Physico-chemical characteristics and geochemical composition of soil from the former Air Terjun Mine, Kota Tinggi, Johor

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Abstract—A study on physico-chemical characteristics and geochemical composition of soil from the former Air Terjun Mine, Kota Tinggi Johor was carried out. The mine was known to produce tin and iron until the end of 2001 when their operation ceased. A number of 15 topsoil (0 - 20 cm) samples were collected east way direction using 'Dutch Auger'. Sampling area represented five different kinds of land use that were forest area (S1), open area (S2), pond edge (S3), sand tailing (S4) and river sediment (S5). Three soil samples were collected to represent every type of land use. The soil samples were determined for their physico-chemical characteristics and geochemical compositions. Major element compositions of soils were dominated by SiO_2 followed by Fe_2O_3 and Al_2O_3. TiO_2 and MnO content were less than 4.11% whereas K_2O and P_2O_5 content were less than 1%. The range of concentration for heavy metal were 105-1066 µg/g for Zr , 31-107 µg/g for Pb, 149-723 µg/g for As, 23-319 µg/g for Zn, 15-586 µg/g for Cu, 5-15 µg/g for Ni, 7-144 µg/g for Co and 25-66 µg/g for Cr. The organic matter content ranged from 1.63 to 2.41%. Bulk density of some of the soil sample was high which indicates the presence of high density mineral. pH of the soil samples in the study area ranged from 3.81 to 5.20, thus acidic. The cation exchange capacity mean ranged from 1.19 to 5.41 cmol(+)/kg, whereas the electrical conductivity value ranged from 2.19 to 2.38 mS/cm.

Keywords: geochemistry, heavy metal, enrichment, pollution, Kota Tinggi

Ciri fiziko-kimia dan komposisi geokimia tanih daripada bekas lombong Air Terjun, Kota Tinggi, Johor

Abstrak— Kajian ciri fiziko-kimia dan komposisi geokimia tanih daripada bekas Lombong Air Terjun, Kota Tinggi, Johor telah dijalankan. Lombong ini diketahui mengeluarkan timah dan besi hingga ke hujung tahun 2001 apabila operasinya telah dihentikan. Sebanyak 15 sampel tanih atas (0-20 cm) telah dikutip dalam pada arah timur barat dengan menggunakan 'Dutch Auger'. Kawasan persampelan mewakili lima jenis guna tanah berbeza iaitu hutan (S1), kawasan terbuka (S2), pinggir kolam (S3), sisa pasir lombong (S4) dan sedimen sungai (S5). Sebanyak tiga sampel tanah dikutip untuk mewakili setiap jenis gunatanah. Sampel tanah yang dikutip telah ditentukan ciri fiziko-kimia dan komposisi geokimianya. Kompisisi unsur major dalam tanih didominasi oleh SiO_2 diikuti oleh Fe_2O_3, dan Al_2O_3. Kandungan TiO_2 dan MnO adalah kurang daripada 4.11%, manakala kandungan K_2O dan P_2O_5 kurang daripada 1%. Kandungan purata logam berat adalah 105-1066 µg/g bagi Zr , 31-107 µg/g bagi Pb, 149-723 µg/g bagi As, 23-319 µg/g bagi Zn, 15-586 µg/g bagi Cu, 5-15 µg/g bagi Ni, 7-144 µg/g bagi Co dan 25-66 µg/g bagi Cr. Kandungan bahan organik adalah antara 1.63-2.41%. Ketumpatan pukal bagi sebahagian sampel adalah tinggi menunjukkan kehadiran mineral berat. Nilai pH mempunyai sela antara 3.81 hingga 5.20 menunjukkan keadaannya yang berasid. Kapasiti pertukaran kation mempunyai sela purata antara 1.19 hingga 5.41 cmol(+)/kg, manakala nilai kekonduksian elektrik mempunyai sela daripada 2.19 hingga 2.38 mS/cm.

