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Assessment of heavy metal contamination in soils irrigated with paper mill effluent in West Bengal, India

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ABSTRACT

The Pulp and paper industry has been associated with potential environmental concern for the release of untreated waste in the surrounding environment. The present investigation was conducted in a village at Manikpara, India, to assess heavy metals present in the soil and their level of contamination due to the accumulation of paper mill waste. Soil samples (0-15 cm depth) were collected from four sampling sites surrounding the paper mill. Some selected metals like Sc, Cr, Mn, Fe, Co, Ni, Cu, Zn, Pb, Cd, were analyzed using Inductively Coupled Plasma Mass Spectrometer (ICPMS) and the heavy metal contamination level are assessed using the indices: geoaccumulation index (Igeo), enrichment factor (EF), contamination factor (CF), degree of contamination (C_d) and the pollution index (PI). The calculated value of CF for Cr (6.08) and Pb (9.37) indicates very high contamination whereas the EF for Cr (2.68) and Pb (4.13) indicates the moderate enrichment. The calculated Igeo values for Cr (1.2) and Pb (1.88) also indicates moderate contamination. From the evaluated EF, Igeo and CF values of other studied metals imply moderate contamination. The calculated degree of contamination (C_d) and pollution index (PI) of different sites decreases with distance from the paper mill and their values indicate that all the soil samples were severely polluted. Principal component analysis (PCA) was performed to estimate the sources and the behaviour of contamination.

Keywords: Geoaccumulation index, enrichment factor, contamination factor, degree of contamination, pollution index

INTRODUCTION

Environmental degradation arises by intense urbanization as well as the introduction of large scale unmethodical industrialization for the last few decades. With the industrial expansion, the need for the treatment and disposal of waste has grown up (Sarala *et al.* 2011). Various industrial activities responsible for soil contamination adjacent to the industry are a serious concern due to the untreated waste disposal. Soil contaminations with metals are found in areas with a high industrial activity where the accumulation of metal content is higher than the uncontaminated area (Loska *et al.* 2004). Many heavy metals with specific oxidation state are toxic due to their non-biodegradable nature, long biological half-lives (Chopra *et al.* 2009) attribute to bioaccumulate in the body and the food chain (Zhang *et al.* 2017). Wastewater and various solid industrial wastes contain appreciable amounts of toxic heavy metals such as Cd, Cu, Zn, Cr, Ni, Pb, and Mn in surface soil which create a problem for safe rational utilization of agricultural soil (Luo*et al.* 2012).

Globally, the pulp and paper industry is considered as the most polluting industry as toxic waste production reached in every year is likely over one million metric tonnes (Kinnarinen*et al.* 2016). The waste disposal is a global dilemma for paper industries due to the high volume of effluent generation, limited space for land-based treatment and disposal, high cost of waste treatment (Thompson *et al.* 2001). The untreated waste contains toxic materials released from the paper mills would destroy the water, land, and air around the paper mills. Coal fly ashes, green liquor dregs, slaker grits, lime mud and pulp mill sludge of wastewater treatment plants (WWTP) derived from pulp and paper mills generate various inorganic and organic wastes (Monte *et al.* 2009). The biodegradable organic contaminants are oxidized to carbon (IV) oxide by microbial action are less persistent, and presumably have transient and less serious effects in the soil environment. But the inorganic substances such as heavy metals are chelated with the organic matter in wastewater often present in substantial quantities as most metals do not undergo microbial or chemical degradation, and their total

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concentration in soils persists for a long time after their introduction (Kirpichtchikova *et al.* 2006). So places near the paper mill waste disposal site displayed a higher potential risk from metal contamination (Borah *et al.* 2017). Dumping of solid waste and fly ash may also contribute to the preferential adsorption not only of heavy metals but also rare earth metals in the soil surrounding the paper mill (Das 2019). The concentrations of heavy metals in the soil are decreased with distance from the paper mill (Phukan *et al.* 2003). Several reports have published about the content of heavy metal, their translocation pattern to different parts of plant grow in the soil affected with the paper mill waste (Kumar*et al.* 2015, Reza *et al.* 2015, Borah *et al.* 2017, Phukan *et al.* 2003).There are many paper mills situated at fast-growing medium or small cities or villages. But less information is available about the soil contamination with heavy metal in these cities or villages. The medium or small industrial cities or villages face serious environmental issues than megacities due to poor environmental protection strategies and inadequate pollution treatment (Lu*et al.* 2014). Manikpara is a developing small village in Jhargram (W.B). Till date, no study has been carried out for the assessment of heavy metal in the soil affected with the paper mill waste at Manikpara. The present work is aimed to assess the selected heavy metals (Sc, Cr, Mn, Fe, Co, Zn, Cu), their contamination level present in the soil around the paper mill of Manikpara.

