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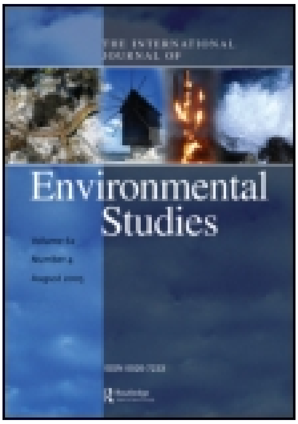

International Journal of Environmental Studies
Publication details, including instructions for authors and subscription information:
http://www.tandfonline.com/loi/genv20

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Published online: 23 Nov 2012.

To cite this article: S.K. Reza , Utpal Baruah & Dipak Sarkar (2013) Hazard assessment of heavy metal contamination by the paper industry, north-eastern India, International Journal of Environmental Studies, 70:1, 23-32, DOI: 10.1080/00207233.2012.746810

To link to this article: http://dx.doi.org/10.1080/00207233.2012.746810

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International Journal of Environmental Studies, 2013
Vol. 70, No. 1, 23–32, http://dx.doi.org/10.1080/00207233.2012.746810

Hazard assessment of heavy metal contamination by the paper industry, north-eastern India

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The spatial distribution and hazard assessment of heavy metals in the paper mill contaminated area of Jagiroad, Assam, India were investigated using statistics, geostatistics and geographic information system techniques. The amounts of Cr, Cd, Ni and Pb were determined from 188 samples collected within the contaminated area. Log-transformation was applied in order to achieve normality in the data-set. The ordinary kriging estimates of Cr, Cd, Ni and Pb maps showed that high concentrations of heavy metals were located in the low-lying areas like bils (lakes). Indicator kriged probability maps of soil Cr, Cd, Ni and Pb were prepared based on the concentrations to exceed the respective Food and Agriculture Organization maximum permissible limit (MPL) value of 100, 3, 30 and 50 mg/kg, respectively. It was seen that more than 80% of the studied area has a higher than 50% probability to exceed the MPL value of Ni. Smaller areas in the north and west side of the study area displayed a higher concentration than the MPL value of Pb. For Cr, only a small portion at the centre of the study area had a higher concentration than the MPL value. In an attempt to identify the source of heavy metals through multivariate analysis, it was concluded that Cr, Ni and Pb mainly originated from paper mill effluent and soil Cd was associated with natural concentration.

Keywords: Heavy metals; Risk assessment; Multivariate analysis

1. Introduction

The paper industry in India does not operate with the modern systems used in Western countries. The pulp and paper mills in India use various cellulose-based materials for paper production [1]. About 43% come from forest wood, 28% from agro-based product and 29% from recycling of waste paper [2]. Among the major industries, the paper industry is a notorious polluter of the environment [3,4]. In India, more than 55% of industries do not have any proper treatment methods and around 20% have partial treatment facilities. All of these industrial effluents are released into the environment without any prior treatment [5]. The pulp and paper mill industry is one of the 12 most polluting industries in India, and releases environmentally hazardous liquid effluent [6,7] containing heavy metals and other organic toxicants. During the last three decades, it has become more apparent that the total concentrations of heavy metals in soils and plants, their chemical forms, mobility and availability to the food chain provide the basis for understanding a range of problems in crop, animal and human health. Soils are the ultimate sink for trace elements in the terrestrial

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*Corresponding author: Email: ubaruah2@rediffmail.com environment and have a great capacity for receiving, purifying and decomposing wastes and pollutants of different kinds [8]. The effects of industrial effluents on soil and the use of municipal and industrial wastewater for irrigation of crops are well documented [9–11].

Environmental risk assessment can provide valuable information regarding feasible rehabilitation options. The methodology is based on the idea of 'source–pathway–target'. Risk is better assessed if quantitative techniques are used to account for spatial and temporal variations. A probabilistic assessment takes into account variability of parameters and uncertainty in measurement [12]. Geostatistics is used to assess the level of soil contamination and calculate the risk in contaminated sites by preserving the spatial distribution and uncertainty of the estimates [13–15]. Geostatistics and geographic information system (GIS) provide useful tools for the study of spatial uncertainty and hazard assessment [16,17].

