

## Research Article

# Distribution Pattern of Some Heavy Metals in the Soil of Silghat Region of Assam (India), Influenced by Jute Mill Solid Waste

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Received 15 June 2012; Accepted 16 July 2012

Academic Editor: Vladislav Chrastny

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Physico-chemical Analysis of the soil of Silghat region of Nagoan district of Assam, India was carried out to assess the possible effect of jute mill solid waste in the distribution pattern of some heavy metals. Soil samples were collected from ten different locations of the dumping site of the mill and their physico-chemical parameters were analyzed. Scanning Electron Microscopy-Energy Dispersive X-ray analysis (SEM-EDX) and Inductively coupled Plasma-Optical Emission Spectrometer (ICP-OES) analysis of the samples shows that soil of the dumping sites is highly enriched with heavy metals. The concentration of Fe, Cu, Zn and Pb in the contaminated soil was found to be very much higher than both the continental crust and world rock average values. Correlation studies indicate strong positive correlation between organic carbon content ( $C_{org}$ ) with all the metals investigated. pH has negative correlation while positive correlation was observed for electrical conductivity with all the metals. Extent of metal contamination was assessed by determining contamination degree (CD) and pollution load index (PLI).

## 1. Introduction

The Assam Co-operative Jute Mills Ltd., situated on the south bank of the river Brahmaputra at Silghat in the Nagaon district of Assam (latitude  $26^{\circ} 36' 52''$  N, longitude  $92^{\circ} 56' 14''$  E), is one of the two jute mills of India in the cooperative sector. It was commissioned in 1970. At present it proves to be a successful cooperative venture and an ISO 9001 : 2000 certified company.

The jute industry in India generates around 40,000 t of processing wastes as by product, commonly known as cadies [1]. The major constituent of cadies is unspinnable short jute fibers. Other constituents of this waste are parts of finished product, dyed stuff, batching oil, inorganic dirt, and leftover chemicals [2]. Traditionally cadies were utilized by jute industries along with coal as fuel for boiler to generate steam which was required to run the sizing and calendaring machine. However, with technological advancement and change in processing technology steam boilers are no longer indispensable. Moreover the jute cadies are not efficient fuel due to their low calorific value, thermal efficiency, and bulk density [1, 3]. Attempts have been made to use jute cadies for

making composites, laminated sheets, papers, and adhesive bonded nonwoven fabric [3]. However, most of them failed to make any promising impact due to huge production cost and less efficiency of the products. Hence huge quantity of this fibrous industrial waste is often discarded as refuse in open landfills.

The Assam Co-operative Jute Mills LTD has been dumping the jute cadies by product since its commission in various locations on the bank of river Brahmaputra close to the mill (most of these locations are extensively cultivated with rice and tuber root vegetables). Very often they are burnt to reduce the volume. Though jute fibers are ecofriendly and biodegradable, jute cadies are enriched with various chemicals, oils, and dyes and prove to be a potential threat for soil and water pollution if dumped on open surface or near water bodies [4]. One of the most important pollutants of cadies is phenolic compounds. Such organic compounds are highly persistent and can eventually contaminate both animal and human food chain [5]. To increase the softness and flexibility and improve the spinnability, jute fibres are conventionally softened before the carding stage of spinning operations by an emulsion of petroleum oil (b.p.  $270-450^{\circ}\text{C}$ )

