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Multivariate Statistical Approach to Identify Heavy Metal Sources in Urban Roadside Soils of Manisa, Turkey

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> The aim of this study was to investigate heavy metal pollution in roadside soils of Manisa, an important city located in the western part of Turkey. Soil samples were collected from roadsides of the city. Heavy metal concentrations were measured with Spectro-Xepos Benchtop X-Ray fluorescence spectrometer. Mean heavy metal concentrations (ppm) of the roadside soils in Manisa were as follows: Sb was 7.56, W 6.19, Ag 5.8, Mo 22.1, Co 28.37, V 94.0, Cu 136.57, As 26.95, Sn 19.99, Hg 1.71, Ni 64.67, Zn 126.19, Cd 4.05, Pb 47.97, Al 58730, Fe 34515, Mg 13841, Cl 350.76, Ti 3857.5, Cr 341.83 and Mn 704.3. Based on the chemical analyses, Ag, Cd, Fe, Al, Mo, Hg, Sb, Cr, W, Sn, Cu, As, Pb, Cl, Co and Zn concentrations of roadside soils in Manisa were higher than those of the earth's crust and North European soil. Results of the chemical analyses were evaluated with multivariate statistics with high accuracy rates. Heavy metal pollution of the roadside soils in Manisa can be attributed to highways and industrial and household wastes. It can be suggested that traffic on the busy highways along schools, hospitals and playgrounds for children should be diverted in the short term and such institutions should be built away from the highways and industrial areas in the long term.

> Key Words: Heavy metals, Urban roadside, Toxic, Multivariate statistics, Manisa.

INTRODUCTION

Heavy metal concentrations in terrestrial environments are increasing due to rapid urbanization and increased demands for metals in industrial units. Increasing heavy metal pollution has a negative effect on the natural geochemical circle of ecosystems and causes severe environmental problems. Anthropogenic emissions of heavy metals are continuously added to the

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nature¹⁻⁹. Manisa is a developed city with a population of over 200,000 which occupies only 2.125 km². Heavy metal concentrations in the city and its surrounding are thought to be increasing due to anthropogenic emissions.

Recently, there has been an increase in the studies in Turkey on heavy metal contents of soil¹⁰⁻¹⁸. However, to our knowledge, there have not been any studies on heavy metal contents of the soils in Manisa and its neighbouring areas. In this study, we made chemical analyses of heavy metal contents of the roadside soils in the city of Manisa and using multivariate statistics, we determined the possible sources of heavy metals.

EXPERIMENTAL

The province of Manisa is located in the Aegean region in the west of Turkey. It is situated between 27°08' and 29°05' east and between 38°04' and 39°58' north. The city of Manisa has a total area of 2.125 km² and constitutes 15.39 % of the total area of the province. 54 Point 3 % of the city was mountainous, 27.8 % plateau and 17.9 % plain. The city of Manisa is located between Mount Spil in the south and the Manisa Plain in the north.

Manisa has formations of all geological periods. The region has units that belong to the Paleozoic, Mesozoic, Tertiary and Quaternary times from the bottom towards the top. The city of Manisa is composed of Dededag, Anadag and Belkahve formations and Miocene, Detrital, Neocene, volcanic, travertine and alluvium units and slopes of debris. It is in the first degree earthquake zone. The biggest fault zone is confined to Mount Spil and the Manisa Plain. There have been a lot of land slides in various severities due to the steep slope of the fault zone in its south. Manisa has a high potential not only for agriculture but also for industry. In fact, there are 93 major industrial units, 1500 minor industrial units and 57 leather factories¹⁹.

In most of the province Manisa, it is very hot in summer and colder than that on the coastal cities in winter.

The temperature is 16.8 °C, dryness index is 27.5, relative humidity is 61 % and rainfall is 750.3 mm on a square meter on average per year. It is covered with snow for 1.6 d and with frost for 36 d and it is foggy for 19.9 d, sunny for 121 d and cloudy for 62.4 d a year. Air pollution caused by increasing population, developing industry and increasing number of vehicles cannot pass over the hot layer on Mount Spil in winter and suspend over the city and then forms an inversion layer. Among the common causes of air pollution are fuels used for heating, motor vehicles (there are about 41,000 motor vehicles according to the local authorities), industrial pollutants, overcrowding, topographic structure and city planning inappropriate for meteorological features¹⁹.