Our study investigates the wastewater produced and released by the Morigaon paper mill factory in Morigaon district of Assam, India, which drains directly into the agricultural fields situated to the north-east, where the water is used for the irrigation of paddy crops which are in various stages of yield. The extensive irrigation using the effluents released from paper mills has resulted in the accumulation of heavy metals in the soil. In order to understand the extent of contamination by heavy metals in paper mill effluents, we selected an area near a paper mill, situated between Jagiroad and Kapili river, for our study. We analysed the chromium (Cr), cadmium (Cd), nickel (Ni) and lead (Pb) contents in soils collected during the investigation. We used statistics, geostatistics and GIS techniques in order to reveal the spatial distribution patterns and provide a basis for hazard assessment.

2. Materials and methods

2.1. Study area

The study site in Jagiroad, Morigoan paper mill, Assam, north-eastern India, extended between 26°05′47″ to 26°11′35″ N latitude and 92°08′33″ to 92°16′11″ E longitude: an area of 4605 ha (figure 1). The climate is humid subtropical. The maximum temperature is 33 °C during July and August; a minimum temperature falls as low as 7 °C in the month of January. Annual rainfall is 2169 mm and about 80% of rainfall is from the south-west monsoon.

2.2. Soil sampling and analysis

A total of 188 surface soil samples were collected from a depth of 0-25 cm (plough layer) using a square 500 × 500 m grid (figure 1) covering not only the waste disposal site, but also the surrounding cultivated areas with the help of a hand-held global positioning system. Soil samples were air-dried and ground to pass through a 2 mm sieve. A combined glass calomel electrode was used to determine the pH of aqueous suspension (1:2.5 soil:solution ratio). Organic carbon was determined by the Walkley and Black [18] method. Digestion of 0.50 g samples was performed with concentrated HNO_3, HF and $HClO_4$ in a microwave digester (model Start D, Milestone). Subsequently, the total concentration of heavy metals was determined by a Shimadzu AA6300 atomic absorption spectrophotometer.

2.3. Geostatistical analysis based on GIS

We used spatial interpolation and GIS mapping techniques to produce spatial distribution and risk assessment maps for the four observed heavy metals, with the software being

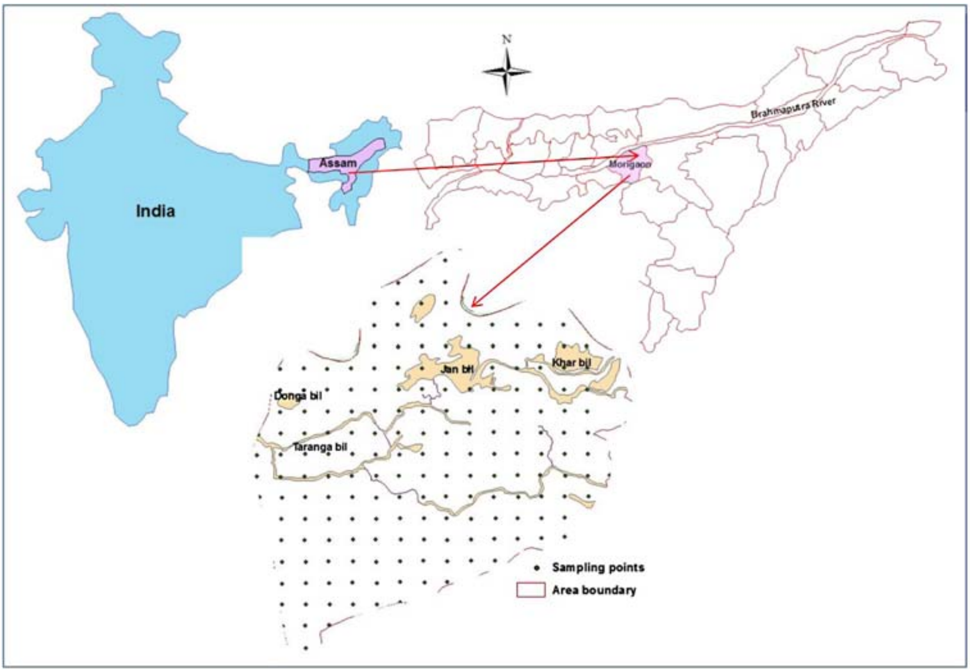

Figure 1. Location and grid map of the study area.

