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Characterization of Urban Soil with SEM-EDX

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Abstract

The coal is largely used for generation of energy in the India, and their huge exploitations cause contamination of the soil. In the present work, the inorganic contamination of surface soil in the coal basin of the country, Korba is described. The concentration of elements *i.e.* C, O, S, Cl, Na, Mg, Al, Si, P, K, Ca, Ti, Fe, Mn and Ni in the surface soils (n = 30) was analysed by technique *i.e.* scanning electron microscope-energy dispersive X-ray spectroscopy (SEM-EDX), ranged from 6.6% - 36.4%, 37.8% - 54.3%, 0.07% - 0.39%, 0.03% - 0.25%, 0.10% - 0.39%, 0.12% - 0.49%, 3.7% - 10.3%, 12.2% - 24.4%, 0.11% - 0.60%, 0.68% - 3.22%, 0.8% - 2.7%, 0.25% - 0.61%, 2.3% - 6.4%, 0.08% - 0.22% and 0.04% - 0.16% with mean value ($p = 0.05$) of 18.8% ± 2.7%, 49.0% ± 1.5%, 0.18% ± 0.03%, 0.11% ± 0.02%, 0.23% ± 0.03%, 0.32% ± 0.03%, 6.1% ± 0.5%, 16.9% ± 0.9%, 0.27% ± 0.04%, 1.6% ± 0.2%, 1.5% ± 0.2%, 0.41% ± 0.03%, 4.3% ± 0.4%, 0.14% ± 0.02% and 0.08% ± 0.01%, respectively. The surface soil morphology, concentration variation of elements, pollution indices and contaminant sources in the soil are discussed.

Keywords

Urban Soil, Element, Characterization, Pollution Indices, Sources, SEM-EDX

1. Introduction

Pollution of urban soil is of a great public health interest due to receiving of large amounts of heavy metals from multiple sources including industrial wastes, vehicle emissions, coal and biomass burnings, etc. [1]-[6]. India is the third-largest producer of coal in the World. It is a naturally occurring combustible materials contains elemental carbon, sulfur, hydrocarbons, trace metals, etc. [7] [8]. Several millions ton of coal is mined out and burnt in the Korba basin for generation of electricity by pouring the effluents into the environment. The whole environment of the basin is covered by the fly ash and black carbon. The air, rain and groundwater were contaminated with higher content of acids and metals [9]-[13]. The SEM-EDX is a versatile, accurate and reproducible technique to quantify the major elements on the surface of geomedia [14]. Hence, in this work, the soil contamination of the Korba basin with elements *i.e.* C, O, S, Cl, Na, Mg, Al, Si, P, K, Ca, Ti, Fe, Mn and Ni is investigated by using technique *i.e.* SEM-EDX. The concentration variations, enrichments and sources of the elements in the soil are discussed.

2. Material and Methods

2.1. Study Area

Korba coalfield (22.35˚N and 82.68˚E) is located in the Chhattisgarh state, India in the basin of the Hasdeo river, extending over ≈500 km^2. The basin is covered with coal and forest where ≈0.5 million population are residing. Several coal mines are in operation with annual production of ≈3 BT coal (non-cooking) annually. Their huge amounts (≈20 MT/Yr) are consumed by various units of thermal power plants to produce ≈6000 MW electricity with emission of ≈6 MT ash into the environment.

2.2. Collection of Samples

The soil samples were collected from 30 locations of Korba area lie over ≈500 km^2 areas (**Figure 1**). One kilogram of surface soil sample from each site (0 - 10 cm) was collected in a clean polyethylene container during January, 2011 as prescribed in the literature [15]. For depth profile study, the samples were collected at depth of 0 - 10, 10 - 20 and 20 - 30 cm at location no. 1 and 12.

2.3. Sample Preparation

The soil samples were dried, milled and sieved out particles of ≤1 mm by drying at 60˚C for 3 days in an oven to remove moisture. A 5.0 g soil sample was mixed with 25 mL deionized water in the 100 mL conical flask. The sample was agitated for 6 hrs in the ultrasonic bath. The pH value of the extract was measured by Hanna pH meter type -HI991300.

2.4. Analysis

A Hitachi SU-6600 Scanning Electron Microscope equipped with the Thermo dispersive X-ray spectrometer (EDS) and cathode luminescence (CL) detector was used for characterization of major elements (*i.e.* O, S, Cl, Na, Mg, Al, Si, P, K, Ca, Ti, Fe, Mn and Ni) in the soil at the Maria Sklodowska-Curie University, Lublin, Poland.The sample was exposed with the X-ray in a polyethylene disc with recording of surface soil photograph and analytical peaks. The mean value of three measurements at different locations of the dried soil sample is summarized in **Table 1** and **Table 2**. The standard soil sample (NCS DC 73382˚C RM) was used for the quality control.