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Soil sampling and geochemical analysis: Soil sampling was performed in February, 2006. First, soil samples were collected systematically 1-3 m away from the roads and 5-10 cm in depth²¹. A total of 10 samples were obtained, each being 20 g (Fig. 1). All samples were collected with a hard, plastic shovel and kept in plastic bags. Second, the samples were dried in an autoclave at 105 °C for 24 h. Third, the dried samples were sifted through a plastic sieve with pores of 2 mm and pebbles were removed. Care was taken to avoid contamination while collecting, preserving, drying and shifting the samples. Fourth, the samples were homogenized in an agate mortar and changed into particles of about 2 mm in size. The agate mortar where samples were homogenized were washed with 6 mol L⁻¹ HNO₃ and rinsed with distilled water after homogenization of each sample. Fifth, each sample was shaped into double sided film tablets in a 32 mm diameter. Last, Spectro Xepos Bechtop X-Ray fluorescence spectrometer was used to for detection of As, Cd, Co, Cr, Cu, Fe, Mn, Mo, Ni, Pb, Sn, Ti and Zn. The detection system was a microprocessor controlled drift detector with Peltier cooling and energy resolution of FWHM < 170 eV, measured for the Mn K. line with an input count vote of 10.000 pulses. In these analyses, detection limits ranged from 0.01 to 0.1 wt % for major elements and 0.1 to 5 ppm for trace elements. Results were expressed in % or ppm.



S=1; X=0536505; Y=4274122; Z=64; the crossroad between Manisa High School and Primary School



S=3; X=0537258; Y=4274727; Z=66; near Ulupark playground for children



S=2; X=0537258; Y=4274727; Z=66; across the building of Tekel on dortyol



S=4; X=0537301, Y=4273844, Z=66; around Sultan Mosque

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S=5; X=0537152; Y=4273390; Z=129; up Ulu Mosque



S=7; X=0536825; Y=4274138; Z=75; near Kafe Sera playground for children



S=9;Z=0537863, Y=4274356, Z=77; near Osmanli bath



S=6; X=0536971; Y=4273242; Z=147; around Aglayan Kaya



S=8;Z=0537834; Y=4274226; Z=72; near the Government Office



S=10; X=0537936; Y=4274720; Z=79; at the crossroad of Maternal hospital

Fig. 1. Magnified views of the sampling locations and their coordinates

Data analyses with computer software: The results of chemical analyses were recorded in Excel. The recorded data were exposed simple statistics to determine the sources of heavy metals in the region. SPSS 10.5 for Windows and multivariate statistics were used for statistical analyses of raw and processed data. Data were exposed to multivariate statistical analyses to determine the elements with similar geochemical features (Frequency histograms, Coefficient correlation, cluster analysis based on Pearson's correlation coefficients, Hierarchical Cluster Analysis dendrogram, Model Summary and ANOVA).

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RESULTS AND DISCUSSION

Heavy metal concentrations: Oxides of the elements in the roadside soil samples are expressed in percentages and SiO₂, Al₂O₃, Fe₂O₃, CaO, K₂O, Na₂O, MgO, TiO₂, SO₃, LOI (loss on ignition) and heavy metal (Sb, W, Ag, Mo, Co, V, Cu, As, Sn, Hg, Ni, Zn, Cd,, Pb, Al, Fe, Mg, Cl, Ti, Cr and Mn) contents of the soil samples are expressed in ppm in Table-1. Based on the results of chemical analyses, median values of the heavy metal contents of the roadside soils in Manisa exceeded those reported from northern Europe²² and those of the earth's crust²³ (Table-2). There was a considerable increase in some metals. In fact, based on the degree of increases, they can be listed as in the following: Ag, Cd, Mo, Fe, Al, Hg, Sb, Cr, W, Sn, Cu, As, Pb, Cl, Co and Zn.

Multivariate statistics: The frequency histogram²³⁻²⁵ of the heavy metal (Ag, Cd, Mo, Hg, Sb, Cr, W, Sn, Cu, As, Pb, Cl, Co and Zn) concentrations originating from anthropogenic sources is shown in the form of box and whisker plot in Fig. 2. Accordingly, Sb, W, Cl, Sn, Pb, Cu and Cr may have travelled short distances, Co, As, Ag, Hg and Zn travelled medium distances and Cd and Mo long distances. The former group of metals may have resulted from highways and the latter two groups of metals may have originated from industrial and household wastes. As a result, the sources of these metals turned out to be the urban areas.

