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Statistical source identification of metals in groundwater exposed to industrial contamination

Saadia R. Tariq · Munir H. Shah · N. Shaheen ·
M. Jaffar · A. Khalique

Abstract Levels of selected metals Na, Ca, Mg, K, Fe, Mn, Cr, Co, Ni, Cd, Pb and Mn were estimated by flame atomic absorption spectrophotometry in groundwater samples from Kasur, a significant industrial city of Pakistan. Salient mean concentration levels were recorded for: Na (211 mg/l), Ca (187 mg/l), Mg (122 mg/l), K (87.7 mg/l), Fe (2.57 mg/l) and Cr (2.12 mg/l). Overall, the decreasing metal concentration order was: Na > Ca > Mg > K > Fe > Cr > Zn > Co > Pb > Mn > Ni > Cd. Significantly positive correlations were found between Na–Cr ($r=0.553$), Na–Mn ($r=0.543$), Mg–Fe ($r=0.519$), Mg–Cr ($r=0.535$), Pb–K ($r=0.506$) and Pb–Ni ($r=0.611$). Principal Component Analysis and Cluster Analysis identified tannery effluents as the main source of metal contamination of the groundwater. The present metal data showed that Cr, Pb and Fe levels were several times higher than those recommended for water quality by WHO, US-EPA, EU and Japan. The elevated levels of Cr, recorded as 21–42 fold higher compared with the recommended quality values, were believed to originate from the tanning industry of Kasur.

Keywords Trace metals · Groundwater · Cr · Principal component analysis · Cluster analysis · Correlation · Pakistan

Introduction

Being the largest reserve of drinkable water for human population, groundwater has always been of major importance to human civilization. Compared with other kinds of water, groundwater is normally preferred because it tends to be less contaminated directly by wastes and organisms. However, in the wake of recent industrialization and fast urbanization the quality of groundwater has become an increasing concern due to contamination by various toxic substances (Mance 1987; Aiyesanmi et al. 2004; Amajor 1986; Ezeigbo 1989; Calderon 2000; Jha et al. 1990; Ramesh et al. 2000). On the global scale, industrial, agricultural and municipal activities have all resulted in groundwater pollution by a variety of contaminants (Oguzie et al. 2002; Ipinmoroti 1993; Lee et al. 2001; Vidal et al. 2000; Moon et al. 1994; Speir et al. 2003; Razo et al. 2004). Specifically, trace metal contamination of water has emerged as a serious health issue highlighted in published work (Watt et al. 2000; Fernandez et al. 1994; Nriagu and Pacyna 1988). It is understood through documented evidence that the primary pathways of toxic metal accumulation in humans are through the ingestion of

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contaminated water and food (Barman et al. 2000; Santos et al. 2004). Health related studies have shown that excessive intake of toxic trace metals results in neurological and cardiovascular diseases, as well as renal dysfunction (Vallee and Ulmer 1972; Jarup 2002; Packham 1996). Therefore, the initial step towards assessment of health risks posed by metal pollution requires regular monitoring of water quality in terms of trace metal distribution and source identification.

In Pakistan, like most other developing countries, the industrialization and urbanization have outpaced with environmental safety, resulting in deleterious effects of industrial waste disposal in water bodies or open land. The groundwater in industrial areas across the country has undergone severe contamination by industrial wastes, effluents and emissions which are discharged indiscriminately without any regulatory system (Japan International Cooperation Agency (JICA)-EPA Joint Report 2000; Samina et al. 2004). The present study stems from the above concerns, with the primary focus on the current status of distribution of some selected toxic metals in the groundwater from Kasur, and then to identify probable sources of the metal contaminants in the water in an area well recognized to be under industrial contamination stress currently. Kasur is a well-known industrial city of Pakistan, housing a large number of tanneries. Over the years these tanneries have been discharging their solid wastes, effluents and emissions in nearby open lands and the atmosphere thus posing an ultimate threat to the quality of groundwater in the area. For the present study, groundwater sampling was carried out from Kasur and the following metals were analysed in the samples by the AAS method: Na, Ca, K, Mg, Fe, Mn, Cr, Co, Cd, Ni, Pb and Zn. The estimated metal levels were compared with the permissible safe levels laid down by world health authorities for acceptable water quality. Multivariate statistical techniques (Lin et al. 2002; Facchinelli et al. 2001; Nolte 1988; Tahri et al. 2005; Yeung 1999; Lin et al. 2004; Sandhu et al. 1976) were adopted to identify possible sources of metal pollutants in the water. For this purpose, the well founded techniques of Pearson correlation analysis, Principal Component Analysis (PCA), and Cluster Analysis (CA) were jointly used, the first affording a direct measure of interdependence of the set of variables under investigation while the latter two provides the

visual grouping of the data to help understand the interrelated metal clusters produced (Hopke 1992).