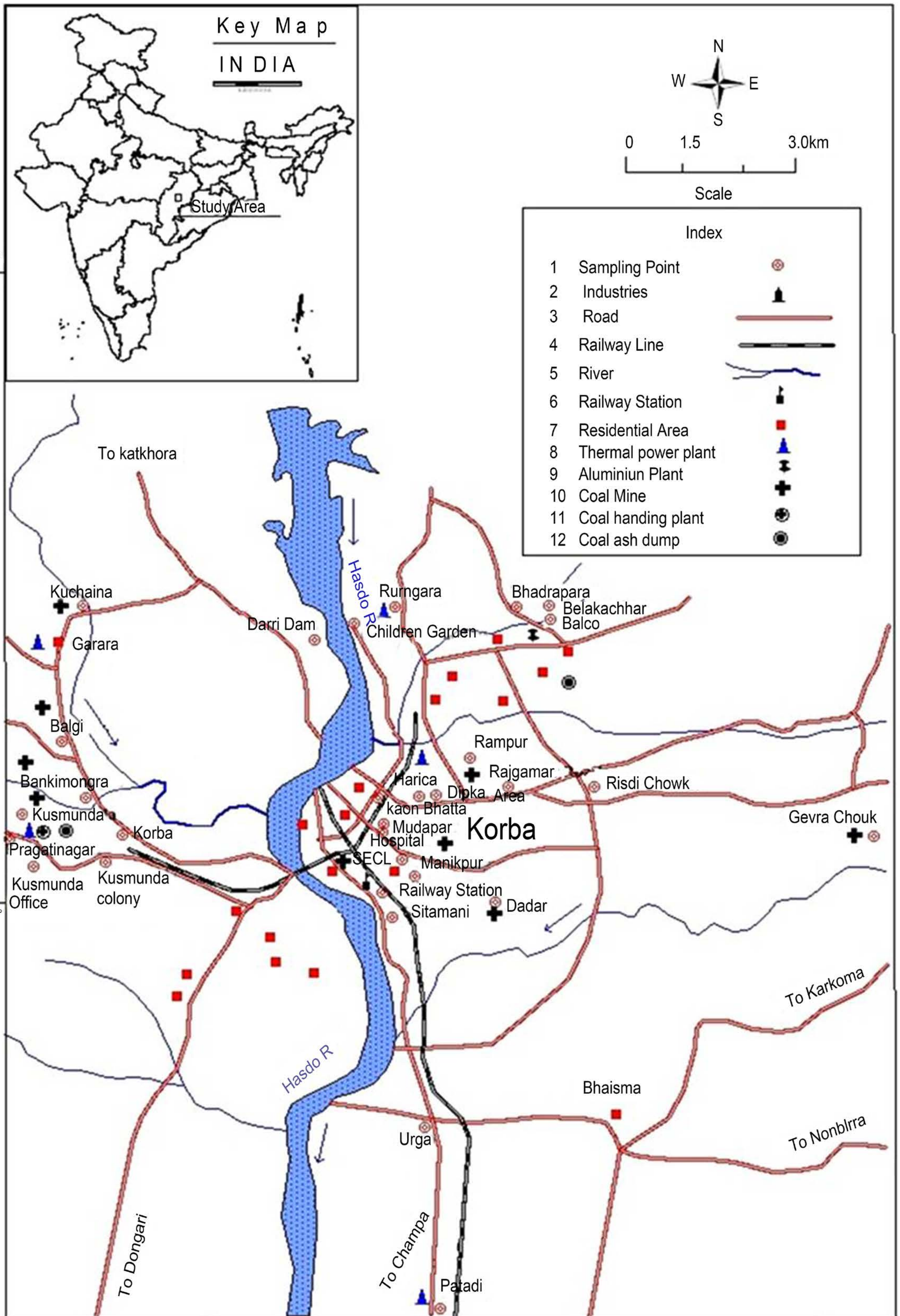


Figure 1. Representation of sampling locations in Korba basin, India.

Table 1. Physico-chemical characteristics of soil.