known as jute batching oil (JBO) along with a chemical emulsifier and water in different proportions (oil : emulsifier : water = 20.0 : 1.0 : 79.0) for the production of fine yarns [5, 6]. JBO hydrocarbons consist of *n*-alkanes, isoalkanes (e.g., pristane, phytane), and aromatics. The *n*-alkanes are degradable by bacterial action and they do not exist in nature. But the pristane, phytane, and so forth, resist bacterial action and accumulate in the nature in an undegradable state. Hence, they are harmful and are also not eco-friendly [7]. The polycyclic aromatic hydrocarbons (PAH) fraction of the JBO is recognized as chemical carcinogen and carries harmful effects [8]. Another serious environmental concern is the use of heavy metal salts and compounds in various stages of industrial production of jute fibres and finished products [6]. These chemicals have easy access to the soil of the waste dumping site. Once these heavy metals are deposited, they are not degraded and persist in the environment for years causing serious environmental pollution [6, 9]. They can be easily absorbed by the green plants, which are primary producers in the ecosystem. As they move up the food chain from producers to consumers, they endanger public health by bioaccumulation in plant and animal tissues and can cause physiological and neurological disorders [9]. These heavy metals can easily bioaccumulate into the tissues of root crops (tuber) which are widely cultivated in the region which is of great concern [9, 10]. Arsenic and heavy metal accumulation in rice plants (*Oryza sativa* L.) is viewed as a newly recognized disaster for SouthEast Asia, where rice is a staple food [6]. Heavy metals can also affect the ecosystem by migrating into ground water [8]. Hence the present study was undertaken to analyze the physico-chemical parameters and assess the heavy metal contamination of the soil of the dumping yard of the jute mill.

## 2. Material and Method

**2.1. Location of the Study Area.** Silghat is a small town in the Nagaon district of Assam, India. The dumping yard of Assam Co-operative Jute Mills LTD. (latitude 26° 36' 52'' N, longitude 92° 56' 14'' E) is a region of around one square km on the southern bank of river Brahmaputra. The map of Nagaon district of Assam (India) showing Silghat region is presented in Figure 1. The soil of the area is alluvial plain soil. It is grey to yellowish grey and is unaltered alluvium representing sand, silt, and humus rich bog clay. Mineral weathering and geochemical changes are nominal. They are slightly alkaline to acidic in nature.

**2.2. Sample Collection.** Soil samples were collected from a depth of 5–10 cm. 20 samples were collected from various locations of the dumping site at a distance of around 200 m. The soil samples were collected in September, 2011 and stored in 1 L air tight plastic containers. They were then air dried, grounded, and sieved through 2 mm sieve.

**2.3. Electrical Conductivity and pH Determination.** Electrical conductivity and pH of aqueous extract of the samples were determined using Weiber  $\mu$ -processor conductivity meter

and Systronics MK VI digital pH meter. Aqueous extract was prepared by mixing 20 g of the air dried sample with 100 cm<sup>3</sup> of double distilled water in a 200 cm<sup>3</sup> beaker. The mixture was stirred for 30 minutes at 40°C using a hot plate with magnetic stirrer. The mixture was then allowed to stand for one hour. The partly settled suspension was then used for the pH and conductivity measurement [9].

**2.4. CHN Analysis.** CHN analysis was done using Perkin Elmer 2400 CHN Analyzer. An inductively coupled plasma optical emission spectrophotometer (ICP-OES), model Perkin Elmer Optima 2100DV, was used to determine the heavy metals in the sample solutions. A scanning electron microscope combined with energy dispersive X-ray spectroscopy (SEM-EDX), make JEOL model JSM-6390LV, was used to record the EDX spectra.

**2.5. Analysis for Na, K, and Ca.** Concentration of Na and K in the soil samples was determined by using Labtronics digital flame photometer. Concentration of Ca was determined by titration method proposed by Piper [10].

**2.6. Elemental Analysis.** CHN analysis was done using Perkin Elmer 2400 CHN Analyzer. An inductively coupled plasma optical emission spectrophotometer (ICP-OES), model Perkin Elmer Optima 2100DV, was used to determine the heavy metals in the sample solutions. A scanning electron microscope combined with energy dispersive X-ray spectroscopy (SEM-EDX), make JEOL model JSM-6390LV, was used to record the EDX spectra.