It is anticipated that the present study, the first of its kind in the area, will provide a basis for metal burden evaluation in the groundwater, as well as help trace the origin of the metals. Accordingly, the study would be helpful to evolve practical measures related to future water pollution abatement programmes, motivating further studies on metal trend analysis based on source identification in related media elsewhere.

Experimental

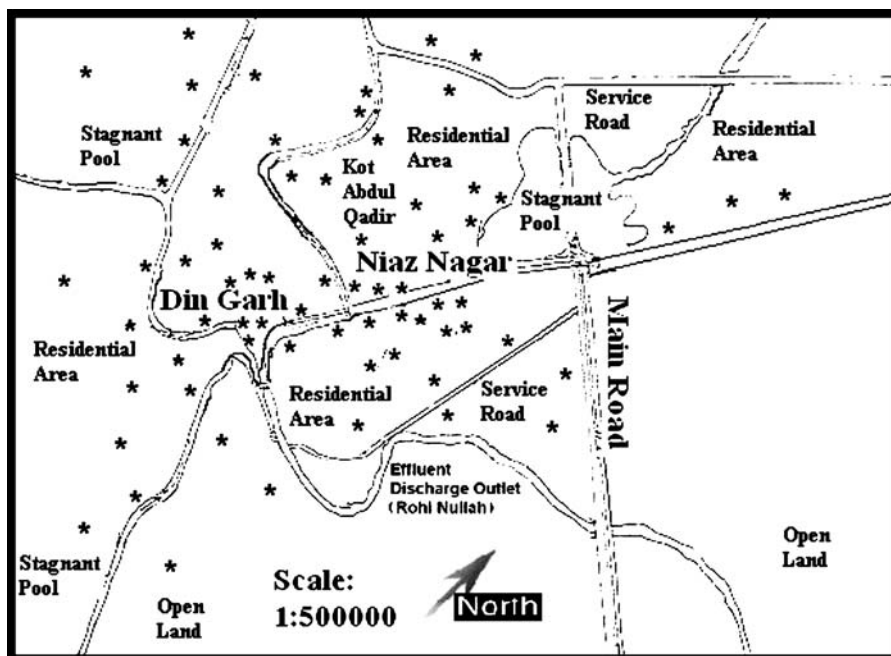
Site description

Kasur, an industrial city in the central Punjab Province, Pakistan, with a population of about 0.25 million, spans over an area of 300 km² most of which consists of plain fields irrigated by water from the Indus River and groundwater drawn by tube-wells. Wheat, rice, sugarcane and cotton are rich cash-crops of the area, feeding more than 5 million people. The city has a traditional industrial setup and houses a large number of small and significant industrial units of which the tanneries are the most dominant (Fig. 1). Other important industries in the city include textile, sugar, glazed pottery, embroidery and woodworks (Federal Bureau of Statistics 2003).

Analytical methodology

The groundwater samples from wells were collected in triplicate in pre-washed, strong polythene bottles (1 l capacity) during May–November, 2003, from several residential areas of Kasur (Fig. 1). The standard sampling procedure (American Public Health Association (APHA), American Water Works Association (AWWA) and Water Pollution Control Federation (WPCF) 1989) was adopted to this effect. No stabilizers or buffers were added to the samples, which were analysed immediately when received in the lab. As a matter of routine the blanks were checked for given metals prior to analysis. A total of 68 wellwater samples were analysed for Na, K, Ca, Mg, Zn, Fe, Mn, Pb, Cr, Cd, Ni, and Co under optimum analytical conditions using a Shimadzu Atomic Absorption Spectrophotometer (Model AA-670) equipped with automatic background compensation.