S. No.	Location	Color	ML	pH	C%	O%	S%	Cl%
1	Niharica	W	IHG	6.98	10.8	52.3	0.19	0.17
2	Kuan Bhatta	W	HG	6.95	22.9	45.4	0.28	0.07
3	Railway Station	B	IHG	6.99	22.0	50.4	0.09	0.05
4	Sitamani	BW	IHG	6.86	13.1	50.1	0.26	0.11
5	Rajgamar	BW	IHG	6.94	21.3	48.7	0.31	0.16
6	Rumgara	W	IHG	6.94	12.2	53.1	0.11	0.09
7	Darri dam	BW	IHG	6.44	19.4	48.8	0.18	0.05
8	Chaildren Garden	BW	HG	6.88	13.4	52.5	0.12	0.04
9	Dipka Area	W	IHG	6.52	32.0	46.4	0.13	0.08
10	Pragati Nagar	BW	IHG	6.94	31.3	45.7	0.09	0.09
11	Gevera Chowk	W	IHG	6.65	16.2	52.3	0.13	0.06
12	Kusmunda-I	B	IHG	6.80	24.7	42.8	0.08	0.07
13	Kusmunda-II	W	IHG	6.65	14.2	53.0	0.07	0.08
14	Kusmunada-III	B	IHG	6.68	15.3	53.8	0.17	0.15
15	Korba	BW	IHG	5.37	10.0	51.8	0.26	0.17
16	Bankimongra	BW	IHG	6.47	14.3	54.3	0.17	0.05
17	Balgi	W	IHG	7.03	21.1	48.5	0.25	0.16
18	Kuchaina	W	IHG	6.75	16.0	49.2	0.35	0.12
19	Balco	BW	IHG	5.7	36.4	37.8	0.32	0.13
20	Bhadrapara	B	IHG	6.96	21.2	51.2	0.13	0.11
21	Risdi Chowk	BW	IHG	6.65	30.0	46.1	0.21	0.18
22	Manikpur	BW	IHG	6.61	6.6	54.3	0.11	0.25
23	Dadar	BW	IHG	6.85	22.3	43.6	0.11	0.03
24	SECL-I	W	IHG	6.37	11.3	50.0	0.19	0.13
25	Mudapar	W	HG	6.37	17.6	49.0	0.12	0.04
26	Rampur	BW	IHG	5.78	31.6	43.3	0.18	0.07
27	SECL-II	BW	IHG	6.16	15.5	40.3	0.19	0.15
28	Belakachar	BW	IHG	6.12	14.4	52.7	0.18	0.16
29	Urga	B	IHG	7.12	10.6	48.2	0.12	0.11
30	Patadi	B	IHG	7.54	15.0	53.5	0.39	0.21

ML = Morphology, B = Blackish, BW = Blackish-white, W = Whitish, HG = Homogenous, IHG = Inhomogeneous, SECL = South east coal Ltd.

2.5. Pollution Indices

The pollution indices *i.e.* enrichment factor (EF), contamination factor (CF) and pollution load index (PLI) were used to determine elemental enrichment in the soil with respect to the base line concentration. The concentration ratio of an element, X, to a

Table 2. Concentration of trace elements in soil, %.

S. No.	Na	Mg	Al	Si	P	K	Ca	Ti	Fe	Mn	Ni
1	0.39	0.33	6.09	19.16	0.34	1.27	1.87	0.57	4.81	0.17	0.07
2	0.13	0.24	5.61	16.45	0.25	0.99	1.53	0.46	4.31	0.11	0.04
3	0.11	0.31	4.43	12.78	0.2	1.02	0.86	0.25	2.32	0.08	0.06
4	0.25	0.36	6.22	19.09	0.39	2.13	1.63	0.41	4.75	0.18	0.08
5	0.37	0.48	6.79	24.42	0.46	2.57	2.34	0.61	6.4	0.21	0.14
6	0.21	0.28	5.96	17.37	0.33	2.01	1.87	0.5	4.71	0.18	0.07
7	0.24	0.25	5.23	15.53	0.18	0.92	0.84	0.38	2.77	0.09	0.04
8	0.12	0.26	5.9	13.77	0.26	0.74	0.9	0.44	4.05	0.08	0.05
9	0.26	0.17	5.42	16.4	0.12	0.82	0.84	0.35	3.88	0.10	0.12
10	0.17	0.23	5.14	14.04	0.28	1.77	1.48	0.48	4.74	0.21	0.06
11	0.33	0.27	5.36	18.4	0.26	1.49	0.83	0.45	4.77	0.16	0.05
12	0.14	0.44	6.09	15.99	0.29	1.29	1.41	0.34	3.02	0.12	0.04
13	0.16	0.33	5.09	17.26	0.21	1.7	1.57	0.37	3.95	0.09	0.05
14	0.35	0.38	9.5	16.15	0.23	1.37	1.64	0.48	5.78	0.16	0.06
15	0.36	0.49	7.36	17.66	0.37	2.18	1.57	0.58	5.15	0.21	0.12
16	0.18	0.27	5.98	17.37	0.16	1.41	1.33	0.25	3.81	0.12	0.08
17	0.25	0.33	8.49	15.42	0.18	1.24	1.26	0.33	3.04	0.11	0.07
18	0.11	0.28	5.21	17.57	0.17	1.69	1.39	0.36	3.44	0.12	0.05
19	0.26	0.38	4.53	12.86	0.29	1.95	1.65	0.56	4.06	0.19	0.09
20	0.13	0.35	3.68	19.15	0.16	1.71	1.58	0.32	3.96	0.17	0.06
21	0.19	0.32	4.76	13.97	0.11	0.68	1.65	0.36	3.25	0.18	0.06
22	0.27	0.28	7.59	14.59	0.21	1.26	1.47	0.31	5.94	0.11	0.07
23	0.17	0.29	5.27	16.2	0.29	2.17	1.01	0.41	5.91	0.09	0.07
24	0.1	0.26	5.85	17.86	0.35	1.06	1.63	0.41	4.41	0.22	0.16
25	0.24	0.35	6.19	17.09	0.26	1.84	1.11	0.36	4.99	0.1	0.05
26	0.27	0.12	4.56	12.19	0.16	1.35	1.27	0.31	2.42	0.12	0.04
27	0.31	0.31	9.85	17.61	0.34	1.6	2.3	0.46	5.63	0.18	0.16
28	0.19	0.33	5.44	17.88	0.30	1.86	0.85	0.32	4.53	0.11	0.05
29	0.21	0.32	6.19	20.34	0.60	1.62	2.17	0.35	4.26	0.12	0.10
30	0.36	0.48	10.31	22.31	0.49	3.22	2.72	0.53	5.22	0.21	0.15