For ICP-OES analysis solutions were prepared by wet digestion with nitric acid and sulfuric acid. 1 gm of the sample was taken in a 300 mL flat bottomed Kjeldahl digestion flask containing 10 mL of concentrated H<sub>2</sub>SO<sub>4</sub> and 10 mL of concentrated HNO<sub>3</sub>. The samples were then digested carefully over a hot plate at low heat (~60°C), avoiding excessive frothing until brown fumes cease to be evolved and much of the water is driven off. The flask is then allowed to cool. The samples were digested further with 10 ml of HNO<sub>3</sub> until white fumes of sulphuric acid are produced. The process is repeated until a clear and colourless digest is obtained. The resulting digest was heated by heating at 100°C for 3–5 minutes and cooled. The solution is then diluted with water until the concentration of the sample becomes about 1 ppm. About 10 mL of the solution is taken for analysis [10].

The ICP spectrometer is calibrated with three calibration standards and a blank (20% HNO<sub>3</sub>) each time it is used to measure the trace elements present in the digested soil samples. The concentrations of the calibration standards used are 0.2 ppm, 0.4 ppm, and 0.5 ppm (for Cr, Ni, and Cu), 50.0 ppm, 100.0 ppm, and 200.0 ppm (for Fe), 1.0 ppm, 3.0 ppm, and 5.0 ppm (for Zn), 0.6 ppm, 1.0 ppm, and 2.0 ppm (for Pb) and 0.02 ppm, 1.0 ppm, and 0.25 ppm (for Cd). Standard solutions were prepared in pyrex volumetric flask using Millipore deionized water and used within 24 hours.