Kata kunci: geokimia, logam berat, pengayaan, pencemaran, Kota Tinggi

INTRODUCTION

Mining activity is one of the largest economy activities in Malaysia. Mining for mineral resources has been one of the back bones of Malaysian economy which span for a period of more than sixty years. Some of the mining activity in Malaysia includes mining of gold, iron, tin and copper. In Kota Tinggi Johor, there are two mines that used to exist, one of them is the former tin and iron mine of Air Terjun Mine, Kota Tinggi (Figure 1). This mine is situated in a valley which, stretches from north to south. Mining of tin and iron in this area was operated by Waterfall Mining Co. Ltd. by open cast method. Rocks found in this former

tin and iron mine consist of metasediment, light to dark in colour, bedded, fine grain and have undergone low grade regional metamorphism. According to Ganesan (1969), the mine area consists of metamorphic metasediment of hornfels calc-silicate types. Previous researcher reported that this area is a Fe-Sn skarn deposit based on magnetit, cassiterite, fluorit and skarn suite (Yeap 1982). Wan Fuad *et al.* (2000), and Wan Fuad and Adong (2001) have reported an occurrence of copper mineral in the study area in substantial amount.

Mining operation involves stripping of soil overburden, blasting of rock and transportation of ore containing material to the milling where separation of ore from the rock takes place. Prolong period of operation renders the area to be
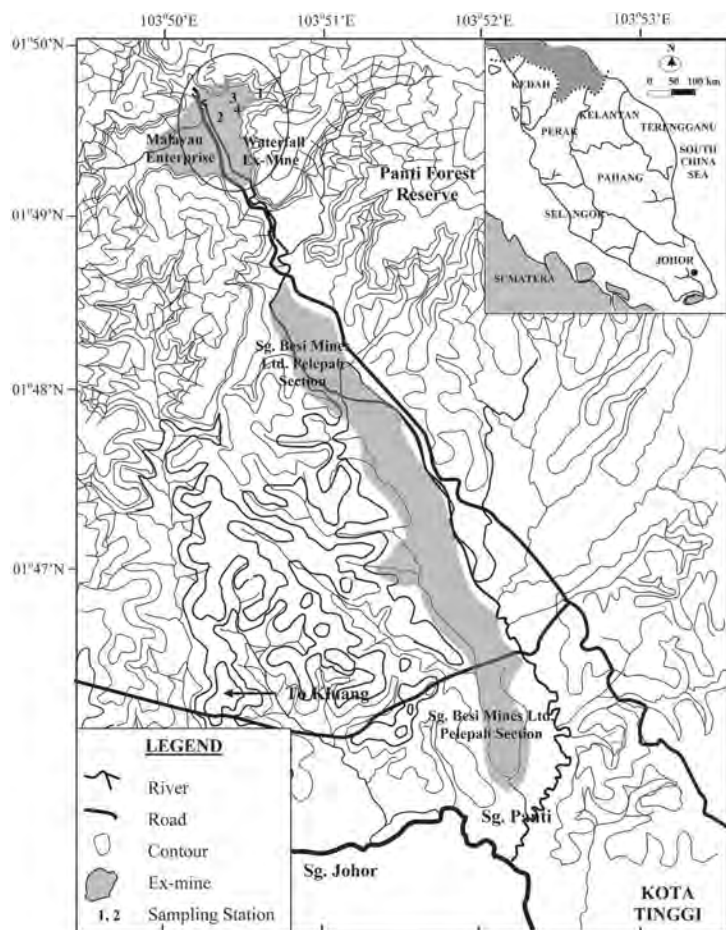
Figure 1: Map showing the location of the study area at Pelepah Kanan ex-mine, Kota Tinggi.

Figure 2: Site map indicating the sampling location.

systematically segregated into several mining micro land use such as rock blasting area, milling area, solid dumping area (sand and silt tailings), amang heap area, and ore storage area. Heaps of solid materials are not properly managed and unprotected from rain and wind erosion. This renders the solid particles to be transported, redistributed and deposited to the lower area of the valley which is the Sg. Pelepah. High heavy metal concentration in soil, amang and solid waste from the mine area brought into the river by water erosion is transported and redistributed by Sg. Pelepah causing a possible contamination in the lower part of the river. This paper discusses the physico-chemical and geochemical contents of solid waste within the former mining areas and discusses their contaminating potential to its surrounding environment.