crustal element (e.g. Al) in the soil sample to the {[X]/[Al]} ratio in the earth's crust was normalised by using following indices [16] [17].

$EF = \{[X_s]/[Al_s]\}/\{[X_e]/[Al_e]\}$

$CF = \{[X_s]/[X_e]\}$

$PLI = (CF_1 \times CF_2 \times CF_3 \times CF_4 \ldots\ldots\ldots CF_n)^{1/n}$

Where, symbols: X_s, X_e, Al_s and Al_e denote concentration of metal and Al in the soil and earth crust, respectively.

3. Results and Discussion

3.1. Soil Properties

Soils are complex assembles of minerals, organic material, living organisms, water, and gases. The size, shape, and chemical composition of soil minerals are highly variables [18]. Scanning electron microscopy coupled with energy dispersive X-ray spectroscopy (SEM/EDX) is the versatile surface analytical technique. High resolution images of surface topography of three soil samples is shown in **Figure 2**. All the soils are composed

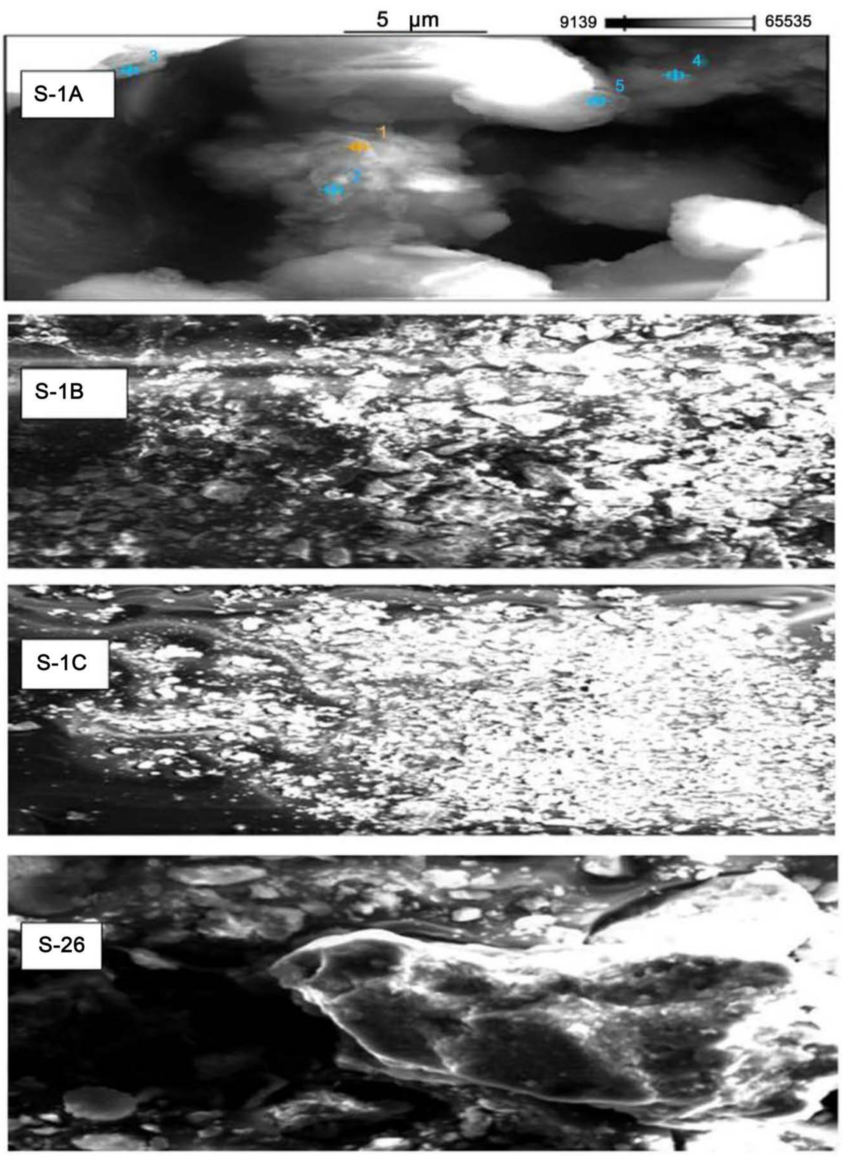
