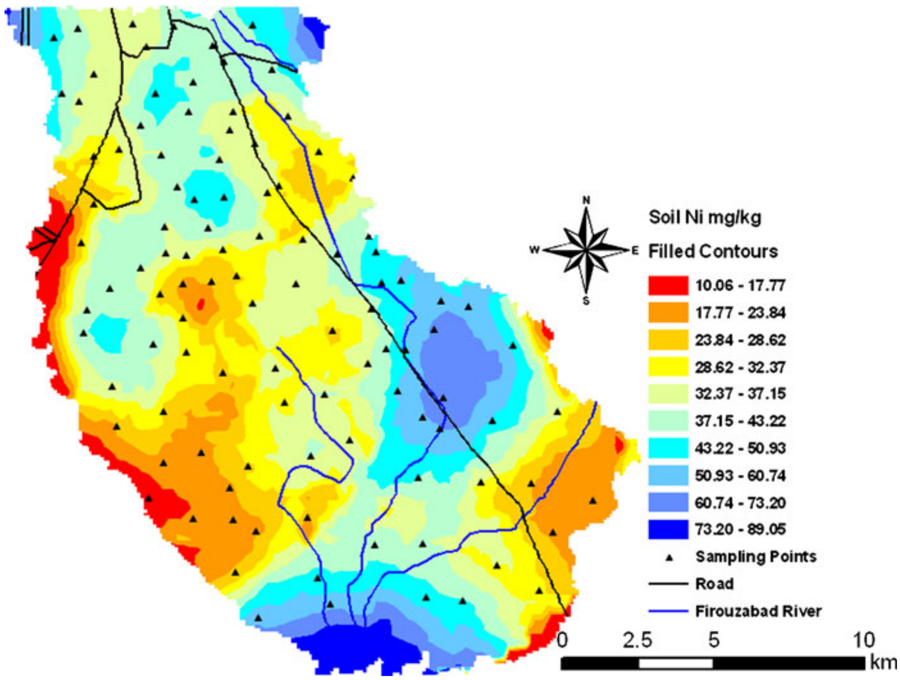
54.79, and 62.94, respectively, implied a great deal of heterogeneity in the soils and suggest that extrinsic factors, such as human activities, may be a primary source of these metals. High concentrations (i.e., above background levels) coupled with a high coefficient of variation suggest anthropogenic inputs for metal elements (Manta et al. 2002). However, some researchers (Aucejo et al. 1997; Facchinelli et al. 2001) suggest that wastewater is the main source of these elements in

Fig. 3 (continued)



agricultural soils, especially in densely populated areas with inefficient cleaning processes. The lowest CV% that we found for Cr, Co, and Ni exhibit weak variation, and their content was almost constant in the study location, suggesting that their presence is probably due to lithogenic process. Additionally, Cr, Co, and Ni levels were likely controlled by the same parent material.

Fig. 3 (continued)



Multivariate analysis

To better describe the relationship among heavy metals, PCA was performed. The results of the PCA for heavy metal concentration in the soil are presented in Tables 2 and 3. The rotation of the matrix helps to clarify ambiguities in the component matrix. Based on eigenvalues (eigenvalue > 1), the three main principal components (PCs) explained 72.49% of the total variance (Tables 2 and 3). The first PC (PC1) explains 31.95% of total variance and consists of Cr, Co, and Ni. The initial component matrix (PC1) indicates that Cr, Co, and Ni are associated, displaying high values in the first component.

This result may indicate that the contamination of Cr, Co, and Ni originate from the same sources, such as lithogenic processes. The second PC (PC2) explains 25.05% of the total variance and consists of Zn, Pb, and Cu. These metals can be defined as anthropogenic components due to the presence of high levels in some soils. Relatively lower loading factors of these metals in the first component implies that other sources, such as wastewater and industrial contamination, may control the concentrations of Zn, Pb, and Cu. Zn used in different manufactured goods (e.g., automobile tires, batteries, and electrical machines) and in agricultural fertilizer, and it is produced by motor traffic and metal-working industries.

