RESEARCH ARTICLE

The AJEC THE ASIAN JOURNAL OF EXPERIMENTAL CHEMISTRY
Volume 7 | Issue 2 | December, 2012 | 91-99

Geochemistry and distribution of heavy metals from Itakpe iron-ore mine tailings on soils

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ABSTRACT - Eight dry season soil samples were collected around the tailings dam in order to evaluate the impact of tailings on the soils. The mean concentrations of cations and heavy metals observed are: K(94.66)>Ca(75.92)>Na(18.47)>Mg(5.24) and Fe(29714.13)>Cd(1.51)>Cu(1.30)> Zn (1.20)> Ni (0.64) >Pb(0.55), respectively. Moderate to strong correlation relationship exist between cations and heavy metals.Among the heavy metals, fewer cases of moderate to strong correlations were observed. The factor and cluster analyses reveal both natural and anthropogenic influences on the variables. Three indices were used for heavy metal evaluation. Contamination factor (CF), enrichment factor (EF) and index of geo-accumulation (Igeo). The degrees of heavy metal impact were also evaluated using pollution load index (PLI). Among the three indices, CF and EF recorded this order: Fe>Pb>Cu>Zn>Ni>Cd and Igeo recorded this order: Fe>Pb>Cu>Zn>Cd>Ni among the heavy metals. The pollution load index suggests that all sites have experienced various degrees of deterioration with locations ITK12, ITK21, ITK23, ITK11 and ITK22 severely deteriorated. This study has revealed the need for statistical and quantitative data evaluation and presentation. From these indices used, Fe, Pb, Cu and Zn are the most impacted of the heavy metals. While long term treatment methods such as phytostabilization, phytoextraction and rhizofiltration are recommended, there is also the need to treat the soil and put monitoring measures in place to forestall further contamination.

Key words - Itakpe, Contamination factor, Enrichment factor, Pollution load index, Multivariate analysis

How to cite this paper - Ameh, E.G. (2012). Geochemistry and distribution of heavy metals from Itakpe iron-ore mine tailings on soils. *Asian J. Exp. Chem.*, **7**(2) : 91-99.

Paper history - Received : *27.11.2012;* **Sent for revision :** *10.12.2012;* **Accepted :** *19.12.2012*

Exploratory work on Itakpe iron ore deposit started 1963 by Geological Survey of Nigeria (GSN). Detailed investigations such as pitting, trenching and drilling of boreholes were carried out by Nigerian Steel Development Authority (NSDA) and Techno-Export of Russian. These investigations were later complimented with large scale geological mapping (1:500), trenching and drilling of boreholes by NIOMP at the mine development stage. The feasibility report of these work indicated a reserve of 306,854,000 tones of iron ore made up of 189,672,000 tones of mine able reserve and 117,182,000 tones of geological reserve (Akinrinsola and Adekeye, 1993). This deposit was expected to supply the required iron ore to the Ajaokuta iron and steel company.

Itakpe iron ore deposit consists of two mine sites-one on the east and the other on the west. The eastern mine has been abandoned and not accessible. Within the mining area on the western site are the view point, overburdened dumps, primary and secondary crushers, washing pond, concentrate area and tailing dam point. Parallel to these areas is a seasonal river called PomPom. The topography of the area is rough and hilly. Mining is by surface method.

Iron-Ore mining either by surface or underground methods have severe consequences on the environment. Surface mining involves tearing up large tracts of earth surface; removing materials and throwing the removed soil back into the cut (Priester and Hentschel, 1993; Kozo and Jaoquin, 1982).

Soils on exposure to the surface are subjected to new weathering and compaction; vegetation is removed entirely, surface and groundwater are also impacted due to mining (Priester and Hentschel, 1993; Koval and Belogolova, 1995). Mining causes physical, chemical alterations of soil/sediment, alteration of drainage patterns, erosion and siltation of streams. Also associated with mining is heavy metal pollution of soil/ sediment and water bodies

Geology :

The study area lies within the Benin-Nigeria shield, situated in the Pan African mobile zone extending between the ancient Basements of West African and Congo Cratons in the region of Late Precambrian to Early Palaeozoic orogenies (Rahaman,1976). The Basement Complex rocks of Nigeria are composed predominantly of migmatite gneiss complex; slightly migmatised to unmigmatised paraschists and metaigneous rocks; charnockitic; older granite suites and unmetamorphosed dolerite dykes (Rahaman,1976).