ArcGIS v.9.3 (ESRI Co., Redlands, USA). The first step was taking the log-transformation of all non-normally distributed target variables (heavy metal contents) to ensure (in most cases) the normality of residuals. In ArcGIS, kriging can express the spatial variation and allow a variety of map outputs, and at the same time minimize the errors of predicted values. Moreover, it is very flexible and allows users to investigate graphs of spatial autocorrelation. In kriging, a semivariogram model was used to define the weights of the function [19], and the semivariance is an autocorrelation statistic defined as follows [20]:

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

where $z(x_i)$ is the value of the variable z at location of x_i, the lag and $N(h)$ the number of pairs of sample points separated by h. For irregular sampling, it is rare for the distance between the sample pairs to be exactly equal to h. That is, h is often represented by a distance band.

Best-fit model with minimum root mean square error (RMSE) was selected for each heavy metal. Using the model semivariogram, basic spatial parameters, such as nugget (c_0), sill $(c + c_0)$ and range (A), were calculated which provide information about the structure as well as the input parameters for the kriging interpolation. Nugget is the variance at zero distance, sill is the lag distance between measurements at which one value for a variable does not influence neighbouring values and range is the distance at which values of one variable become spatially independent of another [21].

2.4. Indicator kriging

The probability maps of soil Cr, Cd, Ni and Pb concentration to exceed the respective Food and Agriculture Organization (FAO) (2000) maximum permissible limit value (MPL) of 100, 3, 30 and 50 mg/kg were prepared using indicator kriging. Indicator kriging is a non-linear form of geostatistics where the conventional linear kriging estimators are applied to the data after a non-linear transformation. Here, the non-linear transform is to a discrete (binary) indicator variable. These techniques have been widely applied by soil scientists [22].

Let us assume that a soil property z at location x takes value $z(x)$. In geostatistics, we treat this value as a realization of the random function $Z(x)$. An indicator transformation of $z(x)$ can be defined by,

$$\omega_c(x) = 1 \text{ if } z(x) \leq Z_c,\ 0 \tag{2}$$

where Z_c is a threshold value of the property. In indicator geostatistics, $\omega_c(x)$ is regarded as a realization of the random $\Omega_c(x)$,

$$\Omega_c(x) = 1 \text{ if } z(x) \leq Z_c, \text{ else } 0 \tag{3}$$

It can be seen that,

$$\text{Prob}[Z(x) \leq Z_c] = E[\Omega_{c(x)}] = G[Z(x); Z_c] \tag{4}$$

where Prob[ ], E[ ] denote, respectively, the probability and the expectation of the terms within the square brackets and $G[Z(x); z_c]$ is the cumulative distribution function of $Z(x)$ at value z_c. The principle of indicator kriging (IK) is to estimate the conditional probability that $z(x)$ is smaller than or equal to a threshold value z_c, conditional on a set of observations of z at neighbouring sites, by kriging $\Omega_c(x)$ from a set of indicator-transformed data.

A set of data on z is transformed to the indicator variable $\omega_c(x)$. The variogram of the underlying random function $\Omega_c(x)$ is then estimated by,

$$\gamma_{\Omega_c}(h) = \frac{1}{2M_h} \sum_{i=1}^{M_h} [\omega c(x_i) - \omega c(x_i + h)]^2 \tag{5}$$

where M_h pairs of observations are separated by the lag interval h. A set of estimates of this indicator variogram at different lags may then be modelled by one of the authorized continuous functions used to describe variograms [19].

An estimate of the indicator random function may then be obtained for a location x by kriging from the neighbouring indicator-transformed data. The IK is equivalent to simple kriging of the indicator variables $\omega_c(x)$ using the mean within the kriging neighbourhood as the expectation.