The Ag concentration in the study area was 5.8 ppm and was expected to range between 2.9 and 16.1 ppm (Table-1). The obtained Ag concentration was considerably higher than its concentration of 0.1ppm in the earth's crust. In fact, it was 58 times higher than that of the earth's crust (Table-2). Mean heavy metal concentrations (ppm) of the roadside soils in Manisa were as follows: Sb was 7.56 (5-13.1), W 6.19 (5.7-7.2), Ag 5.8, Mo 22.1 (21-23), Co 28.37 (22.1-45.2), V 94, Cu 136.57 (23.8-945.6), As 26.95 (17.9-37.3), Sn 19.99 (6.5-43.4), Hg 1.71 (1.3-3), Ni 64.67, Zn 126.19 (67.1-293.3), Cd 4.05 (3.5-4.3), Pb 47.97 (22-107.3), A1 58730 (48250-67510), Fe 34515 (26410-43680), Mg 13841, Cl 350.76 (175.8-761.1), Ti 3857.5, Cr 341.83 (108.2-652) and Mn 704.3. (Table-1). Cd, Mo, Fe, Al, Hg, Sb, Cr, W, Sn, Cu, As, Pb, Cl, Co and Zn concentrations were higher than their concentrations in the earth's crust (they are 0.3, 2, 4.7×10^4 , 9.2 $\times 10^4$, 0.3, 1.5, 100, 1.8, 6, 50, 10, 20, 170, 20 and 90 ppm in the earth's crust). In fact, Cd, Mo, Fe, Al, Hg, Sb, Cr, W, Sn, Cu, As, Pb, Cl, Co and Zn were 13.5, 11.05, 5.7, 5.04, 3.41, 3.4, 3.33, 2.73, 2.69, 2.39, 2.06, 1.41 and 1.4 times higher in the roadside soils of Manisa than in the earth's crust respectively (Table-2). Similarly, Cd, Mo, Fe, Al, Hg, Sb, Cr, W, Sn, Cu, As, Pb, Cl, Co and Zn concentrations were also higher in the roadside soils of Manisa than in north Europe soils²².

