



Pollution assessment and source apportionment of selected metals in rural (Bagh) and urban (Islamabad) farmlands, Pakistan

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Received: 10 September 2018 / Accepted: 28 February 2019 / Published online: 6 March 2019
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Abstract

The upsurge in agricultural food demand due to population explosion and urbanization has great impact on the quality of soil. Therefore, the present study was aimed to assess the contamination and pollution by selected metals (Ca, Cd, Co, Cr, Cu, Fe, K, Li, Mg, Mn, Na, Ni, Pb and Zn) in the soil of typical rural and urban farmlands. The metal contents were quantified by atomic absorption spectrometry employing wet acid digestion methodology. In addition, physicochemical parameters (pH, redox potential, EC, TDS, organic matter, moisture, density, porosity and total alkalinity), anion levels (sulphate, chloride, nitrate and nitrite) and soil texture was also evaluated. Average concentrations of Co, Na, Ni, Pb and Zn were significantly higher in the rural soil, while Ca, Cu and Mg contents were considerably higher in the urban soil ($p < 0.05$). The correlation study revealed significantly divergent association among the metals in both categories of soil samples. Soil texture mostly revealed sandy loam nature of the soil with higher clay and silt contents in urban soil and elevated sand contents in rural soil which was predominantly Ca–Mg–HCO₃ in composition. Significant anthropogenic contribution was shown by the cluster analysis while enrichment factors and contamination factors revealed severe to very high enrichment and moderate contamination of Cd and Pb in the soil. The study showed that elevated levels and anthropogenic enrichment of the metals in agricultural soil could pose serious health risks which should be addressed by appropriate management and sustainable agricultural practices.

Keywords Soil · Rural/urban farmland · Metal · Contamination · Enrichment · Pakistan

Introduction

Soil is a vital component of ecosystem that provides services critical for life: soil act as a reservoir of water, minerals, organic matter and myriad of micro and macro organisms (Borkar 2015). Soil acts as a sink and also as a source of pollution and have ability to transfer the pollutants to groundwater and food chain, and finally to the human beings/animals (Chary et al. 2008; Facchinelli et al. 2001; Pennisi

et al. 2016). Soil pollution is an undesirable change in physical, chemical and biological characteristics, and it results in reduction in the amount of land for cultivation. Human health is closely related to the quality of soil and especially to its degree of pollution (Romic and Romic 2003). Metals are intrinsic component of earth crust, however, today soil contamination with heavy metals is an environmental problem on global scale because of their non-biodegradable and persistence in nature (Li et al. 2004). The soil contamination may originate from both natural and anthropogenic sources and extent of contamination varies from place to place. Natural sources of soil enrichment with heavy metals are mostly geological in nature, whereas anthropogenic sources include irrigation with wastewater, smelting activity, disposal of solid waste and vehicular exhaust (Shah et al. 2010). Major causes of soil pollution include liquid or solid industrial waste, mining activities, corrosion/erosion, excessive use of fertilizers and pesticides, deforestation, acid rain and intensive farming (Gil et al. 2018; Oumenskou et al. 2018; da Silva et al. 2017).

Electronic supplementary material The online version of this article (<https://doi.org/10.1007/s12665-019-8198-z>) contains supplementary material, which is available to authorized users.

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Heavy metals can be harmful due to their potential to accumulate in different body tissues along with their very long biological half-lives for eliminating from the body (Duruibe et al. 2007). Many food plants accumulate heavy metals and result in the rise in the metal contents of farm's production. Food chain translocation of heavy metals is one of the consequences of soil contaminated with heavy metals. Excessive intake of metals through consumption of contaminated vegetables and other plants is associated with numerous human health risks (Arrobas et al. 2017; Toth et al. 2016a, b; Khan et al. 2008). The ingestion of toxic metals rich plants can induce serious clinical abnormalities in both humans and animals, such as massive depletion of some essential nutrients from the body; decrease in immunological defence; growth retardation; impaired psychosocial behaviour; and development of multifactorial diseases (Arora et al. 2008). In addition, studies of Turkdogan et al. (2003), Kashem et al. (2006) and Wang et al. (2006) revealed the link between the consumption of vegetables and fruits having elevated levels of Cd, Cu, Ni, Pb and Zn with the high prevalence of upper gastrointestinal cancer, cardiovascular disease, respiratory illness, dermatogenic problems, bone fracture, kidney dysfunction and hypertension. Moreover, the importance of physicochemical analyses of soil is greatly enhanced especially after the advent of population explosion, urbanization and increasing demand of agricultural food.

Based on the deliberations in preceding sections, the present study is based on following broad objectives: to determine the distribution of selected metals in soil samples from rural and urban farmlands; to explore the plausible associations among the metals in terms of correlation coefficients and multivariate apportionment; to monitor the physicochemical parameters and texture of the soil samples; to evaluate the contamination and enrichment as well as contributing sources of the metals in the soil samples. It is anticipated that the study would provide a baseline data related to the contamination of metals in agricultural farmlands under diverse environmental conditions.

Materials and methods

Site description

The present study included two distinctly different agricultural areas for the collection of soil samples; a typical rural area from District Bagh Azad Jammu & Kashmir (23.53°N–30.89°E) was selected while Islamabad, Pakistan (33.43°N–73.04°E) represented a typical urban area. The climate of the rural area is moderate with an average temperature of 22 °C and annual rainfall of area is about 1500 mm. The agriculture land is cultivated with vegetables, wheat,

rice, maize, jawar and bajra. The topography of rural area is mainly hilly and mountainous characterized by deep ravines, rugged, and undulating terrain. The urban area is located at the edge of Potohar plateau at foot of Margalla hills. Islamabad features a typical version of humid subtropical climate with hot, humid summer accompanied by Monsoon season followed by mild and wet winter. The temperature ranges from < 15 °C in January to > 37 °C in June. Major cultivations of area are wheat, vegetables, fruits, etc. Cultivation is generally based on systematic agriculture in both areas.

Collection and processing of soil samples

In the present study, 16 sampling locations were selected from rural farmlands; while 13 locations were selected from urban farmlands. The soil samples were collected at a depth of 0–25 cm with stainless steel auger. Each sample was collected as composite consisting of 5–10 sub-samples at a distance of about 2–10 m from the first sub-sample in different directions, at each sampling site. These sub-samples were thoroughly mixed to assemble a composite soil sample (Wu et al. 2010). The composite soil samples were brought to laboratory in polyethylene bags and air-dried and covered with cloth to prevent contamination. In the laboratory, large items in soil, such as grass, stones, pieces of wood, etc., were manually removed and then samples were dried in an electric oven at 70 °C for 48 h to achieve constant weight. After drying, the soil samples were mechanically ground and passed through a sieve of 2 mm and properly stored for analysis (Khan et al. 2010). The moisture contents (MC) of the soil were estimated by weight difference of the samples before (m_1) and after (m_2) drying in the oven (Radojevic and Bashkin 1999):

$$MC = \frac{m_1 - m_2}{m_1} \times 100.$$

Digestion of soil samples

Accurately weighed amount (1.0 g) of oven dried and sieved soil was taken in the digestion flask, followed by the addition of 5.0 mL of HNO₃, the flask was covered with watch glass and heated on hot plate for 30 min. Afterwards, the contents were cooled and 5.0 mL of nitric acid was added, heated for another 30 min. Repeated with another aliquot of 5.0 mL of concentrated HNO₃ but not covered with watch glass completely and heated the contents until volume was reduced to 3 mL. The mixture was cooled and 2.0 mL of water and 3.0 mL of 30% H₂O₂ was added. Flask was covered and heated gently. In case of vigorous effervescences, flask was removed from the hot plate. Repeatedly added 1.0 mL of 30% H₂O₂ and heated until effervescences subsided. After that 5.0 mL of concentrated HCl was added and

flask was covered with watch glass and heated for 15 min without boiling. The contents were then cooled and filtered through Whatman filter paper into a 25 mL of volumetric flask. Watch glass and conical flask was washed with water and filtered into the flask. Final volume was adjusted with 0.1 N HNO₃ and the samples were kept in refrigerator before analysis (Radojevic and Bashkin 1999). A blank passing through all steps along with the sample was also prepared with each batch of five samples. Hence, every care was taken to assure the quality of finished data.