MATERIALS AND METHODS

A number of 15 soil samples from different types of micro land use at the former Air Terjun Mine, Kota Tinggi (Figure 2) were analysed for their physico-chemical characteristics, which includes particle size, density, organi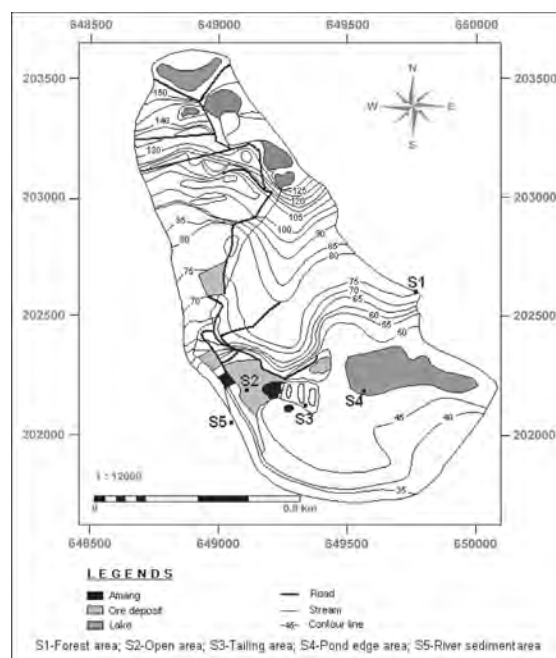c matter content, cation exchange capacity and electrical conductivity. Some selected heavy metals *i.e.* As, Cr, Co, Cu, Ni, Pb, Zr and Zn were determined using XRF technique (De Jongh, 1973, 1979). Texture of soil is obtained by plotting the percentage ratio of sand, silt and clay using the triangle of texture. Particle size distribution was determined by pipette method together with dry sieving (Abdulla, 1966). Organic matter content was determined by loss on ignition technique (Avery & Bascomb, 1982). The pH of soil was determined in soil:water ratio of 1:2.5 (Metson, 1956). True density value was determined using picnometer (Rowell, 1994), whereas the bulk density was determined by waxing method (MAFF, 1982). Exchangeable acidic cation (Al and H) were measured in 1.0 M KCl extract by titration. Exchangeable basic cations were measured in 1.0 M ammonium acetate extract (McLean, 1965) using Atomic Absorption Spectrophotometry. Cation exchange capacity was obtained by summation of acid and basic cations. Electrical conductivity was determined in saturated gypsum extract (Massey & Windsor, 1967). Available potassium and magnesium extracted with double acid (mixture of ammonium acetate and acetic acid extractant) were determined by Flame Atomic Absorption Spectrophotometer (FAAS model Perkin Elmer 3300).

RESULTS AND DISCUSSION

Physical characteristics

Particle size distribution shown in Table 1 indicates that all soil sampling stations are dominated by sand particle with content of more than 66% except for station 3 (pond edge) with sand content that range from 45 to 68%. The forest station (S1) which represents the natural soil condition has a sand content average at 70%, silt content at 9% and clay content at 21%. Stations 2, 3, 4 and 5 represent disturbed areas which can be deduced from the variation in particle content compared to the natural condition. Disturbed areas such as station 2, 4 and 5 in particular, have experienced an increased in their sand content to more than 90% whereas the clay and silt content has markedly decreased. Their sand contents are higher than the sand content in the forest area. The sand content decreased in S3 to 55% and it corresponds to a marked increase in silt content to 38%.

The decrease in sand content in S3 compared to forest area can be attributed to its further distance from the tailing effluent outlet, occupying the very last position in the sedimentation sequence of tailing materials within sedimentation trap. For S2 which is an open area, the sand content was contributed by amang which was redistributed by surface erosion from their place of heap. S4 is a sand tailing area, which receives direct dumping of solid waste from tailing effluent. S5 is of river sediment origin, which naturally undergone particle sorting due to moving water. Soil texture for S2, S4 and S5 are sand whereas the texture for S1 is sandy clay loam and S3 is sandy loam.