Fig. 1 Location of sampling points (asterisk) in the study area of Kasur



All reagents used were of AAS grade (certified purity > 99.9%) procured from E. Merck, Germany or BDH, UK. Redistilled water was used both for the preparation of standards and the dilution of samples. Triplicate sub-samples of each sample were aspirated separately to compute average metal concentrations in a given water sample. A parallel comparative check on the accuracy of quantified results was made through the analysis of standard reference material (NIST, SRM-1573a). Inter-laboratory comparison of the data was also periodically exercised at the laboratories of National Institute of Health (NIH), Islamabad. For routine comparative check, two parallel sub-samples of a given sample or a standard, processed under similar analytical conditions, were subjected to comparative analysis at the two labora-

tories. The individual sets of data were found to agree within $\pm 1.5\%$. STATISTICA software was used to conduct the statistical analyses (StatSoft, Inc. 1999).

Results and discussion

Table 1 provides mean metal concentrations in groundwater samples, along with other relevant statistical distribution parameters. High mean concentrations were found for Na, Ca, Mg and K, while both Fe and Cr remained close to each other. Lower concentrations were noted for Cd, Mn, Ni, Fe, Pb and Co at sub-ppm level. Average Na concentration was 211 mg/l, followed by Ca, Mg and K at 187, 122 and 87.7 mg/l, respectively. The Cr distribution

Table 1 Basic statistical parameters* for the distribution of selected metals in groundwater samples from Kasur ($n=68$)

	Na	Ca	K	Mg	Fe	Mn	Cr	Co	Cd	Ni	Pb	Zn
Minimum	58.0	42.0	15.0	51.0	0.02	0.01	0.05	0.01	0.001	0.001	0.003	0.01
Maximum	326	292	94.1	182	11.8	0.17	9.80	0.73	0.027	0.24	0.26	1.08
Mean	211	187	87.7	122	2.57	0.04	2.12	0.17	0.007	0.08	0.11	0.14
Median	148	81.7	58.6	96.0	1.21	0.03	0.71	0.13	0.006	0.04	0.11	0.14
SD	190	52.9	22.2	48.0	3.02	0.03	2.87	0.16	0.005	0.08	0.06	0.25
SE	83.7	6.42	2.70	20.7	0.37	0.04	0.35	0.02	0.001	0.01	0.01	0.03
Kurtosis	6.13	4.65	2.21	0.47	0.01	5.17	1.98	2.26	2.34	-0.92	-0.83	2.18
Skewness	2.33	1.58	1.64	0.84	1.01	2.13	1.76	1.48	1.38	0.94	0.09	0.78

*in mg/l where applicable

ranged between 0.05 and 9.80 mg/l, with a mean of 2.12 mg/l, thereby indicating a gross pollution of the groundwater with that metal ion. The decreasing trend of average metal levels was as follows: Na > Ca > Mg > K > Fe > Cr > Zn > Co > Pb > Mn > Ni > Cd.

A comparison of the listed mean and median values evidenced an almost randomised distribution of Pb, Zn, Cd and Mn, indicating large dispersion around the mean metal levels. The substantial differences in the symmetry parameters in the case of Ni, Co, Cr and Fe indicated a non-normal distribution, thus supporting a possibility of random infiltration of the metals from some anthropogenic source. Large standard deviations in the case of Na, Ca, Mg, K, Fe and Cr levels revealed their randomly fluctuating concentration levels in the groundwater.

Before evolving a judgement on the observed distribution of metal levels regarding their probable origin, the metal data was first examined on the basis of linear correlation between metal pairs in terms of significant positive correlation coefficient, $r > 0.369$, at $p < 0.001$. Strong positive correlations were observed for Pb–Ni, Na–Cr, Na–Mn, Mg–Cr, Mg–Fe and K–Pb pairs (Table 2), indicating the existence of a common source/origin of these metals in the groundwater under investigation. This provided the primary evidence for a mutual concentration dependence of the metals in the aquatic system under observation. As individual metal concentrations were higher several folds in comparison to the background levels in uncontaminated groundwater sampled from far-off locations, it was inferred that industrial wastes were

affecting the water-table in the area. The recorded higher Cr levels were a good pointer towards tanning industry effluents since chromium compounds are used in large bulk in the industry. Currently as many as 200 tanning units are operative in the area from which water samples were taken for analysis.