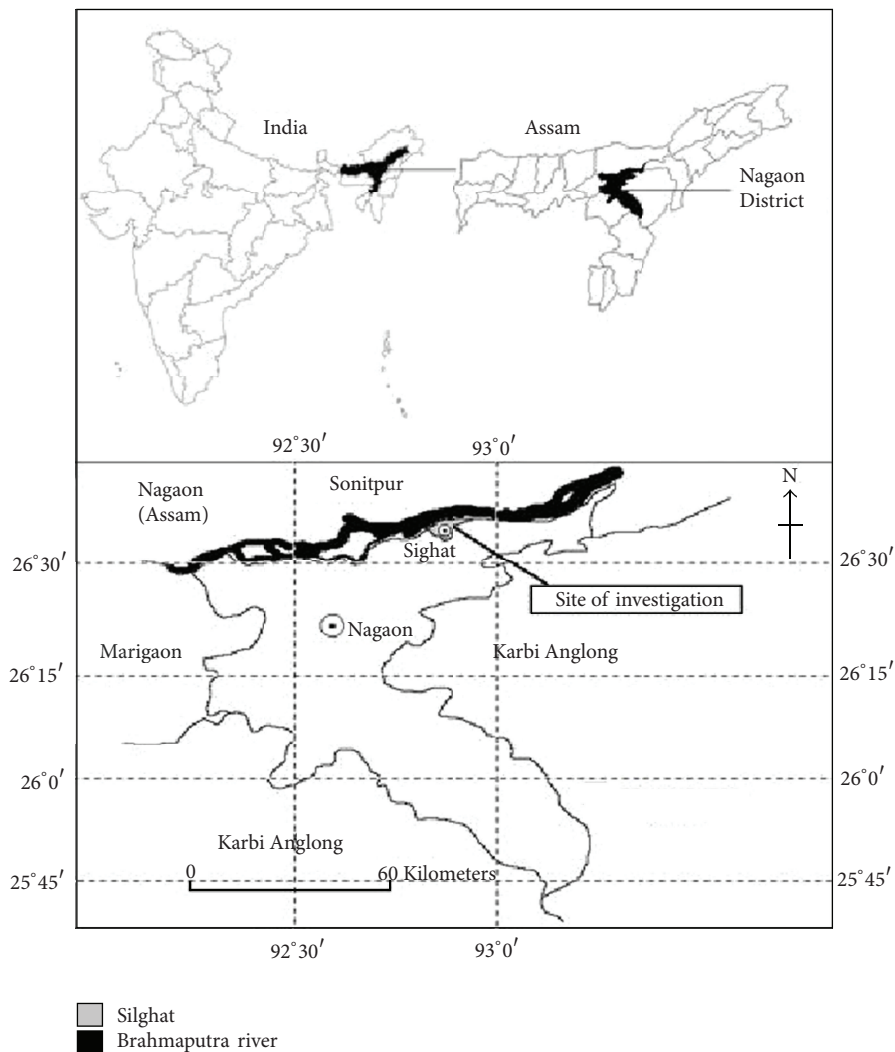


FIGURE 1: Map of Nagaon district of Assam (India) showing the Silghat region.

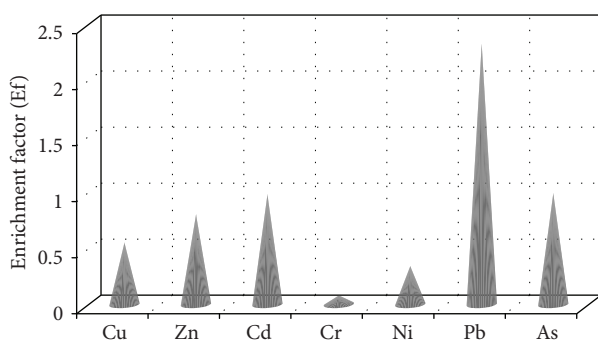


FIGURE 2: Enrichment factors for metals in the soil samples.

### 3. Results and Discussion

The physico-chemical parameters of the analyzed soil samples are shown in Table 1. The soil of the jute mill dumping yard was found to be slightly acidic with an average pH of

6.20. The acidic nature of the soil favours bioaccumulation and biomagnifications of heavy metals [11]. The electrical conductivity of the 1:5 soil suspensions was found to be in the range of 1.02 to 3.02 mS/cm. The high value of electrical conductivity indicates the presence of appreciable amount of ions in the soil samples [12]. Chloride contents of the samples were determined by rapid titration method [10]. Concentrations of sulfate and nitrate in the soil samples were determined according to the method proposed by Ben Mussa et al. [13] and Kolo et al. [14]. The average values of chloride, sulfate, and nitrate in the soil samples were found to be 10.26 mg/Kg, 5.80 mg/Kg, and 0.094 mg/Kg respectively, as shown in Table 2. High concentration of sulfates in the soil indicates anthropogenic influence [15]. The anion holding capacity of the soil increases with decrease in pH [13]. The random distribution of the anions can be, therefore, attributed to the unequal distribution of soil pH. Organic carbon content of the soil was determined by the Walkley and Black rapid titration method [16]. It was observed that the percentage of organic carbon content of the

TABLE 1: Physico-chemical parameters of the soil samples.

Sr. No.	Parameters	Soil Samples									
		S1	S2	S3	S4	S5	S6	S7	S8	S9	S10
(1)	pH	6.02	6.15	6.45	5.98	5.60	6.80	5.53	6.98	7.00	5.40
(2)	Electrical conductivity (mS/cm)	2.12	2.08	2.02	2.34	2.45	1.98	2.56	1.67	1.02	3.02
(3)	Chloride (mg/Kg)	12.50	8.30	7.80	12.80	13.20	7.30	13.56	6.80	6.30	14.01
(4)	Sulphate (mg/Kg)	6.56	5.34	5.01	7.02	7.12	4.23	7.34	4.01	3.84	7.50
(5)	Nitrate (mg/Kg)	0.09	0.06	0.04	0.12	0.16	0.04	0.18	0.03	0.02	0.20
(6)	C <sub>org</sub> (%)	4.02	3.42	3.23	4.67	4.84	3.10	4.90	3.08	3.02	5.02
(7)	C/N ratio	20.26	21.02	22.76	18.84	18.43	25.07	18.76	26.89	29.48	19.56
(8)	Na (mg/Kg)	84.56	67.34	76.34	65.34	88.78	63.67	75.45	65.45	65.43	96.45
(9)	K (mg/Kg)	90.23	88.76	87.65	79.05	84.35	78.34	93.23	96.43	87.65	112.45
(10)	Ca (mg/Kg)	295.62	256.67	234.45	223.67	298.87	213.89	254.89	234.89	225.76	302.45