MATERIALS AND METHODS:

Soil sample collection: The paper mill is located in Manikpara (22^0 $22^{'}$ N, 87^0 $7^{'}$ E), West Bengal. One control soil sample was taken from an area (3 km away from the paper mill) which is uncontaminated with the paper mill effluent/ solid waste but underlain by similar geological units. Soils of other four sites (S-1, S-2, S-3, S-4) with a distance of 1 km, 750m, 500m, 250m respectively, from the paper mill, are selected for the study. Soils of these sites having been irrigated with effluent/solid waste from the paper mill for more than 5 years. 12 soil samples (3 soil samples from each site) at depth ranging 0-15 cm were collected during June-2018.

Sample preparation and analysis: Metal content was measured on a Thermo Fisher Scientific iCAP-Q quadrupole Inductively Coupled Plasma Mass Spectrometer (ICPMS) at the Radiogenic Isotope Facility of IIT Kharagpur. 0.05 g of each powdered soil samples were dried overnight at 100 °C before digested using a mixture of supra pure (3:1) 24 M hydrofluoric acid: 14M nitric acid on a hot plate at 120 °C for two days then evaporated with two repetitions. The samples were refluxed with a mixture of nitric acid and hydrogen peroxide at 120 °C for 12 hours (5 drops of H_2O_2 + 3ml HNO_3), dried and dissolved in 3 ml HNO_3. The final digested samples in 2% HNO_3 with 10 ppb Indium (In) (internal standard) were diluted to 100 ml in pre-cleaned 100 ml HDPE bottles. The international rock standards (JG–2 and JB–3; granite and basalt) along with a couple of procedural blanks were also prepared with the same protocols and digested together with the samples. The samples were centrifuged and an aliquot from the supernatant used for analysis. The solutions were measured using the standard sample introduction system on the quadrupole ICPMS. The sample solutions were introduced to a Peltier–cooled glass cyclonic spray chamber using the peristaltic pump and PFA nebulizer with a flow rate of 100µL/min. Using a custom 1 ppb multi-element solution on the masses ^{7}Li, ^{58}Co ^{115}In, ^{142}Nd, ^{208}Pb, ^{232}Th, and ^{238}U covering the entire mass range, the mass spectrometer was optimized for its maximum sensitivity. The oxide production (ranges between 1.5 and 2%) was monitored on ^{140}Ce^{16}O/^{140}Ce. The concentrations were calculated using the calibration curve method using the rock standards digested together with samples. For each element, the calibration curve is forced through blank.The SPSS 21.0 software package was used to statistical analysis of the data.

Single-element pollution indices: This gives us the information about how an individual metal is concentrated at examined site relative to a background and thus can be used to evaluate metal contamination. These include Enrichment factor (EF), Geo-accumulation index (Igeo), Contamination factor (CF).

Determination of enrichment factor (EF):EF of elements in the soil samples were determined by comparing the concentration of each element against the concentration of a reference element (e.g., Al, Fe, Ti, Sc, and Mn) which is generated

by anthropogenic sources (Reimann and Caritat 2000). The EF value was calculated by using the formulae:

$EF = \frac{C_n(Sample) / C_{ref}(Sample)}{B_n(background) / B_{ref}(background)}$, where C_n (sample), C_{ref} (sample) are the concentration of the element of interest in the examined environment (soil in this study), the concentration of the reference element in the examined environment, respectively and B_n (background), B_{ref} (background) are the concentrations of the element of interest and the reference element in the background sample, respectively. The EF is a tool to determine the possible sources of elements i.e. crustal/geogenic/lithologic or anthropogenic. For this study, Sc is taken as the reference metal. Sutherland suggested five contamination categories of EF: EF < 2 deficiency to minimal enrichment; EF = 2– 5 moderate enrichment; EF = 5–20 significant enrichment; EF = 20–40 very high enrichment; and EF > 40 extremely high enrichment (Sutherland 2000).