Instrumental analysis

Quantitative measurement of the soil samples was performed for Ca, Cd, Co, Cr, Cu, Fe, K, Li, Mg, Mn, Na, Ni, Pb and Zn on flame atomic absorption spectrophotometer (Shimadzu AA-670, Japan) under optimum analytical conditions as shown in Table 1. Reliability of the metals data were ensured by analysing the standard reference material which showed very good recovery (Table 1). Moreover, in the present study, pH, EC, TDS and redox potential of soil samples were measured in their water extract (5 g soil + 50 mL distilled water). pH of the soil samples was measured by the help of pH meter which was pre-calibrated using a three-point calibration with buffer solution of pH 4, 7 and 9 (Arain et al. 2008; Radojevic and Bashkin 1999). Electrical conductivity (EC), total dissolved solids (TDS) and redox potential of the soil samples was measured using multimeter which was pre-calibrated with 0.1 M and 0.01 M KCl solutions.

In this study, organic matter was measured by the method as described by Radojevic and Bashkin (1999). Soil sample (1.000 g) was dried in oven at 105 °C to the constant weight (m_1). This sample was poured in to the pre-weighed crucible and few drops of H₂O₂ were added to promote the oxidation. The sample was placed into the muffle furnace with gradually increasing the temperature to 1000 °C. Sample was left there for 4 h. After ignition sample was cooled in

a desiccator and again weighed (m_2). Organic matter (%) in terms of loss on ignition (L) was calculated by following relationship (Pansu and Gautheyrou 2007):

$$L = 100 \times \frac{m_1 - m_2}{m_1}.$$

Soil texture

The soil texture is directly related to its mineral composition and particle size distribution which is considered as one of the fundamental characteristics of soil (Greve et al. 2012). The distribution of particle sizes larger than 75 µm (retained on No. 200 sieve) can be determined by sieving, while the distribution of particle sizes smaller than 75 µm can be determined by a sedimentation process, using a hydrometer. In the present study particle size of soil was determined by the ASTM 422 (1998) method. Briefly, 50.0 g air dried soil was taken and mixed with the 125 mL of the dispersing agent (sodium hexametaphosphate, 40 g/L) solution. Mixture was stirred until the soil was thoroughly wet. Let the soil soak for at least 16 h. 125 mL of dispersing agent was added into the control cylinder and filled with distilled water up to the mark. Hydrometer and thermometer were inserted into the control cylinder and zero correction and temperature were recorded. After 16 h soil slurry was thoroughly mixed and transferred immediately into the empty sedimentation cylinder and filled with distilled water up to the mark. Readings were taken after elapsed time of 40 s and 2, 5, 8, 15, 30, 60 120 min and 24 h. After taking the final hydrometer reading, the suspension was transferred to a No. 200 (75 µm) sieve and washed with tap water until the wash water was clear. Material retained on the No. 200 sieve was transferred to a suitable container, dried in an oven at 105 °C and passed through the series of sieves. Mass of each fraction retained on each sieve was determined and percentage of each fraction was calculated.

Table 1 Optimum analytical conditions maintained on AAS for the analysis of selected metals using air-acetylene flame (Shimadzu AA-670, Japan)

Metal	Ca	Cd	Co	Cr	Cu	Fe	K	Li	Mg	Mn	Na	Ni	Pb	Zn
Wavelength (nm)	422.7	228.8	240.7	357.9	324.8	248.3	766.5	670.7	285.2	279.5	589.0	232.0	217.0	213.9
HC lamp current (mA)	6.0	4.0	6.0	5.0	3.0	8.0	5.0	4.0	4.0	5.0	6.0	4.0	7.0	4.0
Slit width (nm)	0.50	0.30	0.20	0.50	0.50	0.20	0.50	0.50	0.50	0.40	0.50	0.15	0.30	0.50
Fuel-gas flow rate (L/min)	2.0	1.8	2.2	2.6	1.8	2.0	1.9	1.6	1.6	1.9	1.6	1.7	1.8	2.0
Detection limit (µg/L)	4	4	4	6	4	6	4	1	1	3	2	2	10	2
NIST SRM-2711														
Certified level	28,800	41.7	10	47	114	28,900	24,500	–	10,500	638	11,400	20.6	1162	350.4
Measured level	27,984	40.97	9.60	47.55	114.8	27,800	24,100	–	10,266	654.8	11,130	21.11	1179	341.2
Recovery (%)	97	98	96	101	101	96	98	–	98	103	98	102	102	97

Soil bulk density and soil porosity

In the present study, soil density was determined by following method. Soil sample was dried in oven at 105 °C to the constant weight. Sample was weighed and poured in the measuring cylinder a little at a time while gently trapping the cylinder to compact it. Volume was measured (1 mL = 1 cm³) (Carter and Gregorich 2007; Lestariningsih and Hairiah 2013). Then bulk density (BD) was calculated by formula:

$$\text{BD (g/cm}^3\text{)} = \text{Weight (g)}/\text{Volume (cm}^3\text{)}.$$

Soil porosity (SP) was calculated by following relationship:

$$\text{SP} = 1 - \left(\text{BD (g/cm}^3\text{)}/2.65 \right),$$

where, 2.65 is soils specific gravity or particle density.

Determination of anions by HPLC

In the present study, anions (sulphate, chloride, nitrate and nitrite) were measured by instrument LC-10 KVP (Shimadzu, Japan). Various parameters are as under:

Mobile phase Potassium hydrogen phthalate was used as mobile phase (1.2 mM, pH 4.2, with flow rate of 1.5 mL/min); oven temperature during the analysis was kept at 40 °C.

Stationary phase The column was packed with the anion exchange resins of polymethacrylate, supported with the particle size of 10 µm incorporating a quaternary ammonium based as functional groups, suitable for pH ranges from 2 to 11.

Detector Instrument was equipped with conductivity detector (CDD—10 AVP).

Soil alkalinity

In the present study, alkalinity of the soil samples was measured in their water extract (5 g soil + 50 mL distilled water) by titrating it with the standard 0.0255 M HCl using phenolphthalein as an indicator (if the pH of solution is greater than 8.3). This is called as phenolphthalein alkalinity or carbonate alkalinity. In case of soil extract having pH less than 8.3, only total alkalinity by titrating extract with standard HCl using methyl orange as an indicator was measured. This is called as bicarbonate alkalinity. Final results were expressed in form of mg of CaCO₃/L, and mg of HCO₃⁻/L using formula:

$$\text{Alkalinity} = \frac{V_t \times M}{V_s} \times 1000,$$

where, V_s = volume of sample used; V_t = volume of standard acid (mL); M is mass in mg of CaCO₃ equivalent to 1 mL of titrant (5.000 mg/L for 0.1 M HCl) (Radojevic and Bashkin 1999).