Itakpe iron:

Ore mine sites are located within the Nigerian Basement Complex rocks (Fig.1). Associated rocks in the area are migmatitic gneisses, schists and igneous intrusions (Odigi and Ezepue, 1993; Odigi, 2002). The gneisses and schists include quartz-biotite-hornblende-pyroxene gneiss, quartz-biotite garnet gneiss, amphibolite schist, quartzitic schist and muscovite schist. These gneisses and schists are intruded by igneous bodies such as monzodiorites, granodiorites, granites and pegmatites (Rahaman,1976; Odigi and Ezepue, 1993; Odigi, 2002).

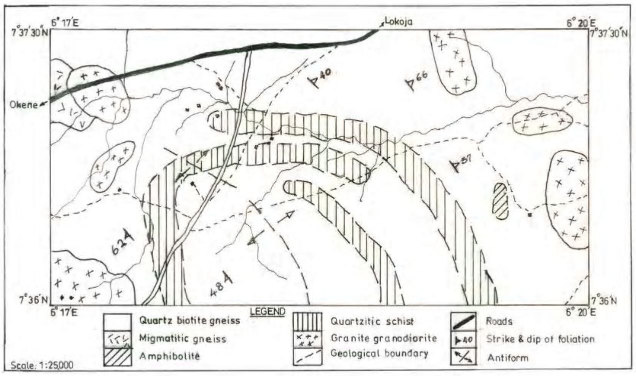

Fig. 1 : Geological map of Itakpe (modified after Odigi, 2002)

To evaluate the degree of tailing impact on soils, eight soil samples were collected during the dry season.

Soil sample collection:

A total of eight dry season soil samples were collected from the tailing area (Fig.2). Sample points were located and recorded using GPS. The samples were collected randomly but evenly distributed around the mines. The soil samples were sun-dried, disaggregated (not crushed) using a pestle and mortar and sieved to minus 80 meshes (0.177mm) with cellulose nitrate filter. 1.0g of each sample was digested with 3ml of 1:2 mixtures of perchloric acid and hydrofluoric acid. The concentrations of six heavy metals and four major cations were determined by AAS. Analytical procedures, operational parameters, calibration and standardization used in this study are according to APHA, 2002.

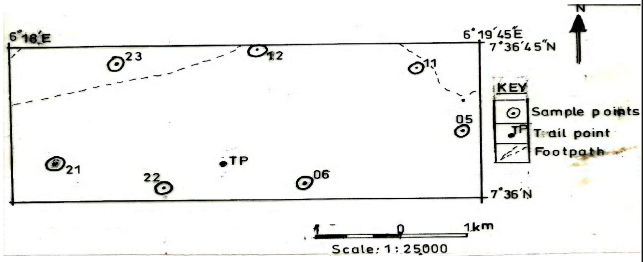

Fig. 2 : Sample location of soil tailings

Analytical methods:

In situ measurements of temperature, pH, Tds and Ec were determined intrusively with their appropriate probes. Spectrophotometer (Model Genesys 20) was used to determine the concentrations of K, Na and Ca Atomic absorption spectrophotometer (Model 210 VGP) was used to determine the concentrations of Mg, Pb, Zn, Ni, Cu, Cd, and Fe. All analyses were performed at Soil Science Dept, Faculty of Agriculture Lab., Kogi State University, Anyigba (APHA, 2002).

SPSS11.0 was used to perform all data analysis after auto-scaling for all parameters. Mathematically, PCA and PFA involve the following five major steps: i) code variables to have zero means and unit variance. ii) calculate covariance matrix iii) find eigenvalues and corresponding eigenvectors iv) discard any component that account for small proportion of variation in data set and v) develop the factor loading matrix and perform varimax rotation on the factor loading matrix to infer the principal parameters (Praveena *et al.,* 2007). In this study only components or factors exhibiting an eigenvalue greater than one were retained.

Hierarchical cluster analysis:

Cluster analysis comprises of a series of multivariate methods used to find the true groups of data (Afshin and Farid, 2007; Rejesh *et al.,* 2002). Here, objects are grouped such that similar objects fall into the same class. Hierarchical clustering which joins the most similar observations and successively the next most similar observations was employed. The levels of similarity at which observations are merged are used to construct dendrogram (Praveena *et al.,* 2007). In this study, squared Euclidean distance method is used to construct dendrogram. A low distance shows that the two objects are similar or close together whereas a large distance indicates dissimilarity.