TABLE 2: Statistics of the elements and physicochemical parameters in soil samples ( $n = 10$ ).

	pH	EC	C <sub>org</sub>	Cl <sup>-</sup>	SO <sub>4</sub> <sup>2-</sup>	NO <sub>3</sub> <sup>-</sup>	Na	K	Ca
Maximum	7.0	3.02	5.02	14.01	7.50	0.20	96.45	112.45	302.45
Minimum	5.4	1.02	3.02	6.30	3.84	0.02	63.67	84.35	223.67
Mean	6.20	2.12	3.93	10.32	5.80	0.09	74.88	89.81	254.12
Median	6.08	2.10	3.72	10.40	5.95	0.07	71.39	88.20	244.89
SD	0.56	0.51	0.80	3.10	1.39	0.06	10.98	9.25	31.89

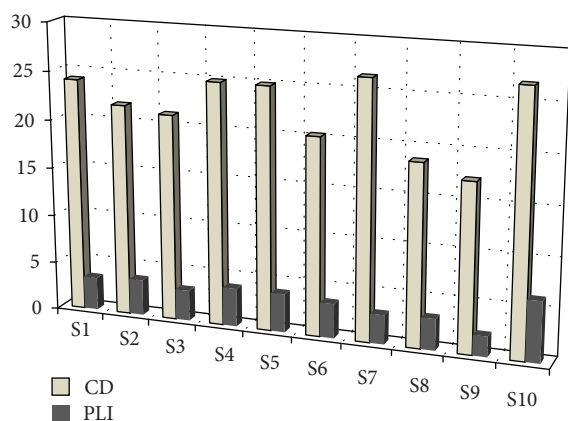


FIGURE 3: Contamination degree (CD) and pollution load index (PLI) of the analyzed soil samples.

soil samples varies from 3.02 to 5.02. The C/N ratio of the samples ranges from 18.43 to 29.48 with an average organic carbon content of 3.93%. The high C/N ratio is probably due to the biodegradation of jute fibers and cadies. The soil samples were found to be highly enriched with Na (average 84.05 mg/Kg), K (average 89.81 mg/Kg), and Ca (average 254.12 mg/Kg). For proper environmental monitoring and assessment, effective and correct method of data interpreting is necessary [17]. Statistical analysis like correlation studies can be used to effectively identify and assess the sources of contamination [11, 18, 19].

The linear correlation coefficient ( $r$ ) often referred to as the *Pearson product moment correlation coefficient* can be defined as [18]

$$r = \frac{n \sum xy - (\sum x)(\sum y)}{\sqrt{n(\sum x^2) - (\sum x)^2} \sqrt{n(\sum y^2) - (\sum y)^2}}, \quad (1)$$

where  $n$  is the number of pairs of data.

The value of  $r$  is such that  $-1 \leq r \leq +1$ . The + and - signs are used for positive linear correlations and negative linear correlations, respectively. If  $x$  and  $y$  have a strong positive linear correlation,  $r$  is close to +1. An  $r$  value of exactly +1 indicates a perfect positive fit. Positive values indicate a relationship between  $x$  and  $y$  variables such that as values for  $x$  increase, values for  $y$  also increase. If  $x$  and  $y$  have a strong negative linear correlation,  $r$  is close to -1. An  $r$  value of exactly -1 indicates a perfect negative fit. Negative values indicate a relationship between  $x$  and  $y$  such that as values for  $x$  increase, values for  $y$  decrease. If there is no linear correlation or a weak linear correlation,  $r$  is close to 0. A value near zero means that there is a random, nonlinear relationship between the two variables [11, 19, 20].