Determination of Geo-accumulation Index (Igeo): The techniques of Igeo and EF were used to determine the intensity of heavy metal pollution of the examined soil. The geo-accumulation index (Igeo) formula calculated for different metals is as follows (Muller 1969): $I_{geo} = \log_2 \frac{C_n}{1.5 \times B_n}$, Where C_n is the measured concentration of element n in the soil and B_n is the geochemical background for the element n which was taken from a controlled site. Factor 1.5 is the background matrix correction factor used to reduce the effect of lithogenic variability. Muller (1969) proposed seven grades or classes of the geoaccumulation index as follows: I geo ≤ 0 practically uncontaminated; 0 < Igeo ≤ 1 uncontaminated to moderately contaminated; 1 < Igeo < 2 moderately contaminated; 2 < Igeo < 3 moderately to heavily contaminated; 3 < I geo < 4 heavily contaminated; 4 < Igeo < 5 heavily to extremely contaminated; and 5 < Igeo extremely contaminated.

Determination of Contamination factor: The assessment of soil contamination was also carried out using the contamination factor and degree of contamination. The contamination factor was calculated using the formula (Hakanson 1980) as follows: $CF = \frac{C_{metal}}{C_{background}}$

Where C_{metal} and $C_{background}$ represents the concentration of metal of interest at a site and concentration of the same metal at a control site (background), respectively. Hakanson, (1980) classified the degree of soil contamination based on contamination factor (CF) as follows: CF < 1: low contamination; 1≤ CF ≤ 3: moderate contamination; 3 ≤ CF ≤ 6: considerable contamination; CF > 6: Very high contamination.

Multi-element pollution indices: Multi-element pollution indices were used for the assessment of soil due to the limitations of a single element pollution indices (Duodu *et al.* 2016). The most common and widely used are the contamination degree (C_d) and pollution index (PI). The sum of the contamination factors of all elements examined represents the degree of contamination (C_d) of the soil: $C_d = \sum_{i=1}^{n} CF^i$. Four classes are recognized according to Hakanson (1980) as follows C_d< 6: low contamination degree, 6≤ C_d<12: moderate contamination degree; 12 ≤ C_d< 24: considerable contamination degree; C_d ≥ 24: very high contamination degree.

Nemerow (1991) introduced the pollution index (PI) as: $PI = \sqrt{\frac{(CF_{av})^2 + (CF_{max})^2}{2}}$

where CF_{av}, CF_{max} are the average of contamination factors and maximum contamination factor. Nemerow (1991) classified PI as unpolluted (PI<0.7), slightly polluted (0.7<PI<1), moderately polluted (1<PI<2), heavily polluted (2<PI<3) and severely polluted (PI>3).

RESULTS AND DISCUSSIONS

Heavy metal contamination in soil samples

The mean concentration values of all metals were higher than the metals present in the soil of the control site (Table 1). In the paper mill the heavy metals mostly present in the black liquor and the sludge generated from the waste water treatment plant (Manskinen *et al.* 2011, Monte *et al.* 2009). So the soil irrigated with the paper mill effluent may presumably increase the heavy metal content. The order of mean concentration values (in ppm) are: Fe (8205.7) > Mn (78.51) > Zn (33.44) > Cr (30.64) > Cu (12.36) > Ni (10.21) > Pb (6.05) > Co (4.74) > Sc (3.01) > Cd (0.1). High concentration of Fe

present in this soil may be attributed to the presence of laterite soil in this geographical area (Ghosh*et al.* 2015). The high values of skewness, kurtosis, SD in the data indicated the occurrence of geochemical abnormalities and human activities (Lu*et al.* 2012). Skewness of Cd exceeds unity which denotes asymmetry and positively skewed towards lower concentration.