2.5. Multivariate statistical analysis

The identification of pollutant sources is conducted with the aid of multivariate statistical analyses, such as principal component analysis (PCA) and correlation analysis. Multivariate analyses of the data in this work were carried out by SPSS v.15.0 software (SPSS, Chicago, USA). In this study, data were log-transformed prior to PCA to reduce the influence of high values. The Bartlett sphericity test and the Kaiser–Mayer–Olkin test indicated that the normalized data were suitable for PCA. Varimax with Kaiser normalization rotation was applied to maximize the variances of the factor loadings across variances for each factor. In addition, the correlations between the original variables are presented in the form of non-parametric Pearson correlation coefficients.

3. Results and discussion

3.1. Descriptive statistics of heavy metals and other soil properties

Table 1 lists the statistical characteristics of soil Cr, Cd, Ni and Pb. The median of each heavy metal was lower than the mean, which indicates that the effects of abnormal data on sampling value were not great except for Cr and Ni. In this investigation, among the heavy metals studied (Cr, Cd, Ni and Pb), the mean concentration of Cr was high. The highest level of Cr in the polluted soil can be attributed to the higher Cr content of the paper mill effluents [23]. The greatest and the smallest standard deviations were observed in the Cr (15.87) and Cd (0.11), respectively. Organic carbon, Cd and Pb exhibit a high variation (>50%) according to the guidelines provided by Warrick [24]. Skewness is the most common form of departure from normality. If a variable has positive skewness, the confidence limits on the variogram are wider than they would otherwise be and, consequently, the variances are less reliable. A logarithmic transformation is considered where the coefficient of skewness is greater than one [19]. Therefore, a logarithmic transformation was performed for Cd and Pb because their skewness was greater than one.

3.2. Semivariogram analysis of heavy metals

Semivariogram analysis was used to characterize and quantify spatial variability and RMSE was used for different theoretical semivariogram models to fit the experimental

Table 1. Summary statistics of heavy metal concentrations and selected soil properties.

	pH	Organic carbon (%)	Cr (mg/kg)	Cd (mg/kg)	Ni (mg/kg)	Pb (mg/kg)
Mean	5.3	1.63	52.36	0.13	32.77	13.12
Median	5.1	1.43	53.83	0.12	34.72	10.41
SD	0.81	0.90	15.87	0.11	13.91	10.71
CV (%)	15.3	55.2	30.31	84.61	42.45	81.63
Minimum	3.6	0.15	12.59	0.01	0.22	0.64
Maximum	7.8	5.40	136.75	0.70	58.07	49.00
Skewness	0.68	1.10	0.42	1.40	−0.35	1.61
Kurtosis	−0.18	1.57	3.64	3.75	−0.81	2.04
Distribution pattern			Normal	Lognormal	Normal	Lognormal

Abbreviations: SD, standard deviation; CV, co-efficient of variation.

Table 2. Semivariogram model and parameters of heavy metals.

Heavy metals	Fitted model	Nugget (C_0)	Sill ($C+C_0$)	Range (A) (m)	Nugget/sill
Cr	Circular	0.894	1.77	1408	0.505
Cd	Gaussian	0.514	0.98	2263	0.524
Ni	Gaussian	0.712	0.97	2499	0.734
Pb	Gaussian	0.615	0.84	2702	0.732

semivariogram values for each micronutrient. Analysis of the isotropic variogram indicated that the Cd, Ni and Pb semivariograms were well described with the Gaussian model, with the distance of spatial dependence being 2263, 2499 and 2702 m, respectively, while the