Factor analysis:

The raw data were treated first to Z-scale transformation for standardization (Rajesh *et al.*, 2002; Shakeri *et al.*, 2008). Multivariate data analysis was utilized to identify the correlations among the measured parameters. Principal component analysis was used to reduce the number of input variables. Spearman's correlation matrix was performed to illustrate the correlation coefficients among the variables.

Determination of enrichment factor:

To evaluate the magnitude of contaminants in the soils, EF were computed for each location relative to the abundances of species in source materials to the control/background value and the following equation as proposed by (Simex and Helz, 1981; Atgrin *et al.*, 2000) was employ to assess degree of contamination, understand the distribution of elements of anthropogenic origin. EF= (C_m/C_{Fe}) sample/ (C_m/C_{Fe}) control/ background value. Where (C_m/C_{Fe}) sample is the ratio of concentration of heavy metal (C_m) to that of Fe (C_{Fe}) in the soil sample and (C_m/C_{Fe}) control/background value is the reference ratio in the control/background value. Fe is selected as reference element because of its relatively higher concentration in the tropics and is one of the widely used reference elements (Navarro *et al.,* 2008; Fagbote and Olanipekun, 2010).

Assessment of pollution load index (PLI): the pollution load index (PLI) proposed by (Tomilson, 1980) was used in this study to measure PLI in soils of Itakpe iron ore mine area. The PLI for a single site is the nth root of n number multiplying the contamination factors (CF values) together. The CF is the quotient obtained as follows: CF= $C_{\text{metal concentration}}/C_{\text{control point concentration of same metal}}$ and PLI for a site = nth√ CF1*CF2….*CFn. Where n is number of heavy metals study (six in this study) and CF= contamination factor. Another index applied is geo-accumulation index (Tijani *et al.*, 2004 and 2011).

Mean concentrations of the cations were K (94.66) >Ca (75.92)>Na (18.47) >Mg (5.24). The heavy metals shows this mean concentration trend: Fe (29714.13) >Cd (1.51) >Cu (1.30) >Zn (1.20) >Ni (0.64) >Pb (0.55) (Table 1). From the correlation (Table 2), the heavy metals Ni-Cu shows strong relationship. The relationship between Zn-Cd; Zn-Cu are moderate while

Table 1 : Concentrations (mg/l) and descriptive statistics of Itakpe soil tailings

Sample locations	Na	K	Ca	Mg	Fe	Cu	Zn	Pb	Ni	Cd
ITK5	16.68	81.62	53.8	3.56	63712.5	.38	.64	.13	.01	1.04
ITK6	13.69	54.61	31.8	4.18	11737.5	.18	.81	.05	.39	1.83
ITK7	23.62	52.61	176.0	4.10	45600.0	.80	1.63	.15	.01	1.78
ITK11	20.20	172.2	60.4	8.20	24500.0	2.14	1.53	.26	1.01	1.54
ITK12	23.45	181.1	62.4	8.49	31201.0	2.17	1.86	.45	1.46	1.74
ITK21	17.01	72.71	84.3	5.49	13600.0	1.71	1.21	1.37	.78	1.59
ITK22	14.32	69.36	75.4	3.07	12321.0	1.37	1.07	1.08	.69	1.48
ITK23	18.76	73.05	63.1	4.81	35041.0	1.63	.81	.91	.76	1.08
Mean	18.47	94.66	75.92	5.24	29714.13	1.30	1.20	.55	.64	1.51
Cp	14.52	60.13	37.30	3.41	307.75	0.17	0.83	0.04	0.55	1.68
Std. Dev.	3.78	51.57	43.40	2.05	18327.84	.76	.44	.50	.49	.30

Table 2 : Correlation co-efficients of variables analysed

	Na	K	Ca	Mg	Fe	Cu	Zn	Pb	Ni	Cd
Na	1.000									
K	.509	1.000								
Ca	.575	-.283	1.000							
Mg	.590	.919	-.179	1.000						
Fe	.418	.002	.270	-.134	1.000					
Cu	.466	.726	-.026	.766	-.288	1.000				
Zn	.801	.621	.477	.699	-.104	.615	1.000			
Pb	-.242	-.155	-.006	-.082	-.478	.487	-.067	1.000		
Ni	.263	.772	-.370	.801	-.476	.871	.519	.368	1.000	
Cd	.223	.114	.283	.287	-.523	.033	.629	-.195	.195	1.000

other relationships are weak. Strong relationship exists between K-Mg while the rest relationships are weak among the cations. Between the cations and heavy metals, strong relationship exists between Zn-Na; K-Cu; K-Ni; Mg-Cu and Mg-Ni. The other correlations were weak.

