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DEPOSITION OF HEAVY METALS IN SOIL AND HIGHER PLANT RELATED TO RARE-EARTH PROCESSING ACTIVITIES

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ABSTRACT

This study evaluated the level of heavy metal content around rare-earth refinery sites by using *Acacia magnium* tree bark and its topsoil substrate. The samples were collected from a total of seven sites surrounding the selected study area. The sampling sites were north, south, and west of the refinery site. Parallel tree bark and topsoil samples were collected from the same points. Six elements Al, Mn, V, Cu, Ni, and Pb were measured by using ICP-MS. The data obtained were analysed for distribution, correlation, deposition consistency, metal ratio, and contamination factor. The metal correlations between tree bark and topsoil were evaluated. The concentrations of the studied elements in soil and tree bark samples were in the same order of magnitude. For almost all of the elements, higher accumulations occurred in soil than in tree bark. Significant correlations between concentrations in soil and tree bark were obtained only for Mn and Cu, types of natural pollutants. The other elements were in a relatively low correlation suggesting a mixture of emitters.

INTRODUCTION

Biomonitoring has been widely used to evaluate airborne contamination and its changes over a long period of time (Catinon *et al.*, 2009). Bioindicators such as tree bark, mosses, soil, and lichen have been widely used in order to pinpoint the airborne contamination degree in a specific area. One of the most common bioindicators used in air pollution studies is tree bark. The outer layer of a tree bark is an effective passive accumulator of airborne particles which settle through wet and dry deposition (Škrbic *et al.*, 2012). Tree bark has the best ability to accumulate a huge amount of

atmospheric dust, thus making it a good substance for indicating the level of air pollution (Catinon *et al.*, 2009). Furthermore, tree bark can represent several years of accumulations. Some of the key factors that determine the effectiveness of the adsorption of airborne suspended particles onto tree bark are the moisture, roughness of the tree bark surface, and the electrically charged surface. All these factors make the tree bark a highly effective accumulator of the suspended particles (Catinon *et al.*, 2009). Though the main concern of the accumulation is not the physical contact between the particles and bark, these factors might help in analysing heavy metal content in the

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tree bark as a whole, as the retention of metals in the plant may be too minute to be analysed (Harju *et al.*, 2002). Tree bark is one of the best bioindicators because it has high lipid content and large surface area (Salamova and Hites, 2010) and shows the amount of pollutants over a period of several years. Tree bark has been used to represent the accumulation of airborne mercury and organic and inorganic pollutants (Lodenius, 2012).

In addition, the heavy metals' migration from the soil through the roots into the bark is usually insignificant (Roganovic and Durovic, 2013). Soil plays a vital role in sustaining the earth's ecosystems: for example, the cycling of nutrients, biochemical transformations, support for plants and infrastructure, filtration of water, and many recreational activities (BI et al., 2013). Soil is considered one of the most reliable substances to be used in atmospheric pollution studies due to the low cost, simplicity of analytical procedures, and ease of chemical analysis. Numerous studies have shown that soils obtain a large input of trace metals from different anthropogenic sources, especially from industrial areas and vehicle emissions (Skrbic et al., 2012). Sedimentation, impaction, and interception processes from the deposition of particles on all environmental surfaces cause soils to act as a sink for the metallic load (Tokalioglu and Kartal, 2006).

It is important to monitor and evaluate the level of metal pollution in the surrounding industrial area for the benefits of local residents and related communities. Furthermore, this type of pollution can also affect crops, soil content, and the surrounding air. Thus, the aim of the present study is to evaluate the atmospheric pollution levels of the selected heavy metals in Gebeng Industrial Area, one of the most important industrial areas in the state, by analysing the tree bark and topsoil collected from the area and to demonstrate the factors that contribute to metal distribution in the selected area.