Table 1: Descriptive statistics of trace element concentration (ppm) in soil samples collected from Manikpara

Element	Mean	Std. deviation	Skewness	Kurtosis	Range	Control
Sc	3.01	1.34	-0.04	-2.04	3.15	1.32
Cr	30.64	4.52	-0.87	-0.69	11.67	5.03
Mn	78.51	13.99	0.05	-1.49	40.60	31.75
Fe	8205.70	1269.71	-0.11	-1.12	3801.31	4530.96
Co	4.74	1.40	-0.35	-0.84	3.78	1.27
Ni	10.21	1.64	0.86	0.28	5.53	4.77
Cu	12.36	3.04	-0.40	-1.04	8.95	6.38
Zn	33.44	10.49	0.25	-1.44	29.66	21.82
Cd	0.10	0.04	1.13	0.12	0.142	0.05
Pb	6.05	2.74	0.73	-0.63	8.24	0.64

Metal pollution assessment in soil samples

Enrichment factor (EF): The mean enrichment factor of Pb (4.13) and Cr (2.68) were higher than other studied metals (Fig.1) and this is in good agreement with the other published report (Kumar and Chopra, 2013).The order of EF value for other metals are: Co (1.64) > Mn (1.08) > Ni (0.94) > Cu (0.85) > Cd (0.81) > Fe (0.79) > Zn (0.67). On the basis of enrichment factor, the examined soil samples were moderately enriched with Pb and Cr (as EF lies between 2 to 5) and minimally enriched with Mn, Co, Ni ,Cu ,Cd , Fe and Zn as EF for these metals are < 2.

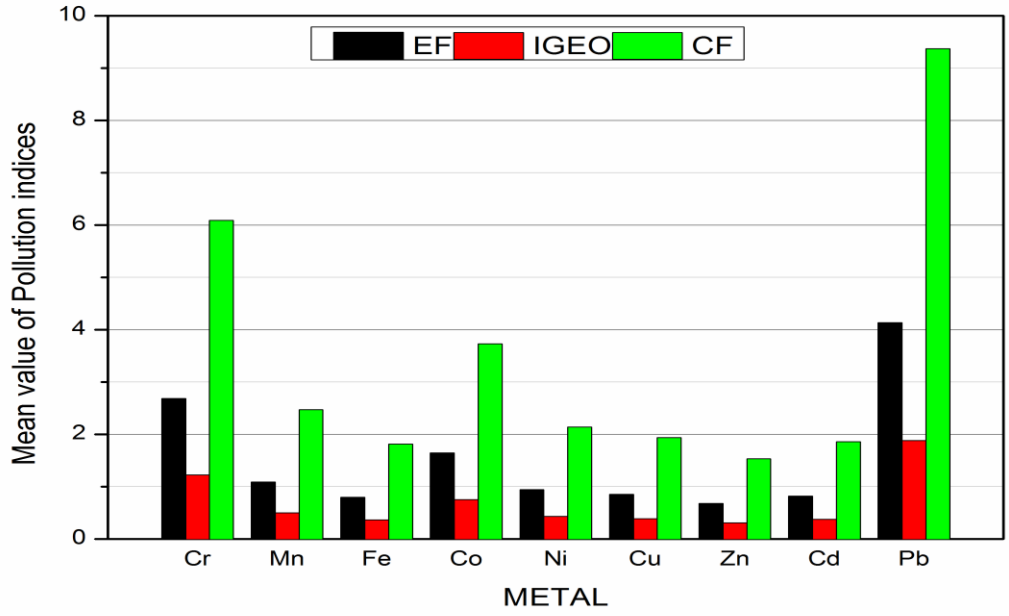

Fig.1: Mean value of single-element pollution indices (EF, Igeo, CF) of the studied metals

Geo-accumulation Index (Igeo): The mean Igeo values of elements Cr (1.22) and Pb (1.88) are higher than other metals (Fig.1). Based on the geo-accumulation index, the soil surrounding the paper mill classified to be moderately contaminated (as 1 < Igeo < 2) with Cr and Pb and uncontaminated to moderately contaminated (as 0 < Igeo ≤ 1) with other metals. The order of Igeo values of other metals: Co (0.74) > Mn (0.49) > Ni (0.42) > Cu (0.38) > Cd (0.37) > Fe (0.36) > Zn(0.30).

Contamination Factor: The mean contamination factor of Pb (9.37), Cr (6.08) which is > 6 (Fig.1) indicates the examined soil samples of surrounding the paper mill are very high contamination with Pb and Cr. This was followed by Co (3.73). Zn (1.53) had the least contamination factor among the metals studied. Based on the rating of contamination intensity, these soils are classified as being very high contaminated with Pb and Cr, considerable contaminated (3≤CF ≤ 6) with Co (3.73), moderately contaminated (1≤ CF ≤ 3) withMn(2.47), Ni(2.14), Cu(1.93), Cd(1.85), Fe(1.81), Zn(1.53).