Pearson correlation (PC) matrix for the analyzed physico-chemical parameters of the soil as influenced by jute mill solid waste is presented in Table 3. From the calculated matrix strong positive correlation of electrical conductivity was observed with chloride content ( $r = 0.856$ ) and organic carbon content ( $r = 0.841$ ) of the soil samples. pH showed negative correlation with electrical conductivity ( $r = -0.899$ ).

A strong positive correlation was observed between Na and K ( $r = 0.603$ ) and between Na and Ca ( $r = 0.916$ ). This may be due to significant geochemical association of Na with K and Ca [20, 21]. The geochemical association of Na-K-Ca

TABLE 3: Correlation coefficient of physico-chemical parameters of soil as influenced by jute mill solid waste.

	E.C.	pH	Cl <sup>-</sup>	C <sub>org</sub>	C/N	Na	K	Ca
E.C.	1							
pH	-0.899	1						
Cl <sup>-</sup>	0.856	-0.944	1					
C <sub>org</sub>	0.841	-0.939	0.976	1				
C/N	-0.881	0.929	-0.878	-0.853	1			
Na	0.687	-0.748	0.729	0.669	-0.583	1		
K	0.402	-0.358	0.328	0.308	-0.098	0.603	1	
Ca	0.609	-0.740	0.708	0.635	-0.605	0.916	0.553	1

TABLE 4: Heavy metal concentration (mg/Kg) in soil samples—SEM EDX, ICP-OES data.

Metal	Soil samples									
	S1	S2	S3	S4	S5	S6	S7	S8	S9	S10
Fe	1790.87	1645.23	1534.11	2020.34	2106.67	1500.12	2205.34	1467.23	1400.11	2269.14
Cu	84.63	80.62	79.57	85.87	86.88	76.45	88.89	70.11	68.23	89.91
Zn	212.09	200.11	198.23	216.09	220.09	196.11	221.67	190.11	176.22	222.12
Cd	9.67	8.34	8.00	9.69	9.71	7.67	10.06	7.01	6.86	10.12
Cr	9.56	8.40	7.34	9.70	9.85	6.12	9.90	5.02	9.96	10.02
Ni	3.45	2.12	2.01	3.57	3.65	1.98	4.02	0.96	0.43	4.10
Pb	112.98	100.11	98.06	114.13	116.23	84.53	123.11	79.67	67.83	125.12
As	23.14	19.12	16.02	24.14	25.26	15.11	25.99	14.03	13.01	26.14

in the soil shows that these metals are deposited from anthropogenic sources, since there is no known geogenic source which can contribute to this type of association in the study area [19].

The soil samples were analyzed for eight different heavy metals. The SEM-EDX and ICP-OES data for the heavy metal concentration is shown in Table 4. The concentration of heavy metals in the soil samples was found in the order Cr < Cd < As < Cu < Pb < Zn < Fe. The average values of the metal concentration are Fe (1793.90 mg/Kg), Cu (81.12 mg/Kg), Zn (205.28 mg/Kg), Cd (8.71 mg/Kg), Cr (8.08 mg/Kg), Ni (2.63 mg/Kg), Pb (102.17 mg/Kg), and As (20.20 mg/Kg). The concentration of Fe, Cu, Cd, Zn, and Pb was found to be very much higher than both the continental crust and world surface rock average values as shown in Table 5 [20, 22, 23]. Concentration of Ni and Cr was found to be much below that of the two reference values [24, 25]. This clearly indicates the excessive use of chemicals in the industrial stages of the jute mill. It may be noted that there is practically no human activity in the dumping yard of the mill except an annual religious fair that took place in April every year and lasted for about 10–15 days. Use of chemical fertilizers in some part of the abandoned yard for agricultural purpose may be one of the sources of heavy metals like Zn. The average concentration of As in the control soil was found to be 10 mg/Kg and within the range of 2.0–16.3 mg/Kg which is quite close to that observed in the investigated region (20.20 mg/Kg). This indicates that the concentration of As in the investigated region may be attributed to geogenic factor rather than anthropogenic factors. Uncontaminated soils usually contain 1–40 mg kg<sup>-1</sup> of arsenic, with lowest