		HEAV	Y MET	DAL CC	NCEN	TRATIC	O SNC	TA THE F	BLE-1 ROADS	IDE S	OILS O	F THE	CITY (JF MAP	VISA (pp	m)	
Coarse					Dxides ((wt %) a	and LOI										
Samples	SiO_2	Al_2O_3	$\mathrm{Fe}_{2}\mathrm{O}_{3}$	CaO	K_2O	Na_2O	MgO	TiO_2	SO_3	LOI	Total						
1	51.01	11.40	4.65	11.53	3.24	2.70	2.96	0.61	0.99	10.03	99.10						
2	36.76	28.16	3.78	13.87	2.93	1.46	2.14	0.53	0.19	9.52	99.32						
3	51.69	10.72	4.21	10.55	3.80	2.90	2.99	0.55	0.12	10.83	98.34						
4	51.94	11.38	4.38	11.93	2.86	3.78	2.40	0.58	0.32	10.30	99.87						
5	53.80	10.48	6.25	11.11	3.45	0.56	2.99	0.84	0.24	10.25	99.97						
9	49.16	11.95	6.00	11.80	3.67	3.42	2.25	0.73	0.20	9.86	99.03						
7	52.61	12.26	4.93	9.58	2.29	3.81	2.40	0.63	0.13	10.72	99.35						
8	52.49	10.50	4.50	12.87	3.17	2.93	2.42	0.61	0.38	10.11	99.98						
6	53.36	12.57	6.02	8.90	3.49	2.46	1.87	0.80	0.96	8.72	99.13						
10	54.86	9.78	4.66	13.17	2.85	3.37	1.18	0.57	0.33	9.04	99.80						
Mean	50.77	12.92	4.94	11.53	3.17	2.74	2.36	0.64	0.38	9.94	99.39						
Coarse								Trac	se elem	ents (pj	(mq						
Samples	\mathbf{Sb}	M	Ag	Mo	Co	Λ	Cu	\mathbf{As}	Sn	Hg	Ni	Zn	Cd	Pb	Al	Fe	Mg
1	5.40	5.90	3.40	22.00	24.00	109.00	23.80	37.30	6.50	1.80	92.20	68.40	4.20	22.00	60360	32490	17830
2	13.10	6.50	7.40	23.00	22.10	101.00	945.60	33.30	12.70	1.40	62.90	96.50	4.00	29.30	48250	26410	12890
3	5.20	5.80	5.20	21.00	25.10	94.00	39.30	27.80	6.60	1.70	76.40	67.10	4.00	25.40	56750	29420	16810
4	6.40	6.00	7.20	22.00	28.80	97.00	30.60	28.60	20.20	1.40	61.30	86.20	3.50	45.80	60220	30620	14480
5	5.40	5.70	4.40	21.00	45.20	103.00	51.30	20.30	43.40	1.40	36.10	117.10	4.20	62.30	55470	43680	11190
9	5.00	6.90	4.70	23.00	28.00	41.00	84.40	17.90	36.80	3.00	57.20	214.60	4.20	107.30	67510	41940	13550
7	10.60	5.70	3.50	21.00	25.00	129.00	27.10	30.40	7.70	1.70	67.60	71.60	4.00	24.90	64880	34450	14450
8	8.60	7.20	16.10	23.00	24.00	34.00	46.90	28.40	22.60	1.90	77.50	293.30	4.00	50.70	55560	31500	14570
6	5.00	5.80	2.90	23.00	37.60	133.00	59.20	18.80	26.90	1.50	54.00	96.50	4.30	42.50	66540	42090	11290
10	10.90	6.40	3.20	22.00	23.90	99.00	57.50	26.70	16.50	1.30	61.50	150.60	4.10	69.50	51760	32550	11350
Mean	7.56	6.19	5.80	22.10	28.37	94.00	136.57	26.95	19.99	1.71	64.67	126.19	4.05	47.97	58730	34515	13841

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Fig. 2. Frequency Histograms of Ag, Cd, Mo, Hg, Sb, Cr, W, Sn, Cu, As, Pb, Cl, Co and Zn (anthropogenic elements) in the roadside soils of Manisa

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COMPARISONS BETWEEN ELEMENT CONCENTRATIONS IN URBAN ROADSIDE SOILS OF NORTH EUROPE [Ref. 22], CRUST SOIL/SHEYL [Ref. 23] AND SOIL SAMPLES FROM MANISA (ppm)

Elements	North Europe soil (A)	Soil (B)	Manisa soil (mean) (C)	Variation of mean conc. in soil (fold) (C/B)	Variation of mean conc. in soil (fold) (C/A)
Fe	1.70×10^{4}	4.7×10^{4}	34.515	7.3	20.3
Al	4.83×10^{4}	9.2×10^{4}	58.730	6.3	12.16
Mg	0.34×10^{4}	1.4×10^{4}	13.841	9.9	40.7
Cu	10	50	136.57	2.73	13.65
Pb	17	20	47.97	2.39	2.8
Zn	43	90	126.19	1.4	2.9
Cd	_	0.3	4.05	13.5	_
Ni	10	80	64.67	-0.8	6.4
Cr	32	100	341.83	3.41	10.68
Co	5.3	20	28.37	1.41	5.35
Mn	426	850	704.3	-0.82	1.65
Ti	2601	4500	3857.5	-0.85	1.48
Sn	_	6	19.99	3.33	_
Mo	—	2	22.1	11.05	-
As	4	10	26.95	2.69	6.73
Sb	—	1.5	7.56	5.04	-
W	_	1.8	6.19	3.4	_
Ag		0.1	5.8	58	-
V	37	130	94	-0.72	2.54
Hg	_	0.3	1.71	5.7	_
Cl	_	170	350.76	2.06	_

There was a strong positive correlation between As and Ni, between Sn and Pb, Fe, Ti and Mn, between Ni and Mg, between Zn and Cl, between Fe and Ti and Mn and between Ti and Mn. There was also a moderate, positive correlation between Sb and As, between Ag and Zn and Cl, between As and Mg, between Cd and F and between Pb and Mn (Table-3). There was a strong negative correlation between V and Zn and Cl, between As and Sn, Pb, Fe, Ti and Mn and between Hg and Pb. There was also a moderate, negative correlation between Sb and Al, Fe and Ti, between Ag and V, between Co and Ni, between Sn and Mg and between Ni and Fe, Ti and Mn (Table-3). The metals with a strong positive correlation with each other were thought to result from the same sources of pollution, but those with a strong negative correlation with each other were thought to come from different sources of pollution.