Assessment of contamination/enrichment

The extent of metal pollution due to anthropogenic activities in the soils can be assessed using enrichment factor (EF) (Duan et al. 2010; Sutherland 2000). EFs can be calculated using the relationship:

$$\text{EF} = \frac{[X/M_{\text{ref}}]_{\text{sample}}}{[X/M_{\text{ref}}]_{\text{crust}}},$$

where, [X/M_{ref}]_{sample} and [X/M_{ref}]_{crust} refer, respectively, to the ratios of mean concentrations (mg/kg, dry weight) of the target metal and 'M_{ref}' in the soil and continental crust. In the present study, Fe was used as a reference element.

The potential soil contamination can be assessed using contamination factor (C_f) and degree of contamination (C_{deg}) (Hakanson 1980; Abraham and Parker 2008). In case of C_f, the mean metal concentration estimated in the soil is compared to the baseline reference level in earth crust according to the following relationship:

$$C_f = C_n/C_b,$$

where, 'C_n' and 'C_b' refer to the mean concentration of a metal in the soil and earth crust, respectively.

The contamination factor (C_f) of each metal is considered as the measure of the contamination by individual metal in the soil (Abraham and Parker 2008). The numeric sum of individual contaminant factors expresses the overall degree of soil contamination (C_{deg}) using the following relationship (Hakanson 1980):

$$C_{\text{deg}} = \sum_{i=1}^{i=n} C_f^i,$$

where, 'C_f' is the single metal index, the sum of contamination factors of all estimated metals represents the degree of contamination 'C_{deg}'.

Abraham and Parker (2008) presented a generalized form of the above equation for the calculation of the overall degree of contamination at a given sampling site in terms of modified degree of contamination (mC_d). The modified equation for a generalized approach to calculating the degree of contamination is given below:

$$\text{mC}_d = \frac{\left(\sum_{i=1}^{i=n} C_f^i \right)}{n},$$

where, n = number of analyzed elements; i = i th element; C_i = contamination factor.

Statistical analysis

Basic statistical parameters included minimum, maximum, mean, median, standard deviation, standard error and skewness along with the Spearman correlation coefficients were computed in this study. Multivariate cluster analysis was also performed on the data-set using the STATISTICA software (1999).

Results and discussion

Concentration of selected metals in soil

Basic statistical parameters pertaining to the distribution of the metal concentrations (mg/kg, dry weight) in the soil samples collected from rural and urban farmlands are shown in Table 2. In the soil of rural farmlands, highest mean level was shown by Ca (32,536 mg/kg), followed by Mg (5700 mg/kg) and Fe (5338 mg/kg). Mean levels of K (1149), Mn (331.6), Na (251.6), Zn (74.02), Pb (54.59), Cr (22.17), Cu (15.78), Li (15.03), Co (15.01) and Ni (13.38) were relatively lower in the soil samples. Among the selected metals, Cd was estimated at the lowest concentration (1.078 mg/kg). In the soil of urban farmlands, predominantly higher average concentrations were noted for Ca (48,514 mg/kg), Mg (6484 mg/kg) and Fe (4976 mg/kg), followed by relatively lower levels of K (1115 mg/kg), Mn (354.2 mg/kg), Na (171.8 mg/kg), Zn (45.76 mg/kg),

Pb (44.48 mg/kg), Cu (26.02 mg/kg), Cr (21.75 mg/kg), Li (15.26 mg/kg), Co (11.77 mg/kg) and Ni (9.655 mg/kg). Lowest mean level was noted for Cd (1.084 mg/kg). Average level of the metals in the soil of rural farmlands increases in the following order: Cd < Ni < Co < Li < Cu < Cr < Pb < Zn < Na < Mn < K < Fe < Mg < Ca, while for urban farmlands the order was: Cd < Ni < Co < Li < Cr < Cu < Pb < Zn < Na < Mn < K < Fe < Mg < Ca. Among the selected metals, Cd, Co, Cr and Li showed somewhat normal distribution pattern in the soil samples of both rural and urban farmlands as manifested by very small SD and SE values. Fairly symmetric distribution was exhibited by Fe, K, Li and Mn as shown by lower skewness values for the soil samples of rural farmlands, whereas, Cd, Co, Cr, Fe, Li, Mg and Na exhibited rather symmetric distribution in the soil samples of urban farmlands.

The quartile distribution of selected metals in the soil of rural and urban farmlands (Figure S1) revealed that among the metals, Cu, Na, Ni and Zn showed significant disparity in their quartile distribution pattern in the soil samples of both farmlands, while rest of the metals revealed approximately similar quartile distribution pattern. Moreover, very broad range spread over several orders of magnitude was manifested by Ca, Cd, Cu, Na and Pb with appreciable asymmetry in the rural soil samples, whereas very broad range and predominantly asymmetric distribution was exhibited by Ca, Cd, Cu, Ni and Zn in case of urban soil. However, the rest of the metals depicted narrow quartile distribution in both rural and urban soil. Two-tailed student's t test of the data (Table 2) revealed that average levels of Co, Na, Ni, Pb and Zn were significantly higher in the soil from rural farmland, while Ca, Cu and Mg contents were considerably higher

Table 2 Statistical distribution parameters for selected metal levels (mg/kg, dry weight) in the soil from rural and urban farmlands

	Rural area							Urban area							p values
	Min	Max	Mean	Median	SD	SE	Skew	Min	Max	Mean	Median	SD	SE	Skew	
Ca	2509	78,572	32,536	25,900	23,098	5775	0.502	5507	115,560	48,514	39,871	31,554	8752	0.789	<0.05
Cd	0.050	2.675	1.078	0.850	0.724	0.181	0.759	0.012	2.675	1.084	1.125	0.744	0.206	0.398	NS
Co	10.45	22.18	15.01	13.93	3.480	0.870	0.794	5.375	21.68	11.77	10.45	5.246	1.455	0.455	<0.05
Cr	15.95	29.85	22.17	21.80	3.436	0.859	0.507	16.18	28.43	21.75	23.05	4.150	1.151	0.036	NS
Cu	0.777	40.65	15.78	14.42	8.547	2.137	1.550	12.90	119.7	26.02	18.90	28.57	7.923	3.425	<0.05
Fe	4992	5764	5338	5358	222.1	55.53	0.076	4607	5304	4976	5014	244.3	67.77	-0.076	NS
K	947.1	1327	1149	1152	97.69	24.42	0.049	929.0	1242	1115	1126	92.12	25.55	-0.550	NS
Li	11.78	18.80	15.03	14.88	2.208	0.552	0.385	10.85	21.00	15.26	14.05	3.605	1.000	0.256	NS
Mg	4067	8195	5700	5343	1115	278.8	0.776	4017	9228	6486	6779	1520	421.5	0.032	<0.05
Mn	234.2	412.3	331.6	329.5	51.07	12.77	-0.397	256.7	410.0	354.2	356.3	44.41	12.32	-0.882	NS
Na	71.30	2012	251.6	127.6	471.5	117.9	3.942	112.6	242.8	171.8	176.2	35.91	9.960	0.358	<0.05
Ni	7.550	24.10	13.38	12.70	5.054	1.263	0.920	0.150	16.88	9.655	11.80	5.853	1.623	-0.635	<0.05
Pb	22.83	143.5	54.59	48.76	32.24	8.616	1.750	19.43	91.10	44.48	34.63	24.58	6.816	1.029	<0.05
Zn	35.75	130.4	74.02	62.60	31.56	7.889	0.947	7.530	94.90	45.76	37.68	21.37	5.927	0.778	<0.05