concentrations in sandy soils and those derived from granites, and higher concentrations in alluvial and organic soils [19, 26]. Therefore, the investigated soils can be regarded as uncontaminated by arsenic, as the pseudo-total amount of As was within the range 13.01–26.14 mg kg<sup>-1</sup>. Among the different oxidation states of As, arsenite As(III) and arsenate As(V) are the main inorganic forms in most contaminated soils and sediments. In oxygen-rich environments and well-drained soils, As(V) species dominate and under reducing conditions As(III) is the stable oxidation state [26].

Pesticides and fertilizers used in the cultivation of jute crops which serve as the raw material of the mill can be a potent source of these heavy metals. However, the industrial activity of the jute mill can be considered as the principal source of the heavy metal contamination in the region.

Correlation coefficient of physico-chemical parameters of soil with heavy metals in soil as influenced by jute cadies is shown in Table 6. All the heavy metals showed positive correlation with electrical conductivity, chloride content and organic carbon content. These strong positive correlations which are statistically significant indicate an intrinsic relation between them [21–23]. The positive correlation between the heavy metals and chloride content is probably due to the industrial use of metal chlorides in various stages of processing jute yarns and producing the finished products. However negative correlation was observed for all the metals with pH and C/N ratio. Among the metals Zn showed the highest positive correlation with electrical conductivity ( $r = 0.939$ ). Pearson coefficient of 0.985 was observed for As with chloride content indicating that levels of As are highly dependent on chloride content [23, 24].

TABLE 5: Statistics of the determined element in soil samples ( $n = 10$ ) and reference value of element concentration in the continental crust and world surface rock average.

	Fe	Cu	Zn	Cd	Cr	Ni	Pb	As
Maximum	2269.14	89.91	222.12	10.12	10.02	4.10	125.12	26.14
Minimum	1400.11	68.23	176.22	6.86	4.96	0.43	67.83	13.01
Mean	1793.90	81.12	205.28	8.71	8.08	2.63	102.17	20.20
Median	1718.05	82.62	205.28	8.71	8.40	2.63	102.17	20.20
SD	302.26	7.58	14.05	1.15	1.87	1.18	17.68	4.79
Reference value								
(i) continental crust	—	25.0	65.0	0.2	126.0	76.0	15.0	—
(ii) World surface rock average	3.59	32.0	129.0	—	97.0	49.0	20.0	—

TABLE 6: Correlation coefficient of physico-chemical parameters of soil with heavy metals in soil as influenced by jute mill solid waste.

	Fe	Cu	Zn	Cd	Pb	Cr	Ni	As
E.C.	0.885	0.933	0.939	0.877	0.929	0.860	0.919	0.868
pH	-0.956	-0.968	-0.948	-0.962	-0.971	-0.964	-0.948	-0.972
Cl <sup>-</sup>	0.967	0.934	0.953	0.981	0.947	0.935	0.965	0.985
O.C.	0.992	0.905	0.933	0.945	0.915	0.899	0.930	0.971
C/N	-0.859	-0.967	-0.955	-0.942	-0.965	-0.975	-0.944	-0.930

3.1. *Enrichment Factor (Ef)*. The Enrichment factor is convenient tool to assess the degree of heavy metal concentration in soil and sediments. It can be expressed as [22, 24]

$$Ef = \frac{[(C_x/Fe)_{\text{sample}}]}{[(C_x/Fe)_{\text{background}}]}, \quad (2)$$

where  $C_x$  is the concentration of metal “ $x$ ” (mg/Kg) in the contaminated soil. The background value is that of the world surface rock average as shown in Table 4. Fe was taken as a normalizing element because of its geochemical abundance in the investigated region and since redox sensitive hydroxides and oxides of iron have a controlling influence on heavy metal distribution.