Factor analysis performed extracted four factors. Factor 1 has eigenvalue of 4.083 and total variance of 4.830 per cent. Factor 1 consists of high loadings of K, Mg, Cu, Ni; moderate loading for Zn and weak loading for Na. Factor 2 consists of high loadings for Na, Ca and moderate loading for Zn. It has eigenvalue of 2.240 and 22.399 per cent total variance. Weak loadings for Fe (negative), Cu and strong factor loading for Pb were recorded for factor three with eigenvalue of 1.732 and total variance of 17.316 per cent. Factor four is an association between Fe and Cd. Both have high factor loadings but Fe is negative. This factor has eigenvalue of 1.701 and total variance of 17.011 per cent. Total cumulative % of 97.555 was enough to explain the latent factors that gave rise to these variations (Table 3).

R-mode cluster analysis yielded two clusters. Cluster one consists of K, Mg, Cu, Ni, Na and Zn. Cluster two consists of Ca and Cd. Bonded to these clusters at a farther Euclidean is Pb and Fe. A closer look shows, Fe may have been influenced uniquely (Fig. 3).

Q-mode factor analysis extracted three factors as being responsible for observed variations. Factor 1 consists of ITK5, ITK6, ITK11, ITK21 and ITK22 with eigenvalue of 3.490 and variance of 43.623 per cent. Factor two is an association of ITK5, ITK7 and ITK22. This factor has total variance of 23.892 per cent and eigenvalue of 1.911. Factor 3 has eigenvalue of 1.806 and 22.575 per cent variance. It consists of locations ITK12, ITK21 and ITK23 (Table 4).

Q-mode cluster yielded 3 clusters. Cluster 1 consists of locations ITK21 and ITK22 with first degree of similarity. Cluster 2 is made up of locations ITK11, ITK12 and ITK6 while cluster 3 consists of locations ITK5, ITK23 and ITK7. Second degree of similarities were observed between ITK11 and ITK 12 in cluster two and ITK5 and ITK23 in cluster three, respectively (Fig.4).

Contamination factor (CF) results shows that Fe has four

Table 3 : R-mode varimax rotated factor analysis

Variable	Factor				Communalities
	1	2	3	4	
Na	.523	.798	-.214	-.105	.968
K	.968	-.005	-.170	-.019	.966
Ca	-.286	.948	.054	.078	.990
Mg	.956	.094	-.096	.146	.954
Fe	-.103	.357	-.500	-.768	.978
Cu	.833	.183	.507	-.003	.984
Zn	.623	.659	-.042	.398	.983
Pb	.013	-.047	.988	-.018	.978
Ni	.878	-.149	.357	.214	.965
Cd	.109	.275	-.183	.932	.989
Eigenvalue	4.083	2.240	1.732	1.701	
% variance	40.830	22.399	17.316	17.011	
Cumulative %	40.830	63.229	80.544	97.555	