Results of multi-element pollution indices: The order of calculated degree of contamination (C_d) of different sites are: S-4 (43.25) > S-3 (35.18) > S-2 (27.43) > S-1 (21.41). Based on the classification, all the sampling sites (S-4, S-3, S-2) are a very high degree of contamination with the metals. The calculated PI value of four sites also shows the same trends as in case of C_d i.e. S-4 (7.16) > S-3 (4.98) > S-2 (3.39) > S-1 (2.57).The PI values indicated that S-1 site is severely polluted and the other sites (S-2,S-3,S-4) are heavily polluted with the studied heavy metals (Kolawole *et al.* 2018). It is apparent from the Fig.2 that the soil samples adjacent to the paper mill had high values of C_d and PI than the soil lying much away from the paper mill (Fig.2). As the soil nearer to the paper mill will be more contaminated with the paper mill effluent and thus accumulates high content of heavy metals. This results in high value of PI and C_d than the other soil samples.

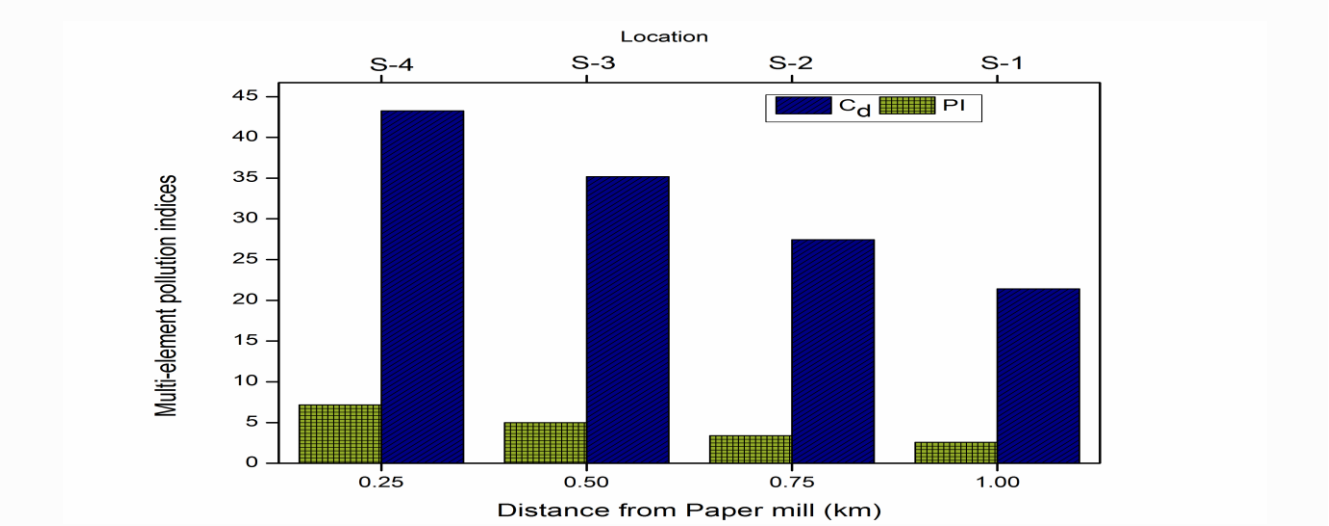

Fig.2: Change of multi-element pollution indices (C_d, PI) with distances from the paper mill at different studied location

Correlation analysis:A very significant correlation at 0.01 level of significance arises between the pair of metals: Cu-Zn (0.932), Sc-Co (0.83), Co-Fe(0.749), Fe-Sc(0.773). The correlation matrix also exhibits a negative correlation between Cr with Co, Cd, Fe, Sc and Pb with Cu, Fe, Sc, Zn, Ni (Table 2). The metals Cu, Co, Cd, Fe, Pb, Zn, Ni were seen to exhibit minimal enrichment (EF<2) and belong to uncontaminated to moderately contaminated (as 0 < Igeo ≤ 1) category.

Heavy metal sourcing: The results were subjected to principal component (PC) analysis by applying varimax rotation with Kaiser normalization to determine the sources of heavy metals and the possible factors contributing towards their concentrations in the studied soil samples (Table 3). The results indicated that there were four factors with Eigenvalues greater than 1.0 are responsible for 95.05% of the total variance in the analysis. The factor loading of the corresponding metals greater than 0.5 (marked as italics in the table) is regarded as significant for the interpretation of the data. Out of the total variance, 33.63% was controlled by component 1 having initial eigenvalues 3.36 showing higher loadings for Sc, Co, Fe, and Mn. Correlation analysis also showed significant correlations between these metals at 0.01 level of significance (Table 2), which indicated that the above metals might have originated from the common source.