MATERIAL AND METHODS

Sampling locations

All the tree barks and topsoil samples were collected in the vicinity of Gebeng Industrial Area in Kuantan Pahang, Malaysia within a radius of 20 km. The site is located between latitude 3° 57′ 21.84"N and longitude103° 22′15.74"-E with a temperate climate and two monsoon seasons; the south-west monsoon (late May to September) and the north-east monsoon (November to March) which result in heavy rainfall. The air quality of the area is being influenced by numerous multinational corporations that are actively operating there. The area is also crossed by a major highway connecting north and south parts of the state. Two samples were collected in the nearby rural area as the control for this study.

Tree bark sampling and chemical treatment

Tree barks from a total of seven Acacia mangium trees (three replicates) were collected in the vicinity of the selected area. Each sampling point site had an area of 50 m × 50 m. The trees were approximately similar in age (based on the size of the tree) and the 5 mm of the outer part of the bark was chiselled with a pre-cleaned stainless steel chisel (Tye et al. 2006). At each sampling station, the samples were taken from two opposite sides of the tree at a height of 1.5 m from the ground. This height was chosen specifically to avoid the areas where soil particles could splash on the trunk during rainfall (Mansor 2008). Before the tree bark samples were crushed, the outermost layer of the bark was superficially brushed, but the innermost layer remained untouched. The sample was not washed in order to avoid the loss of absorbed particles (Ferreira et al., 2012) and to avoid moisture contribution to the tree bark. The samples were dried in an oven for 24 hours at a temperature of 70 °C before being ground into powder form. Two grams of the samples were dissolved in 15 mL concentrated HNO₃ (69-70%) in a PTFE beaker (Fujiwara et al., 2011).

Soil sampling and chemical treatment

Topsoil samples (0-20 cm in depth) were collected from seven different locations (three replicates) around the Gebeng Industrial Area at the exact locations where the mosses were taken from. Each soil sample consisted of four subsamples which were obtained within 1 m2 of each sampling site, and then stored in polyethylene bags for transport and storage. The soil samples were air-dried and then sieved through a 1.0 mm polyethylene sieve. The portions of the soil samples (approximately 20 g) were further ground to pass through a 100-mesh polyethylene sieve. Approximately 0.50 g (100 mesh) of each soil sample was treated with a mixture (1:5, v/v) of 2 mL concentrated HClO, and 10 mL HF. The concentrations of the studied heavy metals of each sample fraction were measured by an inductively coupled plasma-mass spectrometer (ICP-MS). The quality control of the analytical procedure was carried out by analysing the standard reference material SRM-CC141 and the pine needle. The standard reference material (SRM) was analysed in exactly the same way as the actual samples. Three replicate measurements were made for each SRM in order to obtain the average value. The results obtained showed that none of the measurements differed by more than 10%. The high recoveries obtained for all the studied elements confirmed the validity of the method for the determination of selected metals.

RESULTS AND DISCUSSION

Concentrations of metals

The levels of total concentration of selected metals measured in this study are presented in Table 1. In general, the results obtained clearly indicate that all of the studied elements are unevenly distributed throughout the study area. Several factors are suspected as the cause of this, especially factors that are related to meteorological conditions and possible sources that contribute to the concentration of the elements.

The results of relative standard deviation of the metal concentrations in this study clearly indicate that almost all of the studied elements are not evenly deposited around the study area. This strongly suggests that the existence of these metals in the study area is influenced by various types of emitters, either naturally or artificially. Nearly all of the studied metals indicate values of variation of 50 % and higher. The lowest variation of 35 % was recorded for the concentration of the other elements, especially Mn, Cu,

and Pb in tree bark samples and Mn, Ni, and Pb in topsoil samples varied more than 80 %.