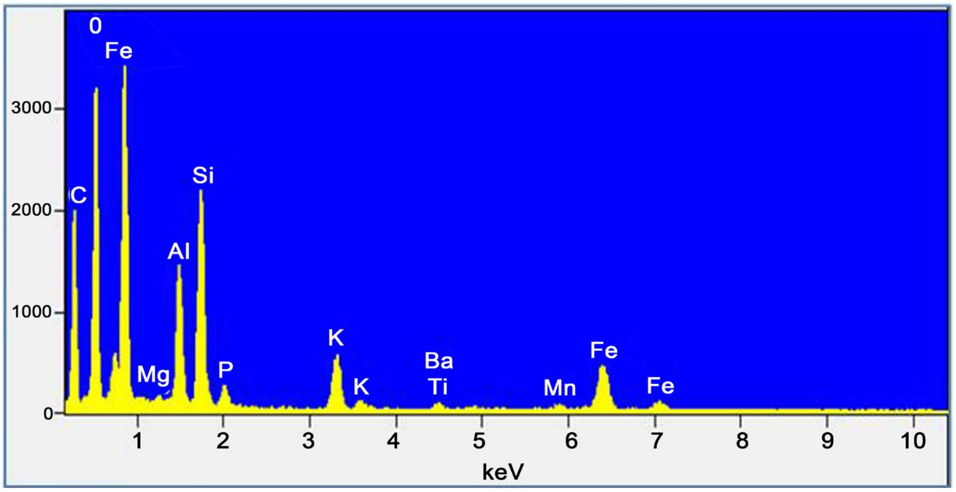


Figure 2. The SEM photograph of surface soil, no. S-1 (A), S-1 (B), S-1 (C) and S-26. A = 0 - 10 cm, B = 10 - 20 cm, C = 20 - 30 cm.

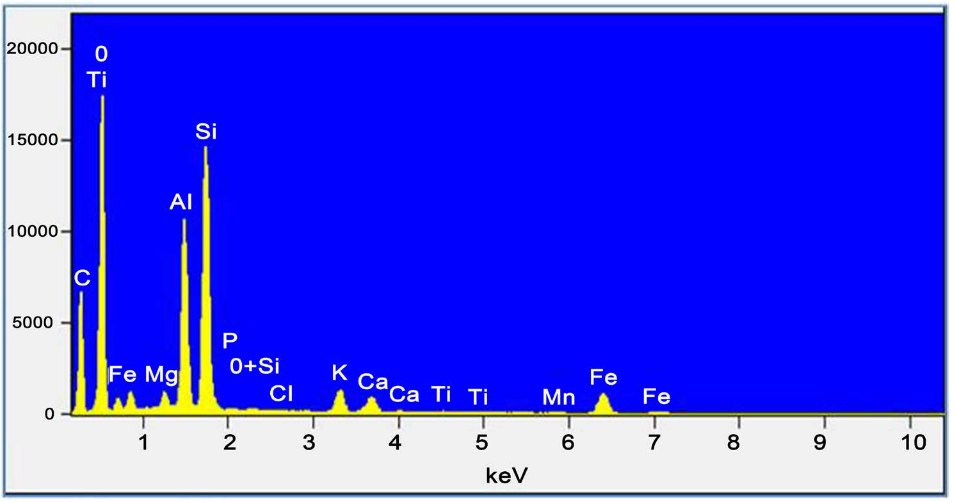
of micrometer-scale grains which consist of smaller particles with nano-scale structure. The soil was colored, ranging from whitish to blackish due to deposition of the fly ash and black carbon, Table 1. The particle sizes of all soil samples except S-2, S-8 and S-25 were observed to be more inhomogenous, see Table 1 and Figure 2. For all of the samples, the area of soil particles was generally in the range of 50 to 500 μm^2. The pH value of the soil extract was ranged from 5.4 - 7.5 with mean value (p = 0.05) of 6.6 ± 0.2. The soil extract was found to be slightly acidic, may be due to presence of higher content of chloride and sulfate. The lowest pH value of the soil was observed in the Korba city, may be due to the vehicular emissions.