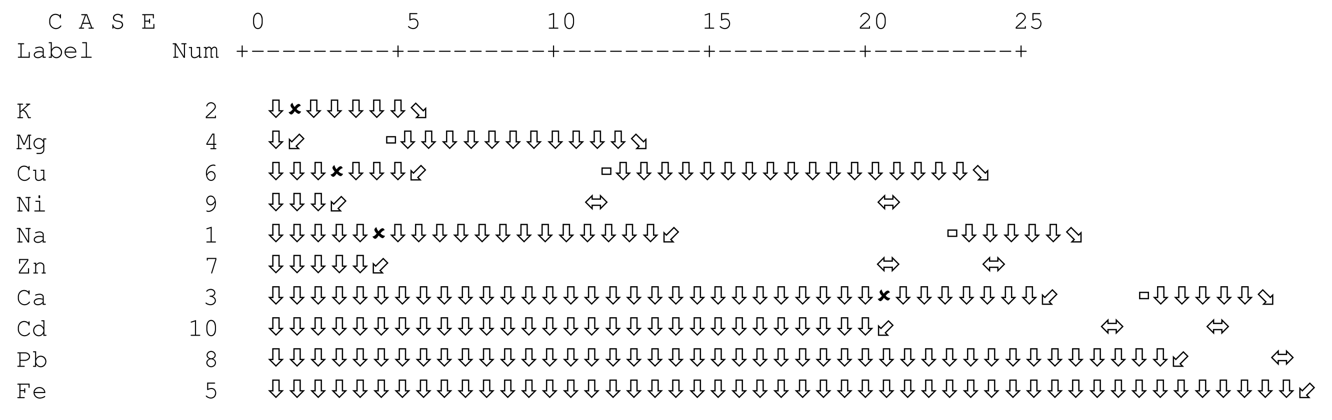

Fig. 3 : R-mode cluster analysis

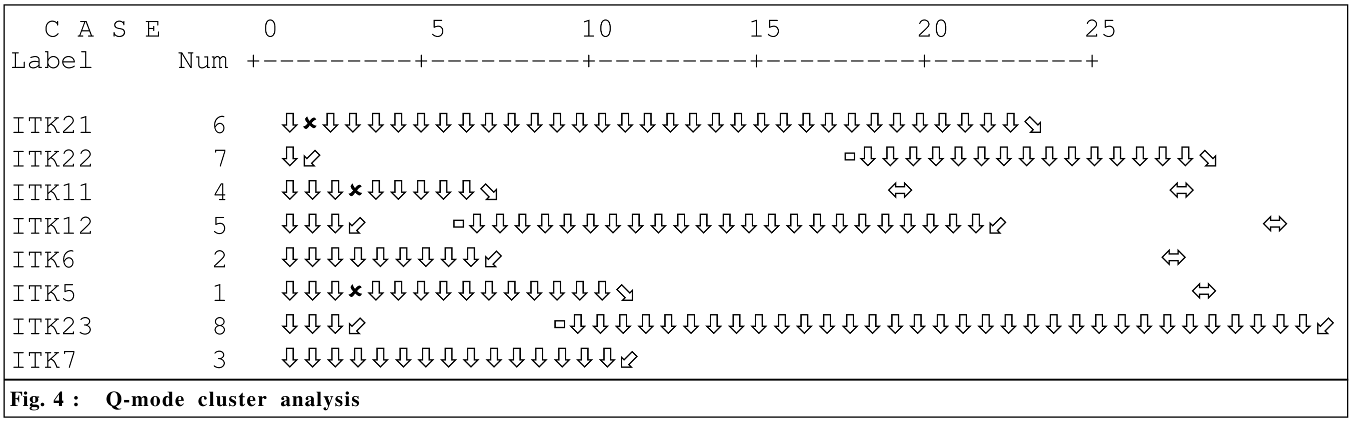

Fig. 4 : **Q-mode cluster analysis**

Table 4 : Q-mode varimax rotated factor analysis

Variable	Facto			
	1	2	3	Communalities
ITk5	.607	.702	-.313	.960
ITK6	.967	-.133	-.112	.966
ITK7	-.165	.974	-.050	.978
ITK11	.979	-.021	.003	.959
ITK12	-.099	.273	-.784	.699
ITK21	.806	.153	.491	.915
ITK22	.731	.590	.050	.885
ITK23	-.071	.073	.914	.845
Eigenvalue	3.490	1.911	1.806	
% variance	43.623	23.892	22.575	
Cumulative %	43.623	67.515	90.090	

folds of CF>6 in all sampled locations (very high contamination). In ITK11, ITK12, ITK21, ITK22 and ITK23, Cu recorded very high contamination while in ITK7 considerable contamination was observed. In locations ITK5 and ITK6, moderate contamination was observed also. In five locations moderate contamination was observed with respect to Zn while three other locations show low contamination. Very high contamination was recorded for Pb at ITK11, ITK12, ITK21, ITK22 and ITK23, considerable contamination at ITK5 and ITK7 while at ITK6, moderate contamination was observed. Low and moderate contamination was recorded for Ni at three and four locations, respectively. Cd also revealed moderate contamination at three locations and low contamination at five other locations (Table 5 and Fig. 5a). The mean concentration of each heavy metals in all locations shows that Fe> Pb >Cu> Zn> Ni> Cd (Fig. 5).