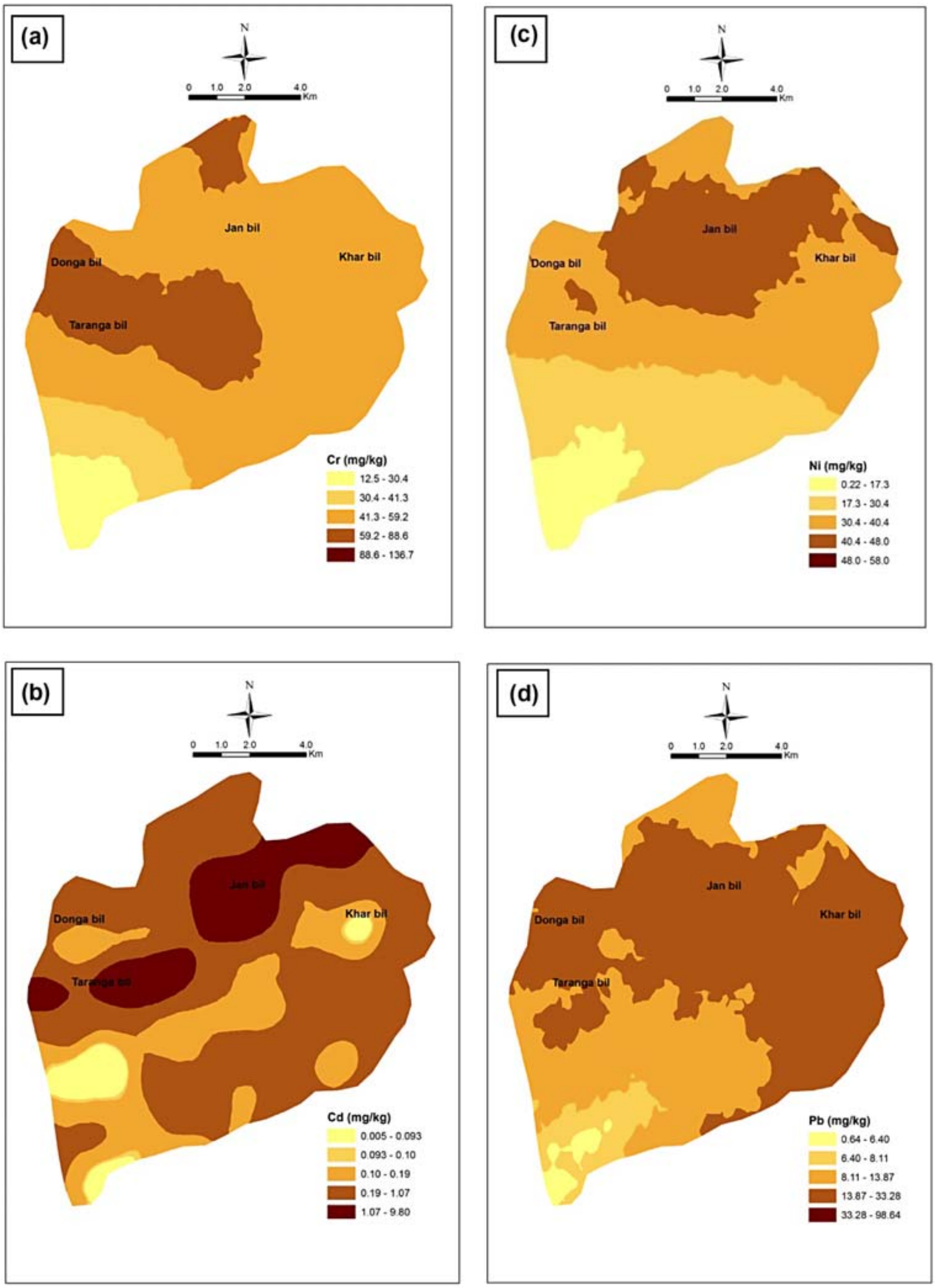

Figure 2. Spatial distribution maps of (a) Cr; (b) Cd; (c) Ni; and (d) Pb.

Cr semivariogram was well described with the circular model, with the distance of spatial dependence being 1408 m (table 2).

In the semivariogarm analysis, the nugget values represent the variability of measured heavy metals level at zero distance, which are positive in this study for all the heavy metals. This spatial random variance is caused by the artificial nature of heavy metal pollution in soil; meaning that anthropogenic inputs are a significant source of heavy metals in the study area. The sill, sum of partial sill and nugget, is the maximum variance between data pairs and reflects the variations of regionalized variables in the study area. The ratio of nugget and sill is commonly used to express the spatial autocorrelation of regional variables, which also indicates the predominant factors among all natural and anthropogenic factors [25]. The ratios of nugget and sill between 0.25 and 0.75 represented moderate spatial dependence; those below 0.25 represented strong spatial dependence; and all others represented weak dependence. All heavy metals were moderately spatially dependent suggesting that they are affected by both anthropogenic and natural factors.