3.2. Distribution of Major Elements

The EDX peaks of elements in the soil samples are shown in Figure 3. The concentration of elements *i.e.* C, Si, P, O, S and Cl in the surface soil is presented in Table 2. The



(a)



(b)

Figure 3. EDX peaks of elements in surface soil no 12 (a) and 16 (b).

C and O content of surface soil (n = 30) was ranged from 6.6% - 36.4% and 37.8% - 54.0% with mean value (p = 0.05) of 18.8% ± 2.7% and 49.0% ± 1.5%, respectively. The C and O content was found to be slightly higher than recommended value of 0.03 and 46.6%, respectively [19]. A negative correlation of the C with the O-content was marked, may be due to reduction of O-content with the C (r = −0.72). The concentration of Si, P, S and Cl in the soil (n = 30) was ranged from 12.2% - 24.4%, 0.11% - 0.60%, 0.07% - 0.39% and 0.03% - 0.25% with mean value (p = 0.05) of 16.9 ± 0.9, 0.27% ± 0.04%, 0.18% ± 0.03% and 0.11% ± 0.02%, respectively. Among them, the content of P, S and Cl was observed to be much higher than the base line value reported of 0.065%, 0.006% and 0.037%, respectively.

3.3. Distribution of Metals

The elevated concentration of 9 metals *i.e.* Na, K, Mg, Ca, Al, Ti, Fe, Mn and Ni in the soil was observed, ranging (n = 30) from 0.10% - 0.39%, 0.68% - 3.22%, 0.12% - 0.49%, 0.8% - 2.7%, 3.7% - 10.3%, 0.25% - 0.61%, 2.3% - 6.4%, 0.08% - 0.22% and 0.04% - 0.16% with arithmetic mean (AM) value (p = 0.05)of 0.23 ± 0.03, 1.6 ± 0.2, 0.32 ± 0.03, 1.5 ± 0.2, 6.1% ± 0.5%, 0.41% ± 0.03%, 4.3% ± 0.4%, 0.14% ± 0.02% and 0.08% ± 0.01%, respectively, **Table 2**. The metal concentration in the surface soil of the Korba basin was found higher than other regions of the country, probably due to huge coal mining and burning [4] [5] [6].

3.4. Variations and Sources

The sum of total content of 11 elements (*i.e.* Na, Mg, Al, Si, P, K, Ca, Ti, Fe, Mn and Ni) in the soil (n = 30) was ranged from 25.4% - 75.9% with mean value of 47.3% ± 3.9%. The higher metal contents (≥350 g/kg) were observed in sample no. 14 - 15, 20, 22 - 25 and 28 - 30 due to higher effluent emissions from point sources *i.e.* thermal power plant and coal mine. The value of arithmetic mean (AM), geometrical mean (GM) and median value for 15 elements (*i.e.* C, O, S, Cl, Na, Mg, Al, Si, P, K, Ca, Ti, Fe, Mn and Ni) was found to be comparable with very high variations, ranging from 8.8% - 50.7%, **Table 3**. The skewness and kurtosis for a normal distribution of data is 0 and 3, respectively. The value of skewness and kurtosis was ranged from −0.87 −1.29 and −1.43 −1.31, respectively. Fifteen elements was found to be distributed in the asymmetric order on the soil surface.

The correlation coefficient matrix for 14 elements is presented in **Table 4**. A poor correlation of elements among themselves was observed, may be due to their deposition in the soil by multiple sources *i.e.* coal mining and burning, atmospheric deposition of fly ash and dust, etc.

3.5. Vertical Distribution

The homogeneity and whiteness of the soil was increased as the depth of soil profile was increased from 0 - 30 cm, may be due to down movement of the ash, **Figure 2**. The vertical distribution of elements with respect to soil depth profile was studied and

Table 3. Concentration statistics of elements in surface soil, %.