The PLI values indicate site deterioration for all sampled points but significant at locations ITK12, ITK21, ITK23, ITK11

Table 5 : CF of Itakpe soil tailings

Heavy	Sample location							
Metals (mg/l)	ITK5	ITK6	ITK7	ITK11	ITK12	ITK21	ITK22	ITK23
Fe	207.03	38.1397	148.17	79.6101	101.4	44.1917	40.036	113.86
Cu	2.2353	1.05882	4.7059	12.5882	12.76	10.0588	8.0588	9.5882
Zn	0.7711	0.9759	1.9639	1.84337	2.241	1.45783	1.2892	0.9759
Pb	3.25	1.25	3.75	6.5	11.25	34.25	27	22.75
Ni	0.0182	0.70909	0.0182	1.83636	2.655	1.41818	1.2545	1.3818
Cd	0.619	1.08929	1.0595	0.91667	1.036	0.94643	0.881	0.6429
PLI	1.3304	1.49829	1.6661	3.00887	3.551	3.14137	2.8502	3.0301

Contamination factor (CF) index	Degree of contamination
CF < 1	Low contamination
1 ≥ CF ≥ 3	Moderate contamination
3 ≥ CF ≥ 6	Considerable contamination
CF > 6	Very high

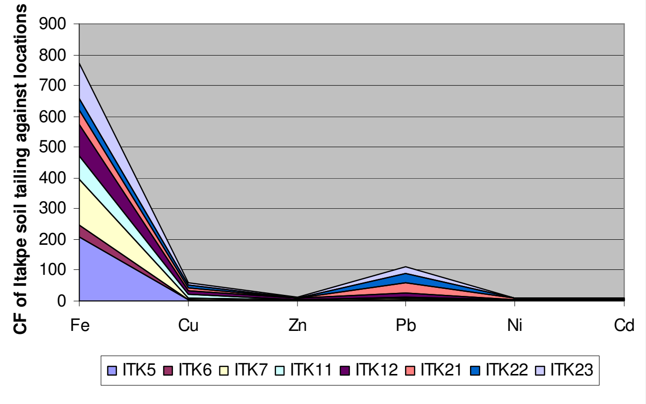

Fig. 5 : CF of Itakpe soil tailings

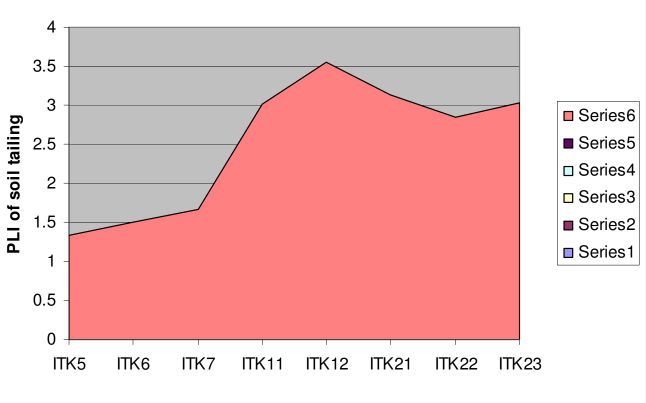

Fig. 6 : PLI of Itakpe soil tailings

Table 6 : Pollution load index (Tomilson *et al*., 1980) of heavy metal classes

PLI indices	Pollution level
0	Perfection
1	only baseline levels of pollutants present
>1	progressive deterioration of the site

and ITK22 (Table 5 and Fig. 6).

Igeo of soil tailings shows that Fe is highly polluted at ITK6, ITK21 and ITK22 and very highly polluted at five other sampled points. Background concentration was observed at ITK5 and ITK6 for Cu, moderate to unpolluted at ITK7, moderately polluted and moderately to highly polluted at three and two other locations, respectively. In three locations, Zn recorded unpolluted and at five other sampled points, background concentrations were observed. At ITK5 and ITK7, Pb was unpolluted and background concentration recorded at ITK6. At locations ITK11 andITK12 moderately polluted was revealed while at ITK23, moderately to highly polluted was observed but at ITK21 and ITK22 highly polluted was observed with respect to Pb. In all the sampled points, Ni and Cd recorded background concentration except at ITK11 and ITK12 where unpolluted was observed with respect to Ni (Table 7 and Fig. 8a). Based on each heavy metal per all sampled points, this trend was observed: Fe >Pb > Cu > Zn > Cd > Ni (Fig. 8b).