NS Non-significant

in the soil of urban farmland ($p < 0.05$). Rest of the metals (Cd, Cr, Fe, K, Li and Mn) revealed almost comparable levels in rural and urban farmlands. Elevated levels of the metals in agricultural soil especially the rural farmland may be associated with excessive use of fertilizers, agricultural sprays and waste emissions (Arrobas et al. 2017; da Silva et al. 2017). Although there are no uniform criteria for the standard levels of trace metals in agricultural soil but some of the recent studies (Reimann and de Cartat 2017; Toth et al. 2016a, b; McIlwaine et al. 2014; Ander et al. 2013) follow the standards set in the Finnish legislation for contaminated soil (MEF 2007). In comparison with the Finnish standard values, average concentration of Cd in the rural and urban farmlands measured in the present study were slightly higher than the threshold value (1 mg/kg) but less than the guideline values (20 mg/kg). However, the mean metal levels found in the agricultural soil in the present study were noticeably lower than the threshold values of Co (20 mg/kg), Cr (100 mg/kg), Cu (100 mg/kg), Ni (50 mg/kg), Pb (60 mg/kg) and Zn (200 mg/kg) set by Ministry of Environment of Finland (2007). Therefore, the average metal levels in the soil of rural and urban farmlands were within the recommended values.

Physicochemical parameters of soil

Statistical summary related to the distribution of physicochemical parameters in the water-soluble fraction of soil from rural and urban farmland is given in Table 3. In case of rural soil, pH remained slightly basic for all soil samples varying from 7.39 to 8.28 with the mean value of 7.92, which revealed predominantly alkaline nature of the soil in study area. But in urban soil, pH remained slightly acidic to slightly basic for all soil samples varying from 6.87 to 8.14 with mean value 7.42. In addition, electrical conductivity (EC) showed large variations in both cases; for rural farmlands it ranged from 383 to 719 $\mu\text{S/cm}$, with average value of 548.9 $\mu\text{S/cm}$, while for urban farmlands EC ranged from 412 to 1047 $\mu\text{S/cm}$, with mean value of 619.5 $\mu\text{S/cm}$. Moreover, EC exhibited random distribution as indicated by elevated SD and SE values in rural as well as urban soil. Likewise, TDS also showed similar characteristics with minimum value of 268.1 mg/L to maximum value of 503.4 mg/L, and mean value of 384.5 mg/L in rural farmlands, whereas in case of urban farmlands, TDS exhibited minimum value of 288.9 mg/L to maximum value of 733.9 mg/L, and mean value of 434.1 mg/L. Higher values of TDS and EC in urban soil as compared to the rural soils are usually associated with elevated concentration of soluble ions particularly the metals salts in urban farmland. Rural and urban soil exhibited the redox potential with mean values of -59.9 mV and -34.7 mV respectively which pointed out the oxidizing conditions in both types of soil samples.

Table 3 Statistical summary of the physicochemical parameters in soil from rural and urban farmlands

	Rural area						Urban area							
	Min	Max	Mean	Median	SD	SE	Skew	Min	Max	Mean	Median	SD	SE	Skew
pH	7.390	8.280	7.920	7.990	0.290	0.070	-0.670	6.870	8.140	7.470	7.480	0.350	0.100	0.030
Redox potential (mV)	-80.00	-26.90	-59.90	-63.70	16.57	4.140	0.790	-71.50	-6.300	-34.70	-38.80	19.38	5.370	-0.050
EC ($\mu\text{S/cm}$)	383.0	719.0	548.9	538.0	115.4	28.84	0.180	412.0	1047	619.5	540.0	182.8	50.71	1.250
TDS (mg/L)	268.1	503.4	384.5	376.7	80.79	20.20	0.180	288.9	733.9	434.1	378.4	128.0	35.50	1.250
Sulphate (mg/kg)	2.148	13.69	7.153	7.107	3.010	0.750	0.160	2.176	29.01	11.41	7.667	7.950	2.210	1.290
Chloride (mg/kg)	0.700	28.73	15.46	13.67	10.32	2.580	-0.030	2.230	81.26	27.11	13.71	26.08	7.230	0.940
Nitrate (mg/kg)	1.220	117.6	21.23	20.60	42.91	16.22	2.550	0.730	176.5	59.05	40.64	68.07	21.53	1.020
Nitrite (mg/kg)	10.87	67.15	29.15	19.29	26.01	13.01	1.710	11.46	39.24	25.54	23.58	10.19	4.560	-0.060
Organic matter (%)	8.184	18.88	12.04	11.83	2.637	0.659	0.924	3.397	19.76	10.42	10.18	3.960	1.098	0.814
Density (g/cm^3)	1.003	1.339	1.177	1.167	0.093	0.023	0.123	1.133	1.431	1.256	1.253	0.093	0.026	0.424
Porosity (%)	49.48	62.17	55.58	55.95	3.507	0.877	-0.123	45.98	57.24	52.60	52.70	3.504	0.972	-0.424
Total alkalinity	127.5	357.0	216.0	197.6	60.25	15.06	0.680	63.75	178.5	128.5	140.3	34.32	9.520	-0.640

Measurement of anions by HPLC (Table 3), showed higher mean value of nitrate (21.23 mg/kg) followed by chloride (15.46 mg/kg) and sulphate (7.153 mg/kg) in rural soil, while similar results were obtained for the urban soil with higher mean levels of nitrate (59.05 mg/kg) followed by chloride (27.11 mg/kg) and sulphate (11.41 mg/kg). Anions levels in urban soil samples were relatively higher and randomly distributed as compared to the rural soil. Only few samples showed the nitrite concentration in rural as well as urban soil samples. However, the mean value of nitrite in rural farmland (29.15 mg/kg) was slightly higher than urban farmland (25.54 mg/kg).

Density of the soil from rural farmland ranged from 1.003 to 1.339 g/cm³ with the mean value of 1.177 g/cm³ which indicated high percent porosity value (mean value = 55.58%). Moreover, low values of bulk density govern the high sand contents as compared to the percent clay and silt. On the other hand, density values for the urban soil samples varied from 1.133 to 1.431 g/cm³ with the mean value of 1.256 g/cm³ which were slightly higher than the rural soils indicating the lower percent porosity (mean value = 52.60%). Furthermore, high values of bulk density govern the low sand contents compared with the rural soils (Table 3). Similarly, Table 3 showed mean values of percent organic matter in rural (12.04%) and urban (10.42%) soil samples. High values of organic contents are generally associated with the use of organic manure as fertilizer in the rural farmland.