The Ef for metals in the soil of Silghat jute mill dumping site is shown in Figure 2. It has been observed that there is a significant difference in Ef values among metals. When Ef value exceeds 1.0, it indicates higher availability and distribution of metals in contaminated soil, thereby, increasing the metal accumulation in plant species grown on the contaminated soil [23]. It has been observed that the Ef for Pb (2.32) exceeds the limiting value of 1.0 as shown in Figure 3. The Ef for As (0.99) and Cd (0.98) is almost unity. Ef value of the metals in the soil samples increases in the order, Cr < Ni < Cu < Zn < Cd < As < Pb.

3.2. *Contamination Degree (CD)*. The assessment of overall contamination of the soil was attempted based on the degree of contamination (CD). The sum of the contamination factors (Cf) of all elements examined represents the contamination degree (CD) of the environment [19]. Contamination factor (Cf) =  $C_s/C_b$ , where  $C_s$  is the concentration of the metal in the soil samples and  $C_b$  is the background value for the metal taken from average shale (here world surface rock average was used). “ $m$ ” represents multiple metals, that is, all the metals

analyzed in this study. The calculated CD values are shown in Figure 3. It was observed that the CD for all the ten soil samples are quite high and within the range of 17–26 which falls in the scale  $14 \leq CD \leq 28$  indicating considerable degree of contamination. CD value was observed to be highest for S10 and minimum for S9.

3.3. *Pollution Load Index (PLI)*. Pollution load index (PLI) for a particular sampling site has been evaluated following the method proposed by Tomlinson et al. [25]. The PLI represents the number of times by which the metal concentration in the soil exceeds the average natural background concentration and gives a summative indication of the overall level of heavy metal toxicity in a particular sample. [When the PLI values exceed 1.0, the soil can be contaminated by anthropogenic inputs and recommends continuous environmental monitoring of the area]. The calculated PLI values of the ten samples are presented in Figure 3. It was observed that the PLI value of the samples ranges from 2.10 to 6.26 against the limiting value of 1.0 indicating comprehensive metal contamination of the soil in the dumping yard of the jute mill.

## 4. Conclusion

The results of the present investigation clearly indicate the adverse effect of jute cadies and waste generated from Assam Co-operative Jute Mills LTD of Silghat on the soil quality of the region. The physico-chemical analysis indicates that the soils has the potential to accumulate heavy metal load which have a tendency to biomagnify and induce long-term adverse environmental impact. The soil of the region is highly contaminated with heavy metal which is possibly due to the long-term deposit of the solid waste generated in the local jute mill. The present study indicates that the untreated jute mill solid waste has adversely affected the soil quality of the region

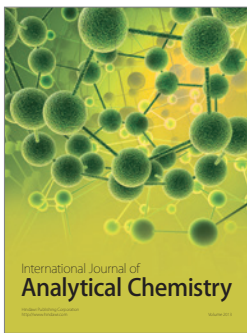
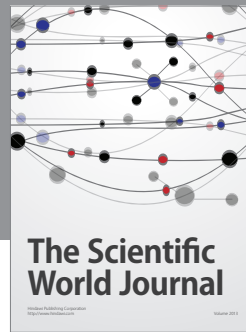
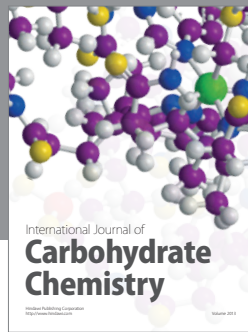
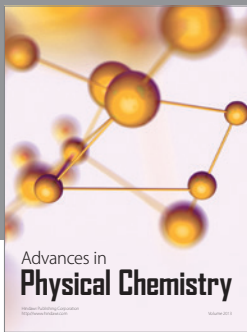
and necessitates periodic monitoring and assessment of the soil and plants of the region.

## Acknowledgments

The authors are thankful to Bornali Barooah and Biren Gohain for CHN analysis in the Department of Chemical Sciences, Tezpur University, Assam, India.

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