The high Cu and Pb levels may be related to wastewater and vehicle and industrial fumes. Cu, Pb, and Zn were found to be associated with anthropogenic sources in our study, as was also shown by several previous studies (Rodriguez et al. 2006; Zhang et al. 2008). Cadmium showed greater values in the third component (PC3). The most important sources of Cd in arable soils in suburban areas are mainly from the long-term use of fertilizer, sewage sludge, and organic manure (Zhang 2006). The spatial appearance of the factor loading plots for the three rotated components is shown in Fig. 2.

Geostatistical analysis

The semivariogram experiment depicts the variance of the sample values at various separation distances. The ratio of nugget to sill (nugget/sill) can be used to express the extent of spatial autocorrelations of environmental factors. If the ratio is low (<25%), the variable has strong spatial autocorrelations at a regional scale. A high ratio of the nugget effect (>75%) indicates spatial heterogeneity of soil properties. To some extent, the spatial variability of heavy metals may be affected by intrinsic factors (i.e., pedogenic factors such as soil parent material) and extrinsic factors (i.e., anthropogenic factors such as agricultural

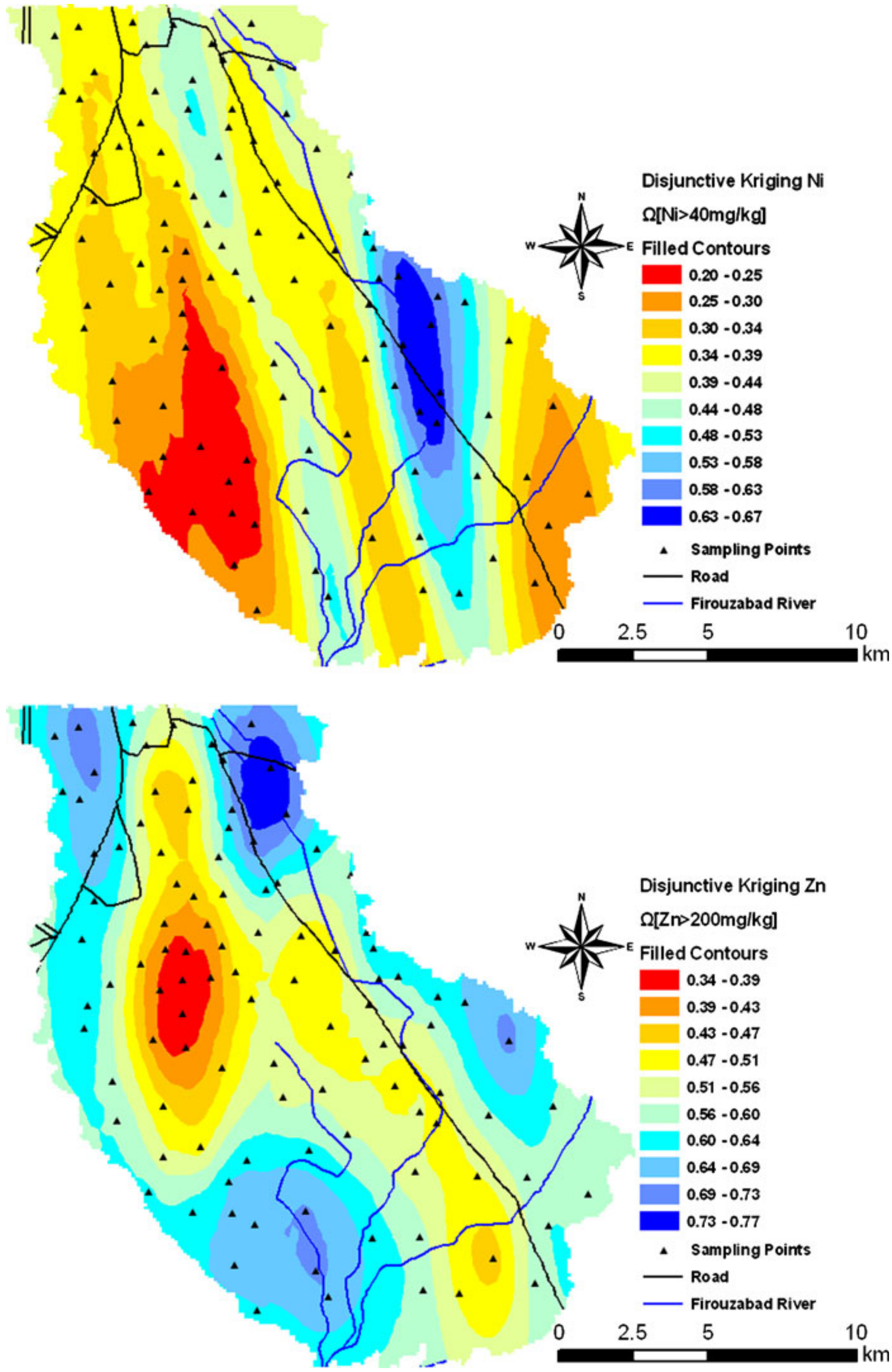
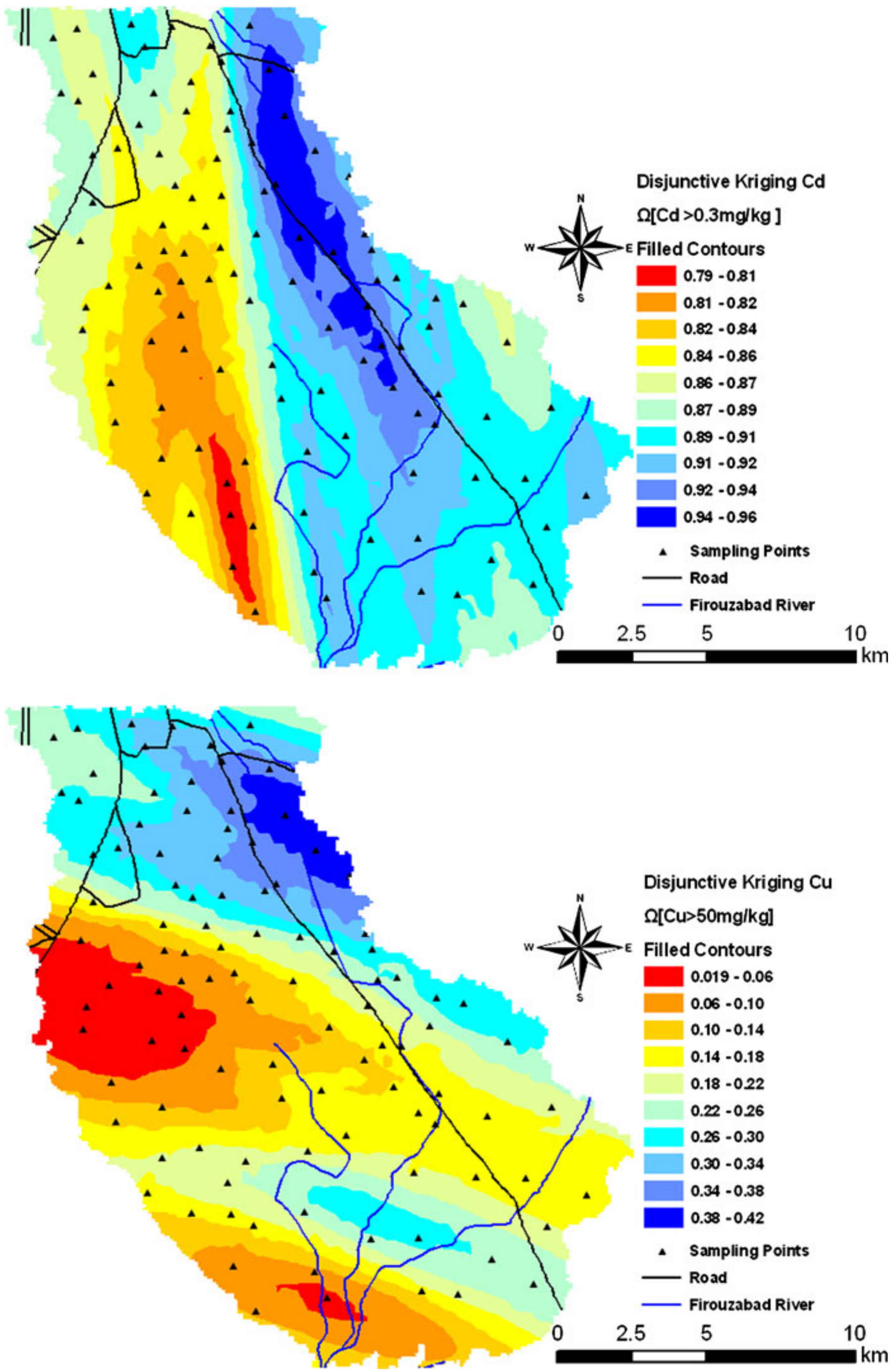


Fig. 4 Estimate probability maps of Cd, Cu, Ni, and Zn

Fig. 4 (continued)



practices). In general, strong spatial dependence of soil properties is likely to be caused by intrinsic factors, and weak spatial dependence is likely to be caused by extrinsic factors (Cambardella et al. 1994).