Phenolphthalein alkalinity was not detected in any soil sample from rural and urban farmland; however, total alkalinity in rural (216.0 mg/L of CaCO₃) and urban soil (128.5 mg/L of CaCO₃) was mostly associated with bicarbonate ions in the study area. As pH of extracts were less than 8.3 so the possibility of the presence of soluble carbonates were ruled out. The alkalinity results are in good agreement with pH results of present study. Low mean value (1.971%) for moisture content of rural soil showed lower water holding capacity of the soil and high average value of moisture contents of urban soil (4.24%) indicated the higher water holding capacity of the soil (Table 3).

The physicochemical analysis of soil is vital to agricultural chemists for plants growth and soil management. Soil pH strongly influences the biomass, activity and composition of the microbial community in the soil (Rousk et al. 2010). Basic soils have high contents of base cations (K⁺, Ca²⁺, Mg²⁺ and Na⁺) and carbonates/bicarbonate anions due to an accumulation of soluble salts (Radojevic and Bashkin 1999). Moreover, increasing the pH of acidic soils may increase the plant availability of macronutrients. Though soil acidification is a very slow natural process, but human activities have considerably accelerated this process from last several decades (Zhao et al. 2011).

Soil electrical conductivity is strongly correlated with soil physical properties that have an effect on crop productivity,

including soil texture, cation exchange capacity (CEC), drainage conditions, organic matter level, temperature, salinity and subsoil characteristics. The electrical conductivity of soils varies depending on the total soluble ion concentrations and the amount of moisture detained by soil particles. Sands have a low conductivity; silts have a medium conductivity, while clays have a high conductivity. Therefore, EC correlates strongly to TDS and soil particle size and texture (Al-Rashdi and Sulaiman 2015; Brevik et al. 2004; Babcock et al. 2009).

Redox potential is the geochemical mobility of pollutants and nutrients (especially S, N, P and heavy metals) in various compartments of environments and consequently their influence on ecosystem. The relative degree of oxidation or reduction in the soil has marked effect on its nature, chemical reactions, microbial population and associated terrestrial population. Microbial respiration in soil provides the electrons that derive the most redox reaction which can affect the speciation of nutrients either directly or indirectly. Well aerated surface soil has high value of redox potential indicating the oxidising conditions, while deeper layers may be completely devoid of oxygen giving rise to the highly reducing condition and low potential (Husson et al. 2016; Radojevic and Bashkin 1999).

Soil organic matter (SOM) is very important from the view point of soil fertility management. Its content serves as a strategic means to safeguard long term farm productivity, especially after the declined in SOM levels over the last century as a result of anthropogenic activities. Thus, from a practical agricultural stand point, it is important for two main reasons: (1) as a “revolving nutrient fund”; and (2) as an agent to improve soil structure, maintain till and minimize erosion (Hijbeek et al. 2018).

Bulk density is inversely related to the pore space and has important influence on root penetration and soil permeability, which in turn can affect the flow of material (air, water, nutrients and pollutants) within soil. Soils having high bulk densities have low pore spaces and therefore, low fertility and are also inhibitive to root penetration. In present study urban soil showed higher bulk density than rural soil (Table 3) (Lestariningsih and Hairiah 2013; Radojevic and Bashkin 1999).

Soil texture

Spatial distribution and variability of the various soil texture fractions (coarse sand, fine sand, silt and clay) is increasingly being required for input into ecological, hydrologic, climatic and other environmental models, particularly due to ever-rising environmental concerns relating to the prognosis for agricultural yields and carbon stocks at a global level (Greve et al. 2012). Ternary diagram for the soil texture from rural and urban farmlands is shown in Fig. 1a.

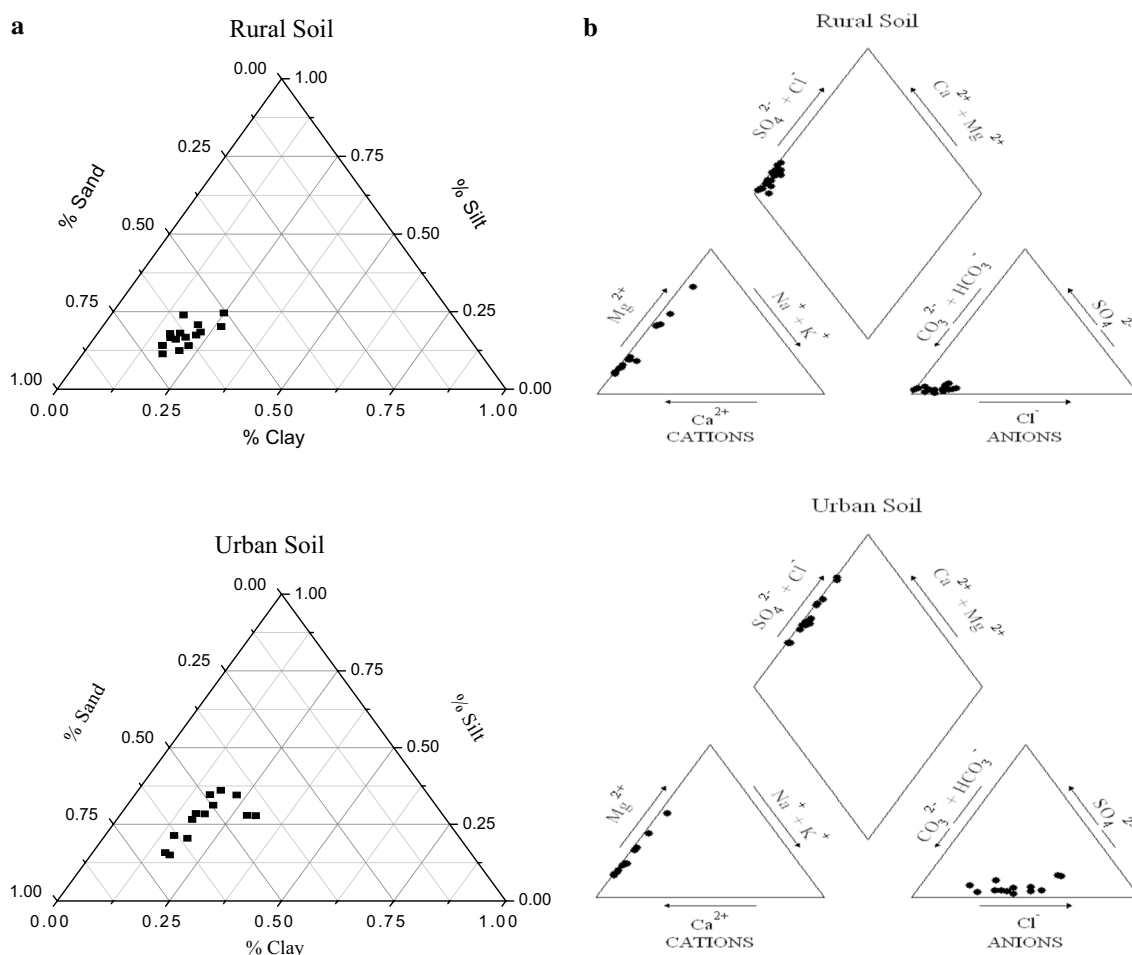


Fig. 1 a Ternary diagram for the soil from rural and urban farmlands. b Piper diagram showing the predominant chemical nature of soil from rural and urban farmlands

The rural soil samples contained high percentage of sand and low percentages of silt and clay. All the samples lied in the triangle of sandy loam. Sandy loam contains 17% clay, 64% sand and 19% silt. Due to the high percentage of sand, these samples have low bulk density and high porosity, low compatibility and low moisture contents as well as low water holding capacity. The urban soil showed the diverse nature as the samples belonged to two classes and four different categories; sandy loam (17% clay, 64% sand, 19% silt), loam (equal proportion of sand, silt and clay), sandy clay loam (20–30% clay, 50–70% sand, 10–20% silt) and clay loam (30–40% clay, 30–50% silt, 20–50% sand). On comparative basis, the urban soil was found to be much fertile compared to the rural soil as it contained the higher percentage of silt and clay and low sand contents.