The enrichment factor (EF) results indicate that Cu, Zn, Pb, Ni and Cd recorded background concentrations in all the sampled locations. Interestingly, only Fe showed very high enrichment and extremely high enrichment at locations ITK6, ITK21, ITK22 and ITK5, ITK7, ITK11, ITK12 and ITK23, respectively (Table 8 and Fig.9a). Heavy metal trend in all sampled points is thus: Fe >Pb >Cu >Zn >Ni >Cd.

Between the heavy metals analysed, Ni-Cu shows strong relationship, Zn-Cu; Zn-Cd were moderate. Between K-Cu, Na-Zn, Mg-Cu, K-Ni and Mg-Ni significant relationship were observed (Table 2). This relatively significantly relationships observed are a reflection of a source area deposited under similar environmental conditions related to anthropogenic sources (Shakeri *et al.*, 2009; Harikumar and Jisha, 2010). Less significant relations observed elsewhere could suggests control due to variable's physical and chemical characteristics (Abimbola *et al.*, 2005; Tijani *et al.,* 2004).

Factor analysis indicates that factor 1 could be due to natural and anthropogenic influence while factor two on the other hand suggests a wholly natural influence. In contrast to above, factor three and four implies influences from anthropogenic inputs (Pathak *et al.*, 2008; Concas *et al.*, 2006 and Rajesh *et al.*, 2002). R-mode cluster extracted two clusters. Cluster two which consists of K, Mg, Cu, Ni, Na and Zn suggests natural inputs while cluster one indicates input from anthropogenic sources. This cluster consists of Ca, Cd, Pb and Fe (Shakeri *et al.*, 2009; Afshin and Farid, 2007). Inclue : QFA and Q cluster.

The three indices used (CF, Igeo and EF) reveals almost the same order except that Cd >Ni in Igeo. While the least enrichment in soils was Cd, Ni and Zn in that order, Fe, Pb and Cu have the most significant enrichments. During mining operations, significant quantities of wastes or tailings are generated. These wastes and tailings from iron –ore mines contain toxic and heavy metals such as Fe, Pb, Cu, Cd, Ni, Zn etc (Lunchakorn *et al.*,2008; Chunxia *et al.,* 2011;Wang and Dong, 2011). These observed elevated concentrations of these heavy metals can be associated with discharges from iron-ore mining processes, tailings, associated sulphides, vehicular maintenance areas and fuel sources (Monalisa *et al.*, 2010;

Table 7 : Igeo of soil tailings

Heavy metals	Sample location							
mg/l	ITK5	ITK6	ITK7	ITK11	ITK12	ITK21	ITK22	ITK23
Fe	7.1087	4.66826	6.6262	5.72992	6.079	4.88074	4.7383	6.2462
Cu	0.5755	-0.5025	1.6495	3.06904	3.089	2.74543	2.4256	2.6763
Zn	-0.96	-0.6202	0.3887	0.29739	0.579	-0.0411	-0.2185	-0.6202
Pb	1.1155	-0.263	1.3219	2.11548	2.907	4.51307	4.1699	3.9228
Ni	-6.366	-1.0809	-6.3663	0.29189	0.824	-0.0809	-0.2578	-0.1184
Cd	-1.277	-0.4616	-0.5015	-0.7105	-0.534	-0.6644	-0.7678	-1.2224

Igeo = log2 [(Cm)/ (1.5* Cp)]: Cm = measured concentration; Cp = control point concentration; 1.5 = a factor for possible variations in reference concentration due to lithologic differences.

Igeo index	Pollution intensity	Igeo index	Pollution intensity
0	Background concentrations	3-4	Moderately to highly polluted
0-1	Unpolluted	4-5	Highly polluted
1-2	Moderately to unpolluted	>5	Very highly polluted
2-3	Moderately polluted		