The ratio of nugget to sill for Zn was more than 0.75, showing weak spatial dependence. The ratios for Ni, Pb, Cd, Cu, and Co were between 0.25 and 0.75, showing moderate spatial dependence indicating that extrinsic factors (such as industrial production or agricultural practices) may have affected their spatial correlations. The ratio of nugget to sill for Cr was less than 0.25, showing strong spatial dependence due to pedogenic factors such as parent material. Additionally, there was little range in the Pb and Cr values, which may indicate their pedogenic sources. The attributes of the semivariogram model and the best-fit model parameters that were used as inputs to the kriging interpolation are summarized in Table 4.

The results show that soil Cu and Pb were fitted with a J-Bassel model, Zn and Ni with a Gaussian model and Cd, Co, and Cr with rational quadratic, pentaspherical, and K-Bassel models, respectively.

Spatial distribution and risk assessment

The distribution maps of heavy metals including Cd, Cu, Co, Pb, Zn, Cr, and Ni concentrations are illustrated in Fig. 3. These maps show the spatial variation of heavy metal concentrations generated from their semivariograms.

The distribution of Co and Ni had a clear boundary at the west of the area, and their spatial distribution maps show similar geographical trends. At the northwest of the study area, Cu, Pb, and Zn had a zone with high concentrations. Because the study sites were located around the Firuzabad River, the use of wastewater from the river obviously affects the soil heavy metal concentrations. In the distribution map of Cd, a wide area from the northeast to the southeast that crosses the outer main channel (Firuzabad) had high pollution levels. However, the arable lands were widely irrigated with urban and industrial wastewater, which had a large effect on Cu, Pb, and Zn soil concentration.

The estimated probability of exceeding the threshold was kriged by disjunctive kriging (Fig. 4). The results show that Cd, Cu, Ni, and Zn exhibited pollution risk. For soil Cd, the west of the study area had a high pollution risk, where the estimated probability (Ω [Cd $\geq$ 0.6 mg/kg]) reached 0.87–0.95. The high probabilities for exceeding of threshold value for Cd found from the north to south are highly correlated with vegetable cultivation areas. For soil Zn, the map shows that the areas with high risk are mainly located in the around the study area, where the estimated probability (Ω [Zn $\geq$ 300 mg/kg]) reached 0.61–0.81. However, the areas with low estimated probability cannot be regarded as safe for vegetable growth. The probability map of Ni exhibits a risk patch in which the highest risk area (Ω [Ni $\geq$ 60 mg/kg]) is distributed in southern Tehran, where it is likely due to the application of waste water with high concentrations of heavy metals (Table 1) and agricultural practices. The main risk sites generally occurred in the north and northeast of the study area, except for Zn, which occurred at four hotspot points. According to the land-use map, the southeast of the study area that is occupied by range is safest place in the region.

Conclusions

The heavy metal concentration in the topsoil of agricultural lands in the south of Tehran showed an increasing trend of heavy metal contamination due to wastewater irrigation and the use of fertilizers and pesticides in the past decades. The Cd, Cu, and Co levels followed a lognormal distribution, and Pb, Zn, Cr, and Ni followed a normal distribution. Based on multivariate statistical and geostatistical analyses, the concentrations of Cr, Pb, Ni, and Co were likely affected by natural factors. In fact, a point source of contamination for these heavy metals was not perceived in the study area. However, we found that the concentrations of Zn and Cu were related to anthropogenic factors (e.g., the discharge of wastewater). Finally, agrochemical inputs may play the most important role for the input of Cd. The results presented here can be used for planning, risk assessment, and decision making in the environmental management of this region.

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