Chemical nature of soil

To assess the predominant chemical nature and composition of the soil from rural and urban farmlands, piper diagram

was constructed, and the results are shown in Fig. 1b. The soil samples from rural farmland were found to be predominantly Ca–Mg–HCO₃ in nature with high proportions of Ca, Mg and bicarbonate. Most of the samples belong to the same category and alkaline in nature. However, the soil samples from urban farmland revealed mixed nature composed of Ca–Mg–HCO₃ and Ca–SO₄; hence no single composition was dominant in the urban soils. Some of the soil samples exhibited higher Ca, Mg and bicarbonate contents while others showed elevated chloride and sulphate contents. Overall, the soil samples from two farmlands were diverse in nature.

Correlation study of selected metals in soil

The correlation coefficient matrix pertaining to the metal levels in the soil samples from rural and urban farmlands is shown in Table 4, wherein bold *r* values are significant at $p < 0.01$. The magnitude of the correlation coefficients is generally considered as an indicative of mutual association and in most of the cases shared origin of the

Table 4 Correlation coefficient matrix for selected metals in soil from rural (below the diagonal) and urban (above the diagonal) farmlands

	Ca	Cd	Co	Cr	Cu	Fe	K	Li	Mg	Mn	Na	Ni	Pb	Zn
Ca	1	-0.096	0.253	0.168	0.313	-0.081	0.390	0.427	0.708	0.416	0.002	-0.035	-0.198	-0.126
Cd	0.366	1	0.627	0.663	0.303	0.279	0.652	0.456	0.071	0.111	0.107	0.725	0.401	0.374
Co	0.022	0.093	1	0.922	0.344	0.505	0.795	0.720	0.237	0.430	0.098	0.620	0.538	0.407
Cr	0.047	0.496	0.658	1	0.438	0.715	0.867	0.853	0.356	0.641	0.259	0.580	0.449	0.249
Cu	0.351	0.643	0.245	0.574	1	0.214	0.532	0.463	0.559	0.268	0.182	0.209	-0.056	0.046
Fe	-0.414	0.289	-0.018	0.497	0.112	1	0.490	0.767	0.255	0.768	0.179	0.140	0.215	-0.091
K	0.080	0.404	0.286	0.793	0.636	0.500	1	0.862	0.586	0.502	0.366	0.663	0.188	0.227
Li	-0.412	0.089	0.200	0.426	0.074	0.755	0.350	1	0.653	0.791	0.265	0.408	0.135	-0.019
Mg	0.528	0.084	-0.082	0.176	0.086	0.218	0.185	0.315	1	0.535	0.099	0.155	-0.193	-0.237
Mn	-0.305	0.210	0.249	0.531	0.320	0.585	0.476	0.696	0.104	1	0.225	0.012	0.032	-0.235
Na	-0.089	-0.224	0.059	-0.264	-0.137	-0.288	-0.493	-0.157	-0.189	-0.090	1	0.040	0.196	0.276
Ni	0.145	0.342	-0.004	0.365	0.350	0.506	0.488	0.157	0.218	0.197	-0.144	1	0.180	0.511
Pb	0.217	0.343	0.287	0.618	0.577	0.370	0.704	0.186	0.372	0.291	0.699	0.453	1	0.641
Zn	-0.006	0.272	0.637	0.704	0.479	0.444	0.667	0.367	0.054	0.416	-0.267	0.289	0.653	1

Bold *r* values are significant at $p < 0.01$

metals. In the soil of rural farmlands, strong positive correlations were observed between K–Cr ($r = 0.793$), Li–Fe ($r = 0.755$), Pb–K ($r = 0.704$), Zn–Cr ($r = 0.704$), whereas, Pb–Na ($r = 0.699$), Mn–Li ($r = 0.696$), Zn–K ($r = 0.667$), Cr–Co ($r = 0.658$), Zn–Pb ($r = 0.653$), Cu–Cd ($r = 0.643$), Zn–Co ($r = 0.637$), Cu–K ($r = 0.636$), Cr–Pb ($r = 0.618$) and Mn–Fe ($r = 0.585$), Pb–Cu ($r = 0.577$), Cu–Cr ($r = 0.574$), Cr–Mn ($r = 0.531$), Mg–Ca ($r = 0.528$) and Ni–Fe ($r = 0.506$) exhibited significantly positive correlations, indicating their probable common origin in soil from rural farmland. Rest of the metal pairs showed either weak positive or negative correlations manifesting their independent variations. The correlation study thus indicated multiple and diverse sources of the metals in the rural soil.

In the soil of urban farmland (Table 4), among all the metals, strongest correlation was noted between Cr–Co ($r = 0.922$), while strong positive correlations were noted between K–Cr ($r = 0.867$), Li–K ($r = 0.862$), Li–Cr ($r = 0.853$), K–Co ($r = 0.795$), Mn–Li ($r = 0.791$), Mn–Fe ($r = 0.768$), Li–Fe ($r = 0.767$), Ni–Cd ($r = 0.725$), Li–Co ($r = 0.720$), Fe–Cr ($r = 0.715$) and Mg–Ca ($r = 0.708$). In addition, significantly positive correlations were observed between Na–Pb ($r = 0.699$), Ni–K ($r = 0.663$), Cr–Cd ($r = 0.663$), Mg–Li ($r = 0.653$), K–Cd ($r = 0.652$), Mn–Cr ($r = 0.641$), Zn–Pb ($r = 0.641$), Co–Cd ($r = 0.627$), Ni–Co ($r = 0.620$), Mg–K ($r = 0.586$), Ni–Cr ($r = 0.580$), Mg–Cu ($r = 0.559$), Pb–Co ($r = 0.538$), Mn–Mg ($r = 0.535$), K–Cu ($r = 0.532$), Zn–Ni ($r = 0.511$), Fe–Co ($r = 0.505$) and Mn–K ($r = 0.502$). Consequently, number of strong relationships in the soil from urban farmland revealed mutual associations among the selected metals in the soil which may be attributed to the maturity of soil as noted in previous section.