Among the studied heavy metals (both types of samples), Al content was always the highest at all sampling sites followed by Mn, V, Cu, Pb, and Ni. The higher value Al concentration in the study area can be related to the various industrial activities there. Some of their operations are related to the production and processing of Al from bauxite ore and others like packaging industry, transportation industry, and building and construction industry (Pais and Jones, 1997). Furthermore, the abundance of Al could also be related to the natural resources such as soil and dust emissions and agricultural activities (Rajsic et al., 2008). The high concentration of the metals V, Ni, and Pb in the lower surface of atmospheric air is related to the burning processes; V and Ni are considered as the main markers of oil combustion (Facchinelli et al., 2002). It was clearly shown that Cu, Pb, Ni, and V are mainly derived from anthropogenic activities such as metal industry and fossil fuel combustion (Abdullah 2011).

Table 2. The ratio (mg/kg dry wt) of soil sample to tree bark sample for all studied elements

Sampling station	Al	V	Mn	Ni	Cu	Pb
A B	2313.4 400.4	12.0 2.8	73.3 2.5	3.9 0.5	4.8 4.7	0.5 0.5
C	1918.6	6.4	9.0	1.7	3.7	0.7
D	6232.6	3.9	59.7	5.3	7.9	3.2
E	1571.7	10.3	42.0	0.6	4.5	1.4
F	18345.3	9.3	71.5	4.2	14.3	0.5
G	2921.2	9.4	3.7	10.7	26.6	6.3

Table 1. The elements concentrations (mean value) in tree bark and topsoil samples at all sampling stations

Sampling station	Soil samples, mg/kg dry weight (average of 3 replications)							Bark samples, mg/kg dry weight (average of 3 replications)					
	Al	V	Mn	Ni	Cu	Pb	Al	V	Mn	Ni	Cu Pb		
А	418492	59.9	87.9	12.1	15.4	17.5	180.9	5.1	1.2	3.1	3.2	33.4	
В	40002	11.1	33.9	2.8	66.2	5.9	99.9	4.2	13.5	6.1	14.2	11.1	
С	422092	19.8	18.8	3.8	15.4	6.9	220	3.1	2.1	2.2	4.2	9.3	
D	648192	27.5	334.4	7.9	17.3	27.3	104	7.1	5.6	1.5	2.2	8.5	
Е	62552	22.6	92.3	4.9	13.5	14.2	39.8	2.2	2.2	8.2	3.1	10.5	
F	753992	67.2	228.8	29.3	58.6	31.5	41.1	7.2	3.2	6.9	4.1	67.5	
G	370993	57.5	80.5	22.5	55.8	66.5	127.0	6.1	21.5	2.1	2.1	10.6	
Average	388045	37.9	125.2	11.9	34.6	24.3	116.1	5.0	7.0	4.3	4.7	21.6	
% RSD	69.07	60.05	91.04	86.01	64.68	79.91	53.46	35.09	100.42	58.13	83.39	94.78	
Control	27196	11.3	11.9	1.5	10.3	2.1	30.6	1.1	1.0	1.1	1.3	5.1	

The concentrations of the studied elements in bark samples were relatively small compared to those obtained in topsoil samples. For the purpose of general comparison, the ratio of metal concentrations of topsoil and tree bark samples were calculated and the results obtained are shown in Table 2. To assess the metal contamination in the area, a contamination factor was calculated. The results of the degree of contamination of each of the studied metals at each sampling station are summarized in Table 3. The CF value was classified according to the calculation proposed by Fernandez and Carballeira (2001).

The result obtained suggests that the Gebeng Industrial Area has been contaminated with certain levels of the studied metals. The values of the metal concentration ratios of topsoil and tree bark clearly suggest that more metals had accumulated in soil samples than in tree bark. The average concentrations of Al, V, Mn, Ni, V, Cu, and Pb in the soil samples were measured as 388045, 37.9, 125.2, 11.9, 34.6, and 24.3 mg/kg dry wt respectively and the same elements which had accumulated in tree bark were recorded as 116.1, 5.0, 7.0, 4.3, 4.7 and 21.6 mg/kg dry weight respectively. Significant differences were recorded for Al in which the probability of occurrence was hundreds to thousands times higher. For other metals, it was observed that the ratios were 1.4 to 73 times higher between soil and tree bark samples. It was clarified that the accumulation of Al, V, Mn, and Cu in soil samples is more a dynamic process compared to tree bark. The different metal concentrations in soil and tree bark samples could also be related to the fact that the deposition of heavy metals in tree bark samples reflects accumulation over a period of three to five years but the accumulation of metals in soil is a long-term process. Therefore, the contents of heavy metals in tree bark samples mostly depend on the dry and wet deposition caused by the local activities in the area and also by long-range transport. The losses of heavy metals from the outer bark may be caused by the stem flow wash-off and by radial transport from bark to xylem (Al-Alawi *et al.*, 2007).