Particle densities for forest and pond edge area were lower with their mean values of 2.42 and 2.37 gcm^{-3}, respectively (Table 2). Open area (S2), sand tailing area (S4) and river sediment (S5) have a higher mean of particle density than ordinary sand (2.77 gcm^{-3}) of 2.82 gcm^{-3}, 3.17 gcm^{-3} and 4.28 gcm^{-3}, respectively. This indicates that heavy minerals were accumulated in these particular stations. The bulk density in S1 and S3 are similar at 1.07 gcm^{-3}, whereas for S2, S4 and S5, the value is higher at 1.75 gcm^{-3}, 1.68 gcm^{-3} and 1.86 gcm^{-3}, respectively. High bulk density value normally indicates a compact soil but in this case the soil is not compact but merely represents a heavy mineral composition of soil or sediment. The mean organic matter content ranged from 1.63 to 2.42%, with the highest content shown by S1 (forest) and the lowest shown by S5 (river sediment).

Chemical properties

Chemical characteristics of the soil and sediment in the study area are shown in Table 3. The mean soil pH for each sampling stations are in the range from 3.80 to 5.60. The lowest pH value occurred at the forest station (S1) whereas the highest pH value occurred at the river sediment station (S5). The value of pH in the forest area is significantly lower than the other station. The significant lower value of pH in the forest area is due to decaying effect of organic matter and leaching process that occur continuously in the

forest environment. In other areas the acidity was due to leaching process since there is very low organic matter content in them. Other contributing factor for low pH is the oxidation of sulfur bearing minerals. The mean values for electrical conductivity in the study area are in the range from 2.17 to 2.38 mS/cm. There is no significant difference of electrical conductivity values between all stations. Acres *et al*. (1975) classified this value as low. The mean cation exchange capacity (CEC) value range from 1.19 to 5.41 cmol(+)/kg. The highest value occurs at forest station whereas the lowest value occurs at river sediment station. Cation exchange capacity is linked to high clay and organic matter content in soil. Correlation analysis show that CEC is significantly correlated with organic matter at 0.10% level (n=15, r=0.796), and significantly correlated with percentage of clay at 1% level (n=15, r=0.663). Station 1 that is located at the forest exhibit higher clay content of 21% thus displays a higher cation exchange capacity compared to the other. The mean base saturation in the stations are ranging from 14% to 88.2%. Station 1 which is in the forest area constitutes the lowest base saturation compared to the other stations. The low base saturation was probably due to its low pH.

Heavy metal composition

Heavy metal composition is shown in Table 4. The mean Zr content ranges from 105 to 1066 µg/g. The lowest value occurs in river sediment whereas the highest value occurs in forest area. Natural content of Zr in undisturbed forest soil is very high compared to the other station. It also indicates that Zr content in soil is associated with clay content of the soil. Zr content increased with increase in clay content.

The mean As content ranges from 149 to 723 µg/g. The lowest value occurs in forest area (S1) whereas the highest value occurs in sand tailing area (S4). The As seems to be

Table 1: Particle size distribution and texture of soil.

Sample	Station	% Sand	% Silt	% Clay	Texture
S1 Forest	1	74	8	18	Sandy clay loam
	2	70	10	20	Sandy clay loam
	3	66	9	25	Sandy clay loam
	Mean	**70**	**9**	**21**	
S2 Open area	1	86	8	6	Sand
	2	95	4	1	Sand
	3	88	9	3	Sand
	Mean	**90**	**7**	**3**	
S3 Pond edge	1	52	44	4	Sandy clay
	2	45	45	10	Sandy clay
	3	68	26	6	Sandy clay
	Mean	**55**	**38**	**7**	
S4 Sand tailing	1	93	4	3	Sand
	2	95	4	1	Sand
	3	98	1	1	Sand
	Mean	**95**	**3**	**2**	
S5 River sediment	1	95	3	3	Sand
	2	98	1	1	Sand
	3	97	1	2	Sand
	Mean	**97**	**1**	**2**	

Table 2: Physical properties of soil in the study area.