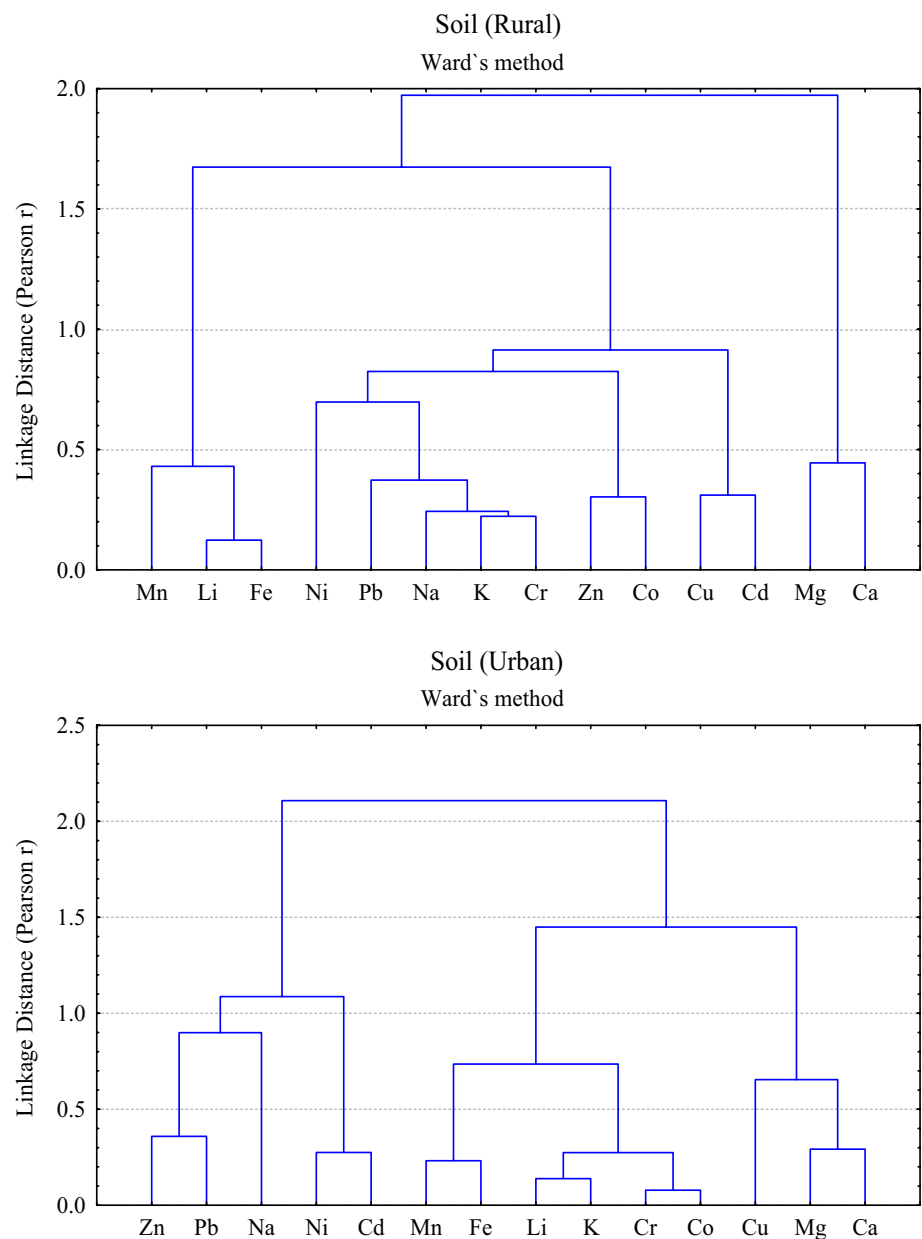
Cluster analysis of selected metals in soil

Another important aspect of the present study was multivariate apportionment of the metals using cluster analyses (CA). The dendrogram of the selected metals in the soil is shown in Fig. 2. In case of rural area, CA revealed five strong clusters: (1) Cr–K–Na–Pb; (2) Zn–Co; (3) Cu–Cd; (4) Fe–Li–Mn; and (5) Ca–Mg. Moreover, Ni showed very weak association with the second and third cluster. First three metal clusters were mostly anthropogenic in origin, predominantly contributed by fertilizers, agricultural sprays and atmospheric deposition, whereas, fourth cluster was considered lithogenic in origin. However, the last cluster may be contributed by natural sources. The counterpart cluster analysis for selected metals in the soil from urban farmland revealed six strong clusters: (1) Zn–Pb–Na; (2) Ni–Cd; (3) Mn–Fe; (4) Li–K; (5) Cr–Co; and (6) Ca–Mg–Cu. First cluster was mainly contributed by the anthropogenic activities, while second cluster was lithogenic in origin. However, third, fourth and fifth cluster was mostly attributed to the agricultural activities. Last cluster is believed to be multisource in origin. Multivariate cluster analysis thus exhibited divergent associations among the metals in soil samples from rural and urban farmlands. Nonetheless, major anthropogenic contributions were found for Pb, Cd, Zn, Ni, K, Cr and Na in both types of soil collected from rural and urban farmlands.

Enrichment factors of selected metals in soil

The foregoing discussion on the metal concentrations and apportionment indicated that anthropogenic sources resulted in the accumulation of the metals in soil samples; however, concentration alone does not provide information

Fig. 2 Cluster analysis of selected metals in soil from rural and urban farmlands



about the extent of modification in soil composition. Enrichment factors (EFs) of selected metals were considered to assess the anthropogenic intrusions of the selected metals in the soil. Figure 3a demonstrated the minimum, mean and maximum values of EFs for selected metals in the soil from both rural and urban farmland. In the case of rural soil, average values of EFs of selected metals were as follows; Ca (8.38), Cd (75.32), Co (6.34), Cr (2.29), Cu (2.77), Fe (1.00), K (0.62), Li (7.90), Mg (2.58), Mn (3.67), Na (0.12), Ni (1.67), Pb (40.75) and Zn (11.09), whereas, for urban soil mean values of EFs of the metals were as follows; Ca (13.29), Cd (81.19), Co (5.29), Cr (2.40), Cu (4.86), Fe (1.00), K (0.60), Li (8.58), Mg (3.15), Mn (4.21), Na (0.08), Ni (1.30), Pb (35.84) and

Zn (7.43). These values were interpreted as suggested by Sutherland (2000) as shown in Table S1. In both types of soil highest average values of EF were shown by Cd and Pb indicating very high to extreme enrichment of these metals which were mostly contributed by combustion processes, metal industries, fertilizers, waste incineration and fossil fuel burning. Both metals are also highly volatile, thus can also undergo long-range transport (Dragovic and Mihailovic 2009; Vega et al. 2009). Among rest of the metals, the rural as well as urban soil were found to be significantly enriched by Ca, Li, Co and Zn, whereas, moderately enriched by Cr, Cu, Mg and Mn, respectively. Nevertheless, minimum enrichment was shown by K, Na and Ni in both categories of soil. Consequently, EFs revealed