3.3. Spatial distribution and risk assessment of heavy metals pollution

With the use of the available measurements for Cr, Cd, Ni and Pb concentration as well as the aforementioned structural models, we produced spatial maps of these pollutants using the ordinary kriging procedure [26]. The spatial distribution maps of Cr, Cd, Ni and Pb (figure 2(a)–(d), respectively) showed that there was a high concentration of heavy metals located in the low-lying areas like bils (lakes and swamps), namely Donga, Jan, Khar and Taranga. Table 3 shows the evaluation indices for soil heavy metals in terms of mean absolute error (MAE), mean square error (MSE) and goodness of prediction (G) obtained from cross-validation procedures. For all soil heavy metals the G value was greater than zero. This indicates that spatial prediction is better than assuming mean of observed value as the property value for any unsampled location. This also shows that semivariogram parameters, obtained from fitting of experimental semivariogram values, reasonably describe the spatial variation.

In order to obtain data that may be used in the future for the assessment of the health risk due to elevated soil heavy metals concentration in cultivated areas, we produced spatial maps of the probability that these pollutants exceed the corresponding MPLs. Figure 3 (a)–(d) shows the indicator kriged probability maps of soil Cr, Cd, Ni and Pb based on the concentrations to exceed the respective FAO [27] MPL value of 100, 3, 30 and 50 mg/kg, respectively. It was seen that more than 80% of the studied area has a higher than 50% probability to exceed this MPL value of Ni. A smaller area in the Jan and Taranga bils of the study site displayed a higher concentration than MPL value of Pb and Cd. For Cr only a small portion at the centre of the study area had a higher concentration than MPL value.

Table 3. Evaluation performance of ordinary kriged map of heavy metals through cross-validation.

Heavy metals	MAE	MSE	G
Cr	0.006	163.3	34.7
Cd	0.049	3.0	70.9
Ni	0.068	98.6	48.7
Pb	0.089	178.2	24.8