The CF values measured in this study clearly indicate that the area has been contaminated with the studied metals with a degree ranging from slightly contaminated to extremely contaminated as summarized in Table 3. Almost all of the Al concentrations in the soil sample were at the level of severe contamination except for the sampling stations of B and E on the outskirts of Gebeng Industrial Area. Other elements were in the range of slight to moderate contamination. The most abundant element contributed to the contamination in the bark samples was Mn at sampling station G, in which the station is approximately 15 km away from the western part of the industrial site. The heavy presence of this element did not reflect the contribution of any industrial activities because of its position that is far away from the industrial region. The cause could be the re-suspension of soil dust or agriculture-related activities.

Correlation of heavy metals in tree bark and topsoil samples

In general, tree bark and soil are seen as the best media for monitoring the fall-out of atmospheric pollutants in the surrounding area. Bark is rough and broad with flat scales, and able to trap various types of pollutants is chosen to be used as a bioindicator of heavy metals (Poykio *et al.*, 2005). Many different factors may influence the content of heavy metals in tree bark such as the concentration of heavy metals in air, bark properties, climatic factors, and leaching processes. The accumulation process is not influenced by the variation of soil parameters and its element

Sampling station	CF, Soil Samples							CF, Bark Samples						
	Al	V	Mn	Ni	Cu	Pb	Al	V	Mn	Ni	Cu	Pb		
A	15.4	5.3	7.4	8.1	1.5	8.3	5.9	5.1	1.1	3.1	2.7	6.5		
В	1.5	1.0	2.8	1.9	6.4	2.8	3.3	4.2	12.3	6.1	11.8	2.2		
С	15.5	1.8	1.6	2.5	1.5	3.3	7.2	3.1	1.9	2.2	3.5	1.8		
D	23.8	2.4	28.1	5.3	1.7	13.0	3.4	7.1	5.1	1.5	1.8	1.7		
Е	2.3	2.0	7.8	3.3	1.3	6.8	1.3	2.2	2.0	8.2	2.6	2.1		
F	27.7	5.9	19.2	19.5	5.7	15.0	1.3	7.2	2.9	6.9	3.4	13.2		
G	13.6	5.1	6.8	15.0	5.4	31.7	4.2	6.1	19.5	2.1	1.8	2.1		

Table 3. CF values for all the studied elements

***CF value: < 1 (no contamination), 1-2 (suspected), 2-3.5 (slightly), 3.5-8 (moderately), 8-27 (severe), > 27 (extremely) – Fernandez *et al.*, 2007.

Pair	Topsoil s	amples		Tree bar	k samples	Moss topsoil				
	Pearson, R	Pair	Pearson, R	Pair	Pearson, R	Pair	Pearson, R	Pair	Pearson, R	t-test value (tree bark & soil)
Al-V	0.60	V-Pb	0.63	Al-V	0.18	V-Pb	0.48	Al-Al	0.05	0.002
Al-Mn	0.71	Mn-Ni	0.34	Al-Mn	0.05	Mn-Ni	0.23	Mn-Mn	0.63	0.002
Al-Ni	0.63	Mn-Cu	0.09	Al-Ni	0.74	Mn-Cu	0.25	V-V	0.19	0.018
Al-Cu	0.04	Mn-Pb	0.25	Al-Cu	0.07	Mn-Pb	0.34	Ni-Ni	0.04	0.082
Al-Pb	0.34	Ni-Cu	0.49	Al-Pb	0.34	Ni-Cu	0.36	Cu-Cu	0.56	0.007
V-Mn	0.25	Ni-Pb	0.73	V-Mn	0.23	Ni-Pb	0.36	Pb-Pb	0.09	0.818
V-Ni	0.91	Cu-Pb	0.37	V-Ni	0.36	Cu-Pb	0.11			
V-Cu	0.21			V-Cu	0.26					