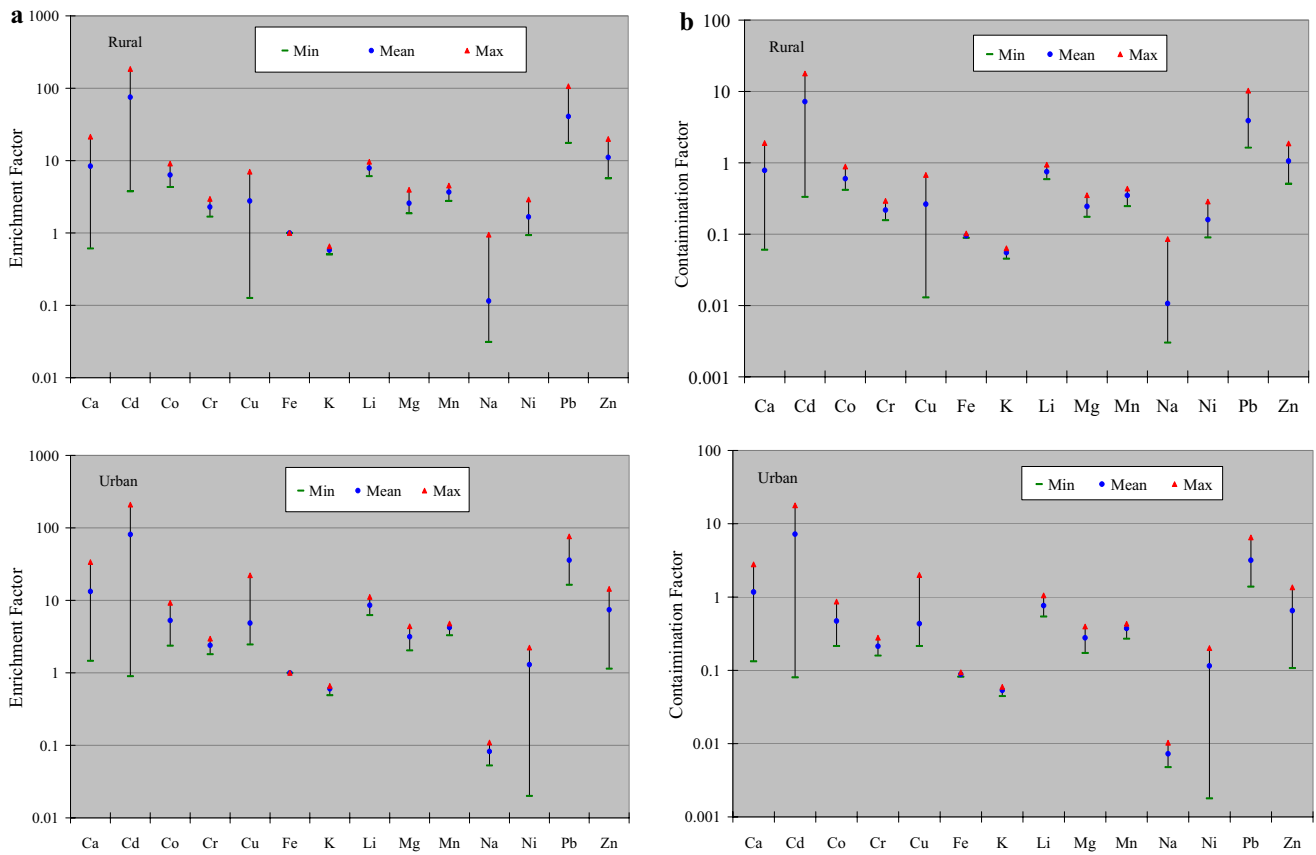


Fig. 3 **a** Enrichment factors of selected metals in soil from rural and urban farmlands. **b.** Contamination factors of selected metals in soil from rural and urban farmlands

significant anthropogenic enrichment of the metals in rural and urban soil.

Contamination factor of selected metals in soil

Figure 3b demonstrates the minimum, mean and maximum contamination factors (C_f) of the individual metals in soil samples from both rural and urban farmland. In case of rural soil, the average values of (C_f) were Ca (0.78), Cd (7.19), Co (0.60), Cr (0.22), Cu (0.26), Fe (0.09), K (0.05), Li (0.75), Mg (0.24), Mn (0.35), Na (0.01), Ni (0.19), Pb (3.90) and Zn (1.06). On the basis of average value of C_f of the metals, the soil samples were classified as least contaminated by Ca, Li, Co, Mn, Cu, Mg, Cr, Ni, Fe, K and Na; moderately contaminated by Zn and considerable to very highly contaminated by Pb and Cd (Table S1). Likewise, in case of urban soil (Fig. 3b) mean values of contamination factor of the metals were Ca (1.17), Cd (7.22), Co (0.47), Cr (0.21), Cu (0.43), Fe (0.09), K (0.05), Li (0.76), Mg (0.28), Mn (0.37), Na (0.01), Ni (0.11), Pb (3.18) and Zn (0.65). The urban soil samples were classified as least contaminated by Zn, Cu, Cr, Fe, Mg, K, Na, Co, Li, Ni and Mn; moderately contaminated by Ca and considerable to very highly contaminated by Pb

and Cd (Table S1). The C_f results were in good agreement with the enrichment factors discussed earlier.

Degree of contamination in soil

The assessment of the overall contamination of the soil from rural and urban farmlands was based on the degree of contamination (C_{deg}). Average degree of contamination by all metals in the rural soil was 15.67 which indicated moderate degree of contamination in the rural soil (Table S1). The cumulative degree of contamination is an old concept and in cases like the present study where one or two metals showed very high contamination, C_{deg} may lead to the misleading conclusions. Hence, the modified degree of contamination (mC_d) is mostly recommended in such cases. Average modified degree of contamination in case of rural soil was found to be 1.12, which manifested nil to very low degree of contamination in the rural farmland. Same results were obtained in case of the urban soil with the C_{deg} equals to 15.02 which also showed the moderate degree of contamination. However, modified degrees of contamination in case of urban soil were found to be 1.07 on the average basis. This also showed the nil to very low degree of contamination in the urban

farmlands (Table S1). Thus, overall nil to low degree of contamination was noted in both rural and urban soil samples.

Implications and recommendations

The present study showed elevated contributions of the trace metals and physicochemical parameters in agricultural soil from both rural and urban areas. Most of the metals exhibited random distribution and diverse correlations in the agricultural soil which was significantly influenced by anthropogenic intrusions. The soil samples were also moderately to severely contaminated and enriched by anthropogenic activities. Overall, the contamination of the soil indicated significant risks which could be addressed by proper management and implementation of sustainable agricultural practices along with reduced use of fertilizers and agricultural sprays. Moreover, systematic agricultural practices should be effectively implemented and organic farming should be promoted to reduce the anthropogenic impact of the pollutants on food chain.

Conclusions

In conclusion, the present study showed marked divergences in the distribution of selected metals in the rural and urban soil samples. Mean contents of Co, Na, Ni, Pb and Zn were significantly higher in rural soil, while Ca, Cu and Mg contents were considerably higher in urban soil ($p < 0.05$). Rest of the metals revealed almost comparable levels in rural and urban farmlands. Physicochemical parameters (pH, redox potential, EC, TDS, organic matter, moisture, density, porosity and total alkalinity), anion levels (sulphate, chloride, nitrate and nitrite) and soil texture exhibited considerable disparities in both types of soil. The correlation study revealed appreciably different mutual variations of selected metals in rural and urban soil samples. Soil texture mostly revealed sandy loam nature of the soil with higher clay and silt contents in urban soil and elevated sand contents in rural soil which was predominantly Ca–Mg–HCO₃ in composition. Significant anthropogenic contribution was shown by the cluster analysis while enrichment factors and contamination factors revealed severe to very high enrichment and moderate contamination of Cd and Pb in the soil.

Acknowledgements Financial and technical help provided by Quaid-i-Azam University, Islamabad, Pakistan to carry out this project is thankfully acknowledged.

Compliance with ethical standards

Conflict of interest There is no conflict of interests towards the publication of this research paper.

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