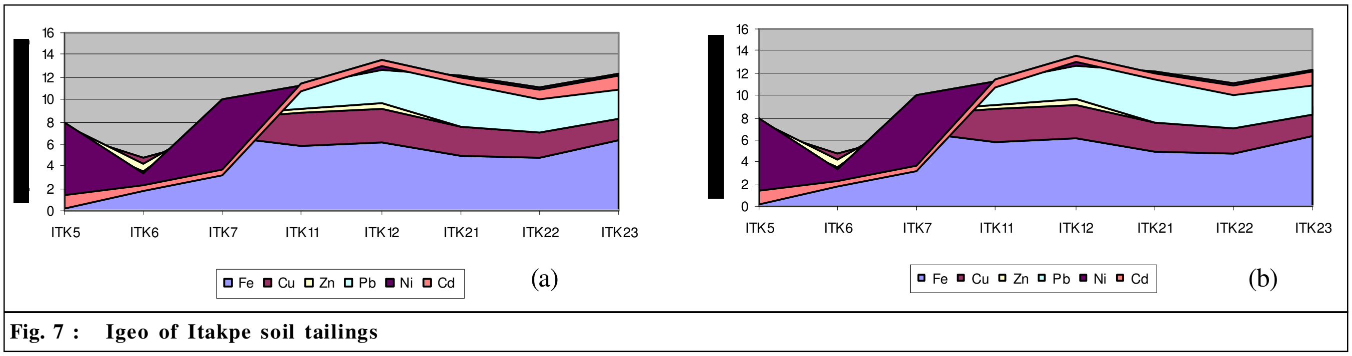

Fig. 7 : Igeo of Itakpe soil tailings

Table 8 : EF of soil tailings

Heavy metals (mg/l)	Sample location							
	ITK5	ITK6	ITK7	ITK11	ITK12	ITK21	ITK22	ITK23
Fe	268.4188	39.0814	75.45	43.187	45.241	30.313	31.056	116.6733
Cu	0.0108	0.02776	0.0318	0.15812	0.126	0.22762	0.2013	0.0842
Zn	0.0037	0.02559	0.0133	0.02316	0.022	0.03299	0.0322	0.0086
Pb	0.0157	0.03277	0.0253	0.08165	0.111	0.77503	0.6744	0.1998
Ni	0.05	0.01859	0.0001	0.02307	0.026	0.03209	0.0313	0.0121
Cd	0.003	0.02856	0.0072	0.01151	0.01	0.02142	0.022	0.0056

EF indices	Degree of enrichment	EF indices	Degree of enrichment
EF $\leq$ 1	Background concentration	EF 5- 20	Significant enrichment
EF 1-2	Depletion to minimal enrichment	EF 20- 40	Very high enrichment
EF 2- 5	Moderate enrichment	EF > 40	Extremely high enrichment

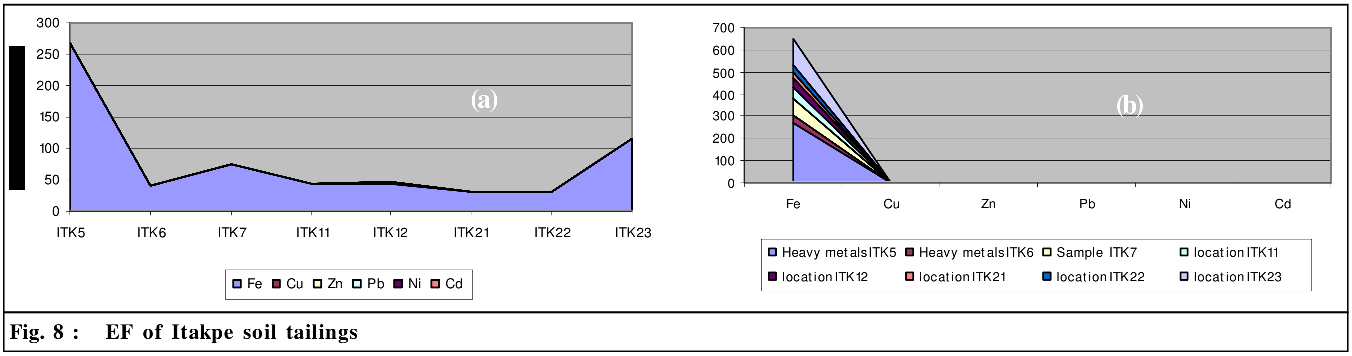

Fig. 8 : EF of Itakpe soil tailings

John, 1995). The elevated levels of these pollutants have direct, acute and chronic impacts on macro invertebrates, micro invertebrates and humans (Joseph, 1986; Monalisa *et al.*, 2010). Pollution load index (PLI) suggests significant impacts on locations ITK (12, 23, 11 and 22) with PLI increasing from up to downstream (Fig. 7).

Conclusion and recommendations:

With respect to Fe, Pb and Cu, the soils were significantly influenced. There is therefore the need to put in measures to reduce heavy metal influx into the soils through substitution of less hazardous materials, improved maintenance and recycling of the wastes/tailings.

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