Further confirmation of this hypothesis was secured through multivariate methods of statistical analysis (Hair et al. 1988). To this effect, two multivariate techniques were applied: Principal Component Analysis (PCA) and Cluster Analysis (CA). The PCA has emerged as a useful tool for better understanding the relationships among the variables (e.g., metal concentrations in the present study) and for revealing groups (or clusters) that are mutually correlated within a data body. This procedure reduces overall dimensionality of the linearly correlated data by using a smaller number of new independent variables, called principal components (PC), each of which is a linear combination of originally correlated variables. On the other hand, Cluster Analysis (CA) exclusively classifies a set of observations into two or more unknown groups based on combination of internal variables. Therefore, the purpose of CA is to discover a system of organized observations where a number of groups/variables share properties in common, and it is cognitively easier to predict mutual properties based on an overall group membership (Everitt 1993; Jolliffe 1986). This helps define source profiles of variables, such as metal concentrations, and their interpretation in terms of possible sources (Jobson 1991).

Table 2 Metal-to-metal correlation coefficient matrix for selected metals in groundwater samples ($n=68$)

	Na	Ca	K	Mg	Fe	Mn	Cr	Co	Cd	Ni	Pb	Zn
Na	1.000											
Ca	0.464	1.000										
K	0.469	0.256	1.000									
Mg	0.267	0.308	0.374	1.000								
Fe	0.120	-0.065	0.075	0.519	1.000							
Mn	0.543	0.315	0.440	0.228	-0.076	1.000						
Cr	0.553	0.491	0.379	0.535	-0.454	0.255	1.000					
Co	0.162	-0.054	0.256	0.341	0.268	0.022	-0.409	1.000				
Cd	0.316	-0.094	0.064	0.207	0.345	-0.032	-0.260	0.422	1.000			
Ni	-0.005	-0.073	0.106	-0.341	-0.333	0.257	0.175	-0.168	-0.417	1.000		
Pb	0.299	-0.238	0.506	0.320	0.205	0.386	-0.453	0.411	0.058	0.611	1.000	
Zn	0.015	0.175	-0.137	-0.019	0.429	0.189	-0.198	0.108	0.166	-0.051	0.010	1.000

Bold values are significant at $p < 0.001$

Table 3 Varimax normalized rotated principal component loadings of selected metals in groundwater samples ($n=68$)

	PC 1	PC 2	PC 3	PC 4
Na	0.714	-0.166	0.375	0.121
Ca	0.787	0.171	-0.234	0.098
K	0.762	0.191	0.216	0.317
Mg	0.801	0.257	-0.098	-0.042
Fe	-0.226	0.496	-0.029	0.535
Mn	0.827	0.126	-0.038	-0.111
Cr	0.722	0.199	-0.278	0.292
Co	0.471	0.672	0.317	0.038
Cd	0.075	0.821	0.204	0.067
Ni	-0.221	0.339	0.749	0.115
Pb	0.377	0.174	0.783	0.21
Zn	-0.137	-0.234	0.357	0.665
Eigen value	4.398	3.046	2.324	2.305
% total variance	39.15	20.37	16.41	15.13
Cumul. Eigen value	4.938	7.984	10.31	12.61
Cumul. % variance	39.15	59.52	75.93	91.06