Sample		Particle density (g/cm³)	Bulk density (g/cm³)	Porosity (%)	Organic Matter (%)
S1	Range	2.33-2.56	1.07-1.08	55.72	2.04-2.73
	Mean	2.42±0.13	1.07±0.01		2.42±0.35
S2	Range	2.37-3.28	1.67-1.89	37.94	1.64-2.48
	Mean	2.82±0.46	1.75±0.12		2.01±0.43
S3	Range	2.22-2.48	1.07-1.10	54.49	1.50-2.61
	Mean	2.37±0.14	1.08±0.02		1.98±0.57
S4	Range	2.97-3.37	1.60-1.77	46.95	1.58-2.24
	Mean	3.17±0.20	1.68±0.09		1.97±0.35
S5	Range	3.91-4.88	1.80-1.89	56.51	1.51-1.73
	Mean	4.28±0.53	1.86±0.05		1.63±0.11

S1, Forest area; S2, Open area; S3, Sand tailings area;
S4, Pond edge area; S5, River sediment area

Table 3: Chemical properties of soil in the study area.

Sample		pH	Electrical conductivity (mS/cm)	Cation Exchange Capacity (cmol(+)/kg)	Base Saturation (%)
S1	Range	3.62-3.90	2.36-2.40	5.15-5.88	4.27-31.57
	Mean	3.80±0.16	2.38±0.02	5.41±0.41	14.04±15.2
S2	Range	5.18-5.21	2.21-2.3	1.53-7.27	83.6-90.7
	Mean	5.20±0.02	2.26±0.05	3.96±2.97	88.2±3.97
S3	Range	4.86-5.40	2.21-2.38	2.59-5.01	79.05-88.57
	Mean	5.11±0.27	2.27±0.09	3.85±1.21	84.71±5.01
S4	Range	5.08-5.14	2.16-2.27	2.56±3.56	75.42-86.80
	Mean	5.11±0.04	2.23±0.06	2.92±0.55	82.19±5.99
S5	Range	5.57-5.64	2.17-2.22	0.99-1.47	64.37-79.79
	Mean	5.61±0.04	2.19±0.03	1.19±0.25	72.11±7.71

S1, Forest area; S2, Open area; S3, Sand tailings area; S4, Pond edge area; S5, River sediment area

concentrated in the soil during mining, in processes ranging from crushing of rocks to separation of ore from solid waste in the form sand tailings and silt. Arsenic concentration in the study area has exceeded the maximum of normal range in soil which is less than 5-40 ppm. Arsenic concentration has also exceeded the potential toxic level in soil at 15-50 ppm.

The mean Pb content ranges from 31 to 107 µg/g. The lowest value occurs in forest area whereas the highest value occurs in the open area. Again the concentration of Pb has increased from natural condition (forest area – S1), which can be attributed to the mining processes. The Pb concentration in all of the sampling station is still within the normal range in soil which is 10-150 ppm, and below the potential toxicity threshold at 100-400 ppm.

The mean Zn content ranges from 23 to 319 µg/g. The lowest value occurs in forest whereas the highest value occurs in open area. The other station also recorded a high Zn concentration compared to the forest area, which indicates the respective enrichment of this metal content in disturbed environment. The Zn concentration in Station 2, 4 and 5 have exceeded the normal concentration in soil at 25-200 ppm, but within the range of potential toxic limit in soil at 70-400 ppm (Kabata-Pendias & Pendias, 2001).

The mean Cu content ranges from 15 to 586 µg/g. The lowest value occurs in forest (S1) whereas the highest value occurs in sand tailings (S4) area. Copper has been concentrated many fold (18 to 39 times) in disturbed area compared to its natural concentration in the forest area. The concentration of Cu in the forest area is within the lower limit of normal concentration in soil, and lower than the potential toxic level. However concentrations in disturbed areas are above the normal range in soil at (2-60 ppm), and higher than potential toxicity level in soil at 60-125 ppm. Copper in soils is held strongly on inorganic and organic exchange sites and complexes with organic matter. Thus it is eroded together with inorganic and organic matter to other location.