Table 4. Relationships between heavy metal concentrations in topsoil and tree bark samples

*** Critical value of t for 10 degrees of freedom is 2.23 at 95% confidence level (two samples equal variance)

content (Huhn *et al.*, 1995). Direct collection and analysis of the deposition by evaluating the soil samples has some advantages and are considered as the conventional approach.

In order to study the relationship of the studied elements in soil and tree bark, Pearson correlation was applied to the elements concentration data obtained in both media. Table 4 shows the correlation coefficient values for all the element pairs in tree bark and topsoil samples obtained in this study. The Pearson correlation between the sets of data is a measure of how well the metals are related. It shows the linear relationship between two sets of metal concentrations. The closer the value of R to one, the smaller the variation of the metal concentrations. As shown in Table 4, Al in topsoil was correlated with V, Mn, and Ni and the R value was observed to be within 0.60 and 0.91. Relatively strong correlations were also found for V-Pb and Ni-Pb. The small variation of the metal concentrations in a group could mean that the metals came from similar sources. Therefore, it is assumed that V, Mn, Ni, and Pb in the soil samples were contributed by the same anthropogenic sources. As for the tree bark samples, the results clearly showed that almost all of the pairs were not significantly correlated except for the pair of Al and Ni with an R-value of 0.74. The weak correlation between the metals in tree bark samples suggests that the different sources of the metals have been effectively retained in the bark samples.

The null hypothesis test was performed on the data set to see if there any significant differences in the element concentrations between the bark and topsoil samples. The results of this study clearly showed that all of the single element pairs (bark-topsoil) were low and moderately correlated with the recorded R-values of 0.04 to 0.63. Although the absolute levels of metal contamination in bark and soil differed, the overall pattern of metal distribution was almost similar. The significant differences between the elements were demonstrated by the student t-test. The value of the t-test calculated in this study (P < 0.05) showed that all of the studied elements in both samples were significantly different. This strongly suggests that the presence of these particular elements in the studied area have been influenced by the same meteorological factors but contributed by different sources either naturally or artificially.

CONCLUSION

The results obtained in this study clearly showed that acacia tree bark is a good complementary bioindicator for soil. It was used to evaluate the effects of local industries on the spatial deposition of selected heavy metals in the Gebeng Industrial Area in Pahang. By combining heavy metal concentration data of tree bark and topsoil samples, this pilot study successfully revealed a wealth information about the origin of heavy metals deposited around the selected study area. By means of tree bark and soil samples, it was possible to study the spatial distribution of heavy metal deposition in the selected study area.

The CF value measured in this study clearly showed that almost all of the concentrations of the studied metals exceeded the normal concentrations of these metals as per the control samples of tree bark and topsoil.

This indirectly reflects the important role played by atmospheric deposition as a major source of metal accumulation in the surrounding bark and soil. In general, higher concentration of some elements in topsoil such as Al, Mn, V, and Cu indicates that bark is a less effective accumulator compared to soil but it is effective for accumulating Ni and Pb. Therefore, the preliminary data obtained from this study are important for future monitoring of the area, especially in terms of appropriate samples selection as a biological indicator.

The application of statistical analyses including Pearson correlation coefficient, contamination factor, and t-test showed that the primary sources of the elements studied in the study area were mainly dominated by the industrial activities especially those related to the work of rare-earth refinery processes, petroleum and related industries, and