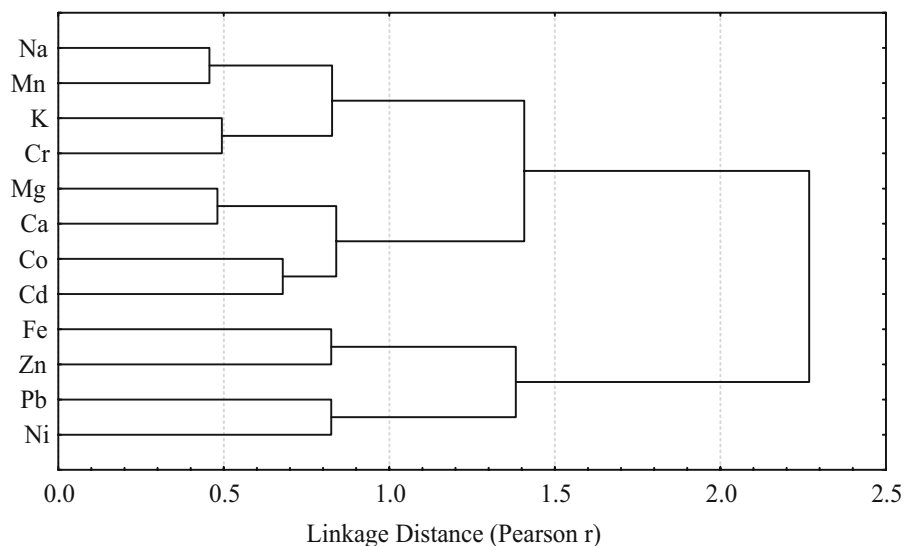
Accordingly, using Varimax normalized rotation, the Principal Component Analysis was conducted for source identification. The rotated Principal Component Loadings are given in Table 3. Four Principal Components (eigen values >1) emerged with more than 91% of cumulative variance. The first PC with 39.15% variance showed higher loadings for Na, Ca, K, Mg, Mn and Cr, with significant contributions from Co and Pb, conceivably originating from the tanning industrial units, since compounds containing these metals are extensively used in various chemical

Table 4 Comparison of average metal levels (mg/l) with some international water quality standards

	Present study	WHO	US-EPA	EU	Japan
Na	211	200	20	150	200
Ca	187	—	—	—	—
K	87.7	—	—	12	—
Mg	122	—	—	—	—
Fe	2.57	0.3	0.3	0.2	0.3
Mn	0.04	0.1–0.5	0.05	0.05	0.01–0.05
Cr	2.12	0.05	0.1	0.05	0.05
Co	0.17	—	—	—	—
Cd	0.007	0.003	0.005	0.005	0.01
Ni	0.08	0.02	0.1	0.05	0.01
Pb	0.11	0.01	0.015	0.05	0.05
Zn	0.14	3	5	0.1	—

processes during the tanning of leather. The second PC, with 20.37% of total variance, had higher loadings for Co and Cd, along with significant contribution from Fe, Ni and Mg. It could be conceived to mainly originate from industrial effluents and influent waste discharged from other industries in the area. The third PC had higher loadings for Ni and Pb at 16.41% of total variance, while the fourth PC at 15.13% of total variance showed higher loadings for Fe and Zn. Both of these components showed the contribution of the metals from the soil/rock based strata structure in the area. The corresponding cluster analysis dendrogram (Fig. 2) showed good agreement with PCA. It was

Fig. 2 Dendrogram showing the clustering behaviour of selected metals in groundwater samples



thus confirmed that the metals with common PC and strong mutual correlations formed primary clusters.

The metal levels in groundwater pertaining to the present study were compared with water quality standards laid down and recommended by international agencies such as WHO, US-EPA, EU and Japan, shown in Table 4 (Radojevic and Bashkin 1999). The levels of Na, Mn and Ni for the present study were comparable with the corresponding recommended values, while the Zn level remained lower than the recommended quality levels by WHO and US-EPA. However, Cr, Pb and Fe were present at substantially elevated levels than those recommended by these agencies. For example, Cr was 21–42 times higher in concentration than the recommended quality value, while Fe and Pb were 8–12 and 2–11 times higher, respectively.

Conclusion

The present study showed that Na, Ca, Mg, K, Fe and Cr were present as major pollutants in the groundwater of Kasur with alarmingly high concentration levels, while Co, Pb and Zn emerged as minor pollutants. Strong positive linear correlations were found between Na, Mn, Cr, Fe and Mg. The PCA and CA identified industrial effluents, particularly from tanneries, to be the probable source of the observed metal pollution of the groundwater. Comparison with international standards for water quality evidenced that Cr, Pb and Fe levels were in far excess of the recommended safe limits. The recorded higher levels of Cr were of major concern, since the metal originates from the tanning industry where Cr compounds are extensively used. Overall, the influx of industrial metal contaminants in effluents discharged indiscriminately into open land in Kasur has already started showing its impact on the aquifer. It is anticipated that the results of the present study would provide the requisite basis towards formulating a future industrial pollution abatement programme by the concerned authorities.

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