	Mn																					00.	
	Cr																				1.00).548 1	
	Ti																			1.00).382	905‡ (V= 10
	CI																		1.00	0.228	0.373 (0.374 .	iled). N
IANISA	Mg																	1.00	-0.022	-0.519 (0.005 (-0.592 (nt (2-ta
S OF N	Fe																1.00	-0.499	0.258	.965‡ .	0.442	.959‡ .	ignifica
E SOIL	Al															1.00	0.595	0.127	0.258	0.496	0.103	0.476	01 are s
ADSID	Pb														1.00	0.176	0.541	-0.568	0.587	0.411	0.57	.705†	el of 0.
HE RO,	Cd													1.00	0.4	0.333	.638†	-0.336	-0.168	0.574	0.361	0.565	the lev
LL NI S.	Zn												1.00	-0.053	0.622	-0.044	0.154	-0.198	:849	0.103	0.199	0.29	ttions at
3-3 EMENT	Ni											1.00	0.072	-0.429	-0.348	-0.29	684†	:792‡	0.015	741†	0.046	665†	:Correla
TABLH HE ELI	Hg										1.00	0.271	-0.255	-0.457	831‡	-0.307	-0.461	0.364	-0.358	-0.313	674†	-0.627	ailed); ‡
/EEN T	Sn									1.00	-0.569	-0.618	0.454	0.529	.766‡	0.201	‡ <i>T</i> 97‡	642†	0.509	.815‡	0.595	:859	ant (2-t
BETW	As								1.00	828‡	0.603	:766‡	-0.321	-0.441	775‡	-0.421	819‡	.647†	-0.433	765‡	-0.411	873‡	signific
SNOITA	Cu							1.00	0.303	-0.157	-0.082	0.014	-0.105	-0.382	-0.145	-0.568	-0.441	-0.183	-0.12	-0.336	-0.074	-0.265).05 are
RREL	Λ						1.00	0.045	0.184	-0.339	0.367	-0.349	870‡	0.124	-0.574	0.073	0.031	-0.131	861‡	0.097	-0.472	-0.128	svel of (
INT CO	Co					1.00	0.442	0.069	-0.434	0.401	0.016	-669	-0.363	0.327	0.072	-0.244	0.282	-0.585	-0.371	0.404	0.031	0.245	at the le
FFICIE	Mo				1.00	-0.216	-0.433	0.393	-0.142	0.212	-0.323	-0.194	0.545	0.112	0.309	0.041	-0.003	-0.222	0.456	0.003	-0.097	0.116	lations
COE	Ag			1.00	0.392	-0.289	691	0.139	0.168	0.048	0.283	0.318	.703†	-0.517	0.045	-0.327	-0.374	0.149	:665†	-0.282	-0.13	-0.303	†Corre
	M		1.00	0.085	0.243	-0.336 -	-0.282 .	0.189	-0.103	0.054	-0.067	0.055	0.431	-0.201	0.151	-0.134 -	0.163 .	-0.11	0.273	0.118	0.115	0.297	lations;
	Sb	1.00	0.056	0.268	-0.155	-0.157 -	0.116 -	0.508	.652† -	-0.505	0.409 -	0.445	-0.057	-0.58 -	-0.337	732† -	705†	-0.018	-0.31	659	-0.435	-0.626	= Corre
	CR	\mathbf{Sb}	M	Ag	- oM	- Co	>	Cu	\mathbf{As}	Sn -	Hg	ïŻ	- uZ	Cd	- dq	- I	Fe .	- gM	Ü	Ë	C	- Mn	ß