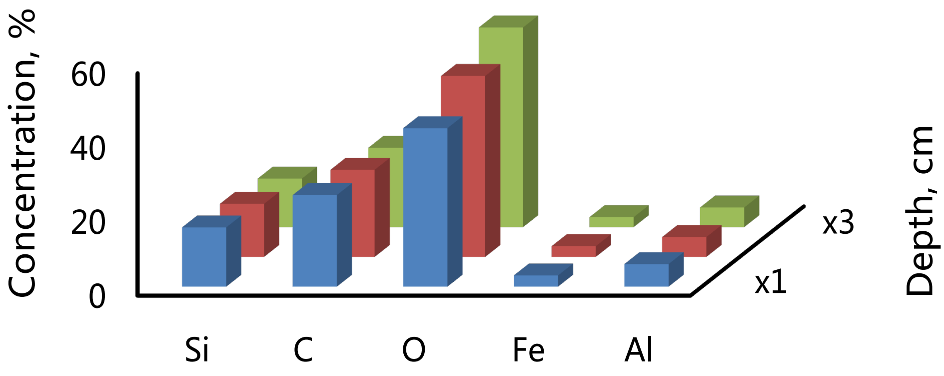
Element	Range	CL, ±	AMV	GMV	Median	RSD, ±	*Ku*	*Skw*
C	6.6 - 36	2.7	18.8	17.4	16.1	40	-0.18	0.75
O	38 - 54	1.50	49	49	50	9	0.20	-0.87
S	0.07 – 0.39	0.03	0.18	0.17	0.18	47	-0.10	0.83
Cl	0.03 -0.25	0.02	0.11	0.10	0.11	50	-0.32	0.52
Na	0.10 - 0.39	0.03	0.23	0.21	023	38	-0.99	0.30
Mg	0.12 -0.49	0.03	0.32	0.30	0.32	26	0.65	0.23
Al	3.7-10.3	0.50	6.1	5.8	5.9	26	1.31	1.29
Si	12.2-24.4	0.90	16.9	16.7	17.2	16	1.08	0.61
P	0.11-0.60	0.04	0.27	0.25	0.26	42	1.25	0.99
K	0.68 - 0.22	0.20	1.6	1.5	1.6	35	1.22	0.80
Ca	0.8 -2.7	0.20	1.5	1.4	1.5	32	0.29	0.59
Ti	0.25 -.61	0.04	0.41	0.40	0.40	24	-0.61	0.41
Fe	2.3 - 6.4	0.40	4.3	4.2	4.4	25	-0.50	-0.08
Mn	0.08 - 0.22	0.01	0.14	0.13	0.12	29	-1.43	0.25
Ni	0.04 - 0.16	0.01	0.08	0.07	0.07	50	0.35	1.20

AMV = Arithmetic mean value, GMV = Geometrical mean value, RSD = Relative standard deviation, CL = Confidence limit at 95% probability, Ku = Kurtosis, Sk = Skewness.

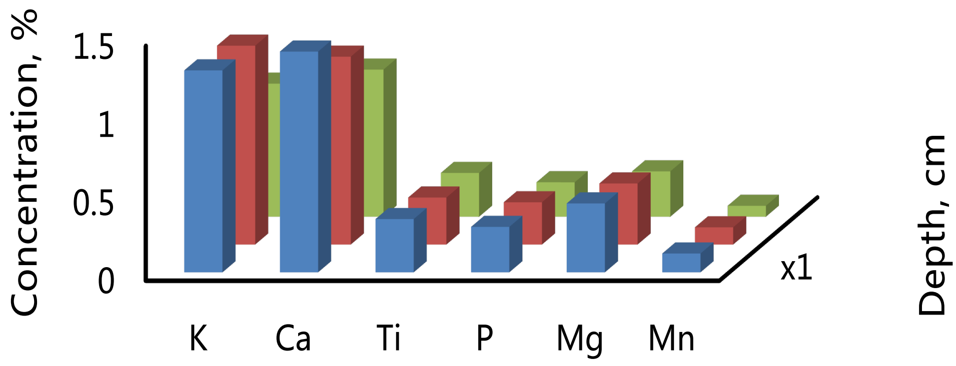
Table 4. Correlation coefficient matrix of elements.

	C	S	Cl	Na	Mg	Al	Si	P	K	Ca	Ti	Fe	Mn	Ni
C	1.00													
S	0.01	1.00												
Cl	0.22	0.46	1.00											
Na	0.24	0.34	0.49	1.00										
Mg	0.14	0.38	0.44	0.38	1.00									
Al	0.19	0.33	0.54	0.58	0.44	1.00								
Si	0.12	0.35	0.29	0.38	0.53	0.35	1.00							
P	0.42	0.25	0.26	0.34	0.52	0.40	0.65	1.00						
K	0.38	0.43	0.27	0.40	0.61	0.33	0.61	0.61	1.00					
Ca	0.30	0.46	0.57	0.40	0.51	0.53	0.59	0.65	0.55	1.00				
Ti	0.28	0.45	0.31	0.59	0.46	0.33	0.39	0.53	0.53	0.53	1.00			
Fe	0.34	0.12	0.39	0.50	0.39	0.51	0.53	0.51	0.51	0.44	0.58	1.00		
Mn	0.18	0.44	0.51	0.42	0.43	0.27	0.40	0.44	0.47	0.66	0.67	0.42	1.00	
Ni	0.03	0.38	0.44	0.37	0.33	0.51	0.51	0.54	0.39	0.61	0.41	0.46	0.59	1.00

shown in Figure 4. The concentration of 10 elements (*i.e.* C, Si, P, Al, Mg, K, Ca, Ti, Fe