The mean Co content ranges from 7 to 144 µg/g. The lowest value occurs in forest (S1) whereas the highest value occurs in the river sediment (S5). The concentration of Co in disturbed areas is many fold higher than the content in the forest area. Cobalt content in its natural condition is well below the potential toxic concentration in soil at 25-50 ppm, however under disturbed condition the Co content is high and above the threshold limit for potential toxicity, except for Station 2.

The mean Ni content ranges from 5 to 15 µg/g. The lowest value occurs in river sediment (S5) whereas the highest value occurs in forest area (S1). There is no significant difference between mean values of Ni content in all of the station, except for S5 which is lower. Nickel content in the forest area is slightly higher than the disturbed area. Nickel content in all stations of the study area is within the lower boundary of the normal content in soils, and well below the potential toxicity limits in soil.

The mean Cr content range from 25 to 66 µg/g. The lowest value occurs in forest area (S1) whereas the highest value occurs in pond edge area (S3). Concentration of Cr in the forest area is at least two-fold lower than concentration in the other stations. Cr concentration in all the stations is below the potential toxicity threshold in soils.

Data on heavy metal concentration indicated that heavy metal contents are higher in all the station compared to the forest area, except for Ni and Zr. This indicates that heavy metal has been concentrated in the top soil of disturbed areas. The enrichment of heavy metal in the top soil could be associated with natural secondary ore forming processes during weathering or concentrated during separation of ore from the host rock. Correlation analysis indicates that the enrichment of heavy metal in the topsoil is partly influenced by pH which has a significant positive correlation at least at 5% level (n=15) with Zr, Pb, As, Zn, Cu and Co. Other soil properties that influence heavy metal enrichment are organic matter content and percentage of clay particle.

Table 4: Heavy metal composition in soils (µg/g)

Sample		Zr	As	Pb	Zn	Cu	Co	Ni	Cr
1	Range	995-1126	128-188	14-60	20-26	7-20	0-11	14-17	24-26
	Mean±SD	1066±66.2	149.3±33.6	31.3±25	23.3±3.1	15.0±7	7.0±6.1	15.3±1.5	24.7±1.2
2	Range	64-197	571-797	91-135	289-345	327-752	49-173	0-16	48-68
	Mean±SD	140±68.5	652.3±125.6	107.3±24.1	319.3±28.3	500±223	112.7±62.1	8.0±8.0	55.3±11
3	Range	208-471	408-585	82-90	122-231	331-764	29-59	13-17	61-72
	Mean±SD	305.3±144	523.7±100	84.7±4.6	194±62.4	546±216	46.7±15.7	14.3±2.3	66.0±5.6
4	Range	88-164	398-949	42-156	114-465	288-902	82-171	4-13	42-66
	Mean±SD	122±38.6	723±288	95±57	288.3±175	586.3±307	120±46	9.0±4.6	57±13
5	Range	73-128	449-560	59-98	265-369	195-321	133-156	3-6	57-71
	Mean±SD	105±28.6	517.3±58	79±19.5	317.3±52	269.7±66	143.7±11.6	5.0±1.7	64.7±7.1

S1, Forest area; S2, Open area; S3, Sand tailings area; S4, Pond edge area; S5, River sediment area

Table 5: Mean and standard deviation of major elements composition in soils in the form of oxide (%).