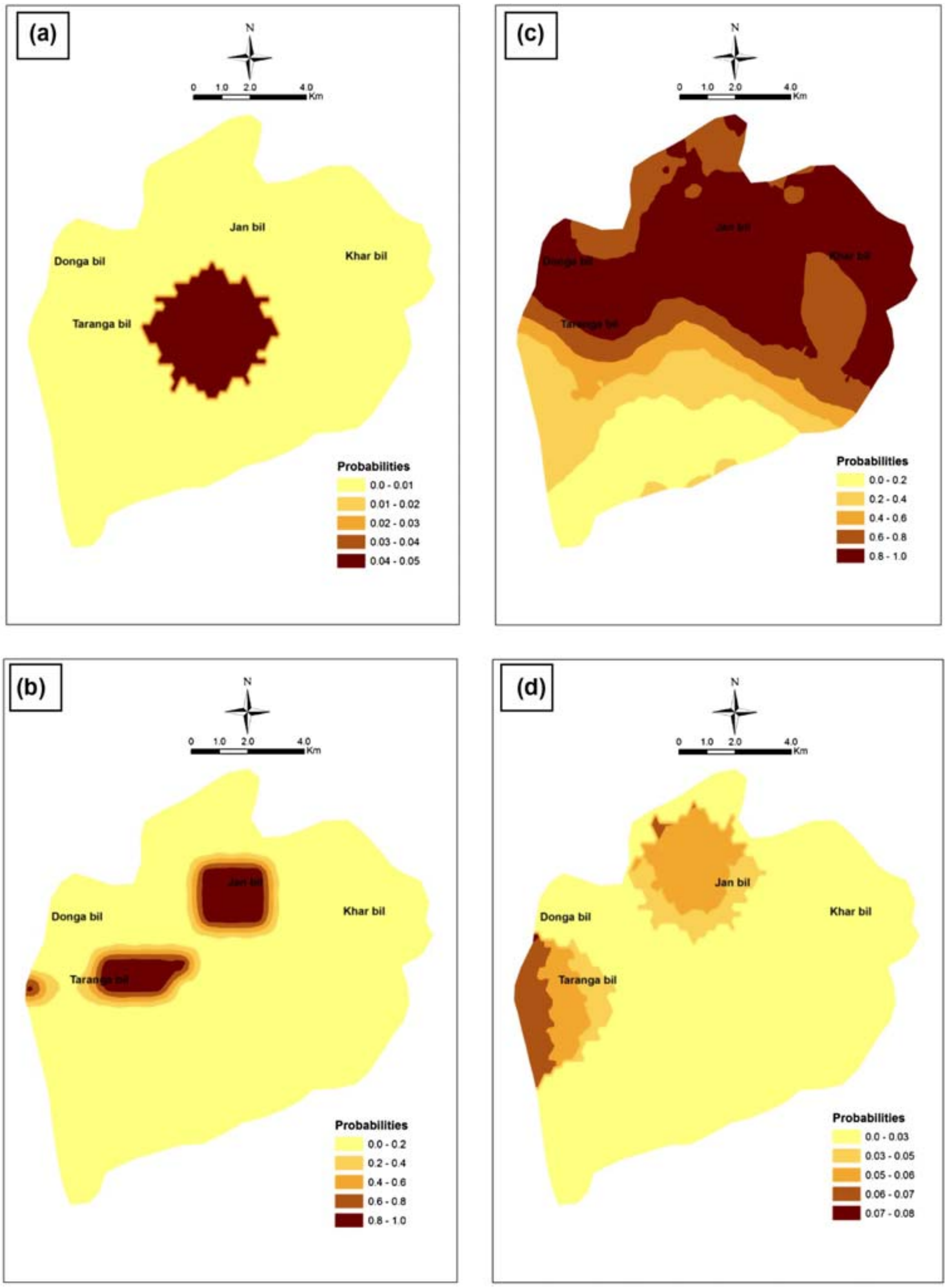

Figure 3. Risk assessment maps of (a) Cr; (b) Cd; (c) Ni; and (d) Pb.

3.4. Source identification based on multivariate statistics

We obtained the loadings of measured heavy metal concentrations in the coordinate system of three principal components (PC) by analysing the correlation matrix. The values of

Table 4. Matrix of the three PC accounting for most of the total variance.

Heavy metals	PC1	PC2	PC3	Communities
Cr	0.82	0.07	−0.02	0.93
Cd	0.08	0.98	−0.02	0.97
Ni	0.70	0.53	−0.23	0.83
Pb	0.91	−0.10	0.20	0.89
Percentage of variance	38.88	30.51	21.15	
Cumulative per cent	38.88	69.39	90.54	

Table 5. Pearson correlation coefficient of heavy metal contents.

Heavy metals	Cr	Cd	Ni	Pb
Cr	1.00			
Cd	0.06	1.00		
Ni	0.40^{**}	0.11	1.00	
Pb	0.22^{**}	0.05	0.40^{**}	1.00

$^{**}p < 0.01$ level (two-tailed).

loadings as well as the cumulative percentage of variance (table 4) showed that Cr, Pb and Ni are well represented by the first three PC, which account for over 80% of the total variance for Cr and Pb. The Cr, Pb and Ni are highly loaded in PC1 which explains 38.88% of the total variance. There are significant correlations between their levels in the soils of the study area (table 5), which imply that these three heavy metals in soils may have originated from a similar source, such as paper mill effluent because of the use of various cellulosic-based raw materials and chemicals used during the manufacturing process [1]. But, in PC2, Cd showed a high value, distinguishing itself from other heavy metals. This may reflect different anthropogenic or geogenic sources and possible pathways of accumulation from other investigated heavy metals.

4. Conclusion

Raw data-sets of heavy metals in the soils of Jagiroad paper mill area are strongly positively skewed and may contain outlier values. The application of log-transformation was effective in normalizing the data in addition to weakening the negative effect of outliers. A good variogram structure of heavy metals was observed, showing that there are clear spatial patterns of heavy metals on the distribution map and also that the current sampling density is sufficient to indicate such spatial patterns. The kriging interpolated map showed areas with high values of heavy metal concentrations. The probability map based on kriging interpolation provided useful information for hazard assessment. The Cd in soils was more likely influenced by natural factors, namely weathering from parent materials. In other words, this study demonstrated that geostatistical and multivariate analyses were effective tools for assessing heavy metal contamination in industrial areas.

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