(a)



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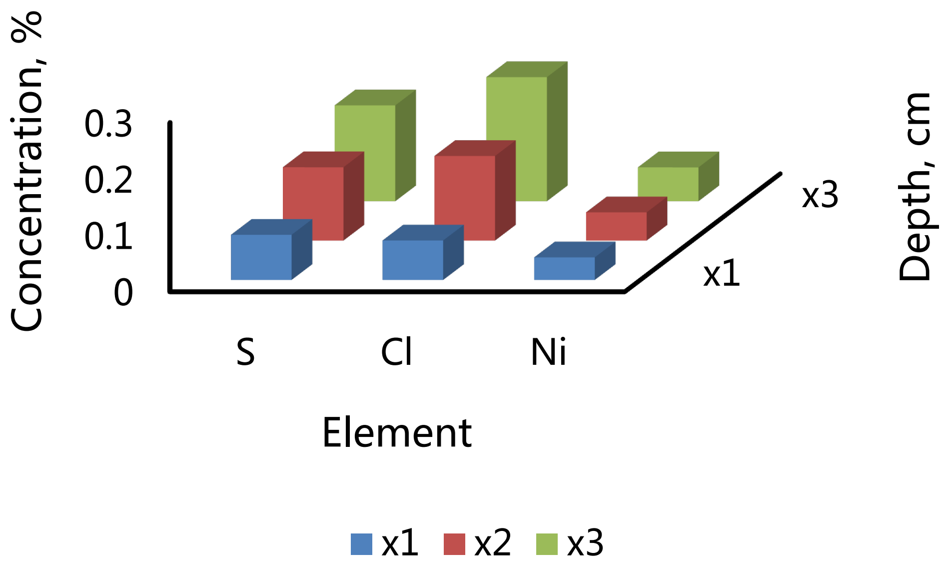

(c)

Figure 4. Vertical distribution of element via soil: x1 (0 - 10 cm), x2(10 - 20 cm) and x3 (20 - 30 cm).

and Mn) was found to decrease with respect to increasing depth profile, may be due to their strong binding with soil organic compounds. The reverse trend was observed with 4 elements (*i.e.* O, S, Cl and Ni), may be due to their less binding with the organic compounds.

3.6. Pollution Indices

The background concentration of Si, Al, Fe, Na, K, Ca, Mg, Ti, Mn, P, S, Cl and Ni reported was 311000, 82000, 39000, 24000, 23000, 22000, 19000, 4650, 775, 62, 655, 370 and 47 mg/kg, respectively [19] [20]. The mean EF value (n = 30) for Ti, Fe, Mn, Cl, P, Ni and S was found to be 1.5, 1.8, 2.4, 3.6, 5.7, 22and 31, respectively. The elements *i.e.* S and Ni; P and Cl; and Mn were enriched highly (20 ≥ EF < 40), significantly (5 ≥ EF < 20) and moderately (2 ≥ EF < 5), respectively in the soil. The mean CF value for Ti, Fe, Mn, Zn, Cl, P, Ni and S was 0.9, 1.1, 1.8, 2.3, 3.0, .2, 16 and 30, respectively. The soil was contaminated highly (CF ≥ 6), considerably (3 ≥ CF < 6) and moderately (1 ≥ CF < 3) with S and Ni; Cl and P; and Mn and Fe, respectively [21]. The PLI value for elements *i.e.* S and Ni was found to be 27 and 16, indicating their significantly high contamination in the soil.

4. Conclusion

The SEM-EDX is a simple and versatile technique, quickly used for quantification of macro elements (*i.e.* C, O, S, Cl, Na, Mg, Al, Si, P, K, Ca, Ti, Fe, Mn and Ni) in soil. The elevated concentration of elements *i.e.* S, P, As, Fe, Mn and Ni in the surface soil was observed probably due to demobilization. The occurrence of S and P at the excessive levels lead to soil to be acidic in nature. The highest pollution indices for S and Ni were recorded. The increasing vertical transport of elements *i.e.* S, Cl and Ni via the soil media was observed may be due to less binding with the soil matrices.

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