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The dendrogram²⁶⁻²⁸ based on the Pearson's correlation coefficients, which seemed to support correlations between the elements, showed three distinct groups of elements. Fe and Ti formed a distinct group (Group I) and Mn, Sn, Pb, Cr, Cd and Al joined this group. Zn and Cl also formed a distinct group (Group II) and Ag, Mo and W joined this group. In addition, Ni and Mg formed a distinct group (Group III) and As, Hg and Sb-Cu joined this group. Co and V joined Groups II and III (Fig. 3). Based on these groups, heavy metal pollution in Manisa could be ascribed to three different sources. The results of correlation analyses were consistent with those of correlation coefficients. In fact, the heavy metals strongly correlated with each other (As and Ni; Sn and Pb, Fe, Ti and Mn; Ni and Mg; Zn and Cl; Fe and Ti and Mn; Ti and Mn) had also a high correlation coefficiency in the dendrogram.



Fig. 3. Classification of the elements based on Pearson's correlation coefficients using cluster analyses

Hierarchical cluster analysis²⁹⁻³¹ was made to determine similarities between the samples from ten different sampling locations and the analysis showed that the number of samples was sufficient to determine the similarities (Fig. 4). In fact, there were some similarities between the samples collected from different locations. In general, the sampling locations could be classified into three groups. Locations 1, 7 and 4 had some similarities and formed a group and locations 9 and 3 observed in this group. In addition, locations 2 and 10 were similar and 6 and 8 were similar. They formed two

different groups at first. Then, they formed a single group and location 5 observed in this group. Sampling locations similar to each other were thought to be polluted by similar pollutants.



Fig. 4. Hierarchical cluster analysis dendrogram of urban roadside soil samples of Manisa

Regression analyses of Pb concentrations were made (Table-4) and the explanatory power of the regression formula for Model Summary was $R^2 = 100 \%$ and gave considerable accuracy rates. According to the results of ANOVA, nine variables (Mn, Sb, Na, Sn, Cr, S, V, Ag, K, Cd, Ni, Cl, Cu, Pb, Ca, W, As, Mo, Ti, Co, Mg, Si, Zn, Al, Hg) explained changes in Pb concentrations at a high level.

MODEL 3			IAD	LES OF KEOKI	2021014	DAIA
Model summary	R	\mathbf{R}^2		Adjusted R ²	Stan of th	dard error e estimate
1	1,000(a)	1,000		1,000		
Model ANOVA(b)		Sum of Squares	df	Mean Square	F	Sig.
1	Regression	6295.661	9	699.518		.(a)
	Residual	0.000	0			
	Total	6295.661	9			

TABLE-4 MODEL SUMMARY AND ANOVA TABLES OF REGRESSION DATA

(a) Predictors: (Constant). Mn, W, Cu, Cd, Al, Sb, Ag, Mo, As.

(b) Dependent variable: Pb.

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In the light of the geological data and the results of the chemical analyses, there was no geogenic pollution in the city of the Manisa. However, there was an increase in heavy metal pollution due to highways^{9,15} with 41,000 vehicles, industrial facilities such as petrol stations and household wastes. Higher concentrations of heavy metals in industrial facilities³ such as petrol stations without waste disposal systems can be explained by the fact that household wastes contain smaller amounts of heavy metals.

Conclusion

Using multivariate statistics, we determined the severity of heavy metal pollution in the urban roadside soils of Manisa. In fact, Cd, Mo, Fe, Al, Hg, Sb, Cr, W, Sn, Cu, As, Pb, Cl, Co and Zn concentrations were quite high in the urban roadside soils of Manisa. They exceeded the concentrations in the earth's crust and northern Europe soil and were thought to have toxic effects.

Based on their similarities, ten locations where samples were collected were classified into three groups: Group I included locations 1, 7, 4, 9 and 3. Group II included locations 2, 10, 6 and 8. Group III included location 5. The locations with similar features were thought to be polluted by the same pollution sources.

Considering the results of the correlation analyses, heavy metals with strong positive correlations were thought to result from the same sources and those with strong negative correlations were thought to originate from different sources. Regression analyses of Pb concentrations revealed that Model Sumary had a high explanatory power and that ANOVA explained changes in Pb concentrations with nine variables at a high level.

The higher metal concentrations in three different locations were thought to come from anthropogenic sources: highways and industrial and household wastes.

As a short-term solution to the problem, traffic on the highways along residential places such as schools and hospitals should be diverted and in the long term the residential places should be moved away from the highways and industrial facilities.

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