Sample		SiO_2	TiO_2	Al_2O_3	Fe_2O_3	MnO	MgO	CaO	Na_2O	K_2O	P_2O_5	LOI
S1	Mean	67.30	0.70	16.90	3.00	0.01	0.14	0.05	0.87	0.52	0.04	10.52
	SD	2.50	0.03	1.10	0.40	0.00	0.07	0.01	0.96	0.07	0.01	0.90
S2	Mean	32.90	0.56	8.60	47.00	0.52	0.41	0.89	2.01	0.97	0.08	6.00
	SD	24.00	0.48	5.70	32.40	0.28	0.11	0.48	1.86	0.89	0.02	2.74
S3	Mean	54.70	0.66	16.80	18.00	0.50	0.62	1.27	0.95	1.46	0.08	5.01
	SD	6.90	0.05	7.00	0.30	0.14	0.14	0.41	0.07	0.31	0.01	1.03
S4	Mean	28.60	0.39	9.20	52.40	0.52	0.52	1.37	1.76	0.81	0.09	4.44
	SD	16.60	0.22	5.30	26.90	0.20	0.10	0.49	0.37	0.55	0.03	3.51
S5	Mean	18.20	0.35	7.50	65.90	0.55	0.34	0.14	1.79	0.45	0.09	4.67
	SD	2.40	0.04	1.03	3.03	0.22	0.09	0.01	0.45	0.08	0.02	1.24

S1, Forest area; S2, Open area; S3, Sand tailings area; S4, Pond edge area; S5, River sediment area

Major elements content

Major elements content in the form of oxide varies significantly between sampling station. This is clearly demonstrated by the three major oxides, namely SiO_2, Fe_2O_3 and Al_2O_3 (Table 5). SiO_2 content significantly decreases from 67.3% at the relatively undisturbed forest environment to 54.7% at the pond edge, 32.9% in open area, 28.6% in sand tailings area and 18.2% in river sediment. The decrease in the content of SiO_2 from the open area, sand tailings and river sediment was probably influenced by the particle size distribution. Most of the silicates components of soil are in the form clay particles. Separation of these particles decreases this particular area in terms of SiO_2 content. Second possible reason is the selective leaching of the silicates from the area. In the case of pond edge the content of SiO_2 does not significantly differ with the forest area because it comprises clay and silt particle resulted from sedimentation of solid waste from ore separating and

Table 6: Correlation between major elements (oxides) with heavy metal

Oxide Metals	SiO_2	TiO_2	Al_2O_3	Fe_2O_3	MnO
Zr	0.737**		0.616*	-0.726**	-0.799***
Pb					0.714**
As					0.685**
Zn					0.761***
Cu					0.551*
Co	-0.990***	-0.851***	-0.814***	0.986***	0.671**
Ni	0.916***	0.888***	0.910***	-0.950***	-0.532*
Cr					0.704**

n=15; r= 0.514* correlation significant at 5% level; r=0.641** correlation significant at 1% level r=0.760*** correlation significant at 0.1% level

cleaning activity. The same trend occurs with Al_2O_3 content in soil samples.

The most enriched oxide is Fe_2O_3. Its content in forest environment is only 2.98% but the content was increased many fold in the open area, sand tailings and river sediment. The soil samples in the pond edge still show a six-fold increase in Fe_2O_3 compared to the forest soil. High concentration of Fe_2O_3 is expected because the main activity in this mine is iron mining. Other oxide contents such as MnO, MgO, CaO, Na_2O and P_2O_5 recorded a significant increased from the forest environment to other soil sampling stations except for the pond edge station.

Heavy metal content in soil and sediment is also influenced by the major elements (Table 6). Heavy metals such as Co, Zn, As, Co, Cr and Pb show a significant positive correlation with MnO which indicated the occurrence of co-precipitation and adsorption of heavy metal into the oxide minerals (Alloway, 1995). Co also co-precipitates with Fe_2O_3. Zr and Ni show a significant negative correlation with iron and Mn oxide but show significant positive correlation with SiO_2 and Al_2O_3. This indicates that Zr and Ni are components in the alumino-silicates mineral such as smectite and illite.

CONCLUSION

Topsoil in the study area is dominated by sandy texture, lack of organic and clay content, thus low in cation exchange capacity.

Major elements in the form of oxides are dominated by SiO_2, Fe_2O_3 and Al_2O_3 in varying ratio. River sediment, sand tailing and open area contain very high concentration of Fe_2O_3 compared to other areas.

Heavy metal contents in topsoils of disturbed area are higher than undisturbed area (forest) except for Ni and Zr which indicate heavy metal enrichment in disturbed area.

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