Table 2: Pearson's correlation matrix of heavy metals in the soil samples of Manikpara

	Cr	Co	Ni	Zn	Pb	Cu	Cd	Mn	Fe	Sc
Cr	1									
Co	-0.075	1								
Ni	0.364	-0.579*	1							
Zn	0.406	0.300	0.452	1						
Pb	0.216	0.014	-0.460	-0.654*	1					
Cu	0.627*	0.185	0.602*	0.932**	-0.518	1				
Cd	-0.194	0.255	-0.167	-0.008	0.127	-0.139	1			
Mn	0.355	0.627*	-0.081	0.540	0.076	0.465	0.489	1		
Fe	-0.294	0.749**	-0.369	0.140	-0.133	0.099	-0.225	0.193	1	
Sc	-0.577*	0.830**	-0.513	0.196	-0.300	-0.026	0.278	0.333	0.773**	1

**Correlation is significant at the 0.05 level (2-tailed), ** Correlation is significant at the 0.01 level (2-tailed)*

The component 2 with initial eigenvalues 3.22 showing higher loadings for Cu, Zn, Ni, and Cr metals, accounting for 32.24% of the total variance. Correlation analysis also confirms that very significant correlation at 0.01 level of significance between Cu-Zn and significant correlation at 0.05 level of significance are present between other metals of this component. This also indicates the origin of these metals is common. The dominant metals with high factor loadings in component 3 (17.1% variance with initial eigenvalues 1.71) are Pb, Mn, Cr, and Cd. Factor 4 explaining 12% of the total variance but no other metal contributing with high factor loadings. From the rotated component matrix (Table 3) it is evident that Cr and Mn exhibits a joint relationship with factor 2 and 3, and with factor 1 and 3, respectively suggesting that Mn and Cr has a combined origin. The factor loadings from Table 3 points out that Cr (0.65, 0.54) and Mn (0.61, 0.62) have multiple sources of origin.

Table 3: Rotated component matrix of trace elements studied for Manikpara

Metal	Component			
	1	2	3	4
Co	*0.954*		0.185	0.185
Sc	*0.93*	-0.220	-0.254	-0.138
Fe	*0.777*	-0.122	-0.336	0.428
Cu	0.211	*0.965*		0.104
Zn	0.390	*0.892*		
Ni	-0.469	*0.740*	-0.217	-0.209
Cr	-0.255	*0.653*	*0.543*	0.429
Pb	-0.207	-0.534	*0.706*	0.358
Mn	*0.610*	0.390	*0.62*	
Cd	0.294	-0.141	*0.526*	-0.762
Eigenvalue	3.363	3.224	1.717	1.2
% of Variance	33.632	32.247	17.175	12.004
Cumulative %	33.632	65.88	83.056	95.06

The metal association can be attributed to two sources: anthropogenic and geogenic (locked in the mineralogy of the soil). The anthropogenic (industrial and related activity) sources were defined by the groupings in component 1 and 2. The metals associated with these components are generated by the paper mill waste and dispersed indiscriminately within the nearest soil. The geogenic contribution to metal concentrations is defined by component 3 and 4.

It may be concluded from the results that all the trace metal concentrations from the four sampling sites (within one km radius from the paper mill) were higher than the uncontaminated soil (control soil). Among the all studied metals, the evaluated EF, Igeo, and CF revealed that there had been considerable enrichment of Pb and Cr than the other studied metals. The source of this heavy metal enrichment mainly attributed to the industrial and associated geogenic activity. The degree of contamination

as well as the pollution index values alarming very high contamination with the heavy metals of the soil adjacent to the paper mill. Hence continuous monitoring of the industrial waste generated from the paper mill is required for the control of possible contamination of heavy metals to the adjacent soil. Implementation of policies should adopt that encouraging the internal recycling of different waste products and thus reduction of waste generation. Further study is also recommended using the soil samples to determine the translocation of heavy metals from the soil to different plant parts, to evaluate the potential impact of heavy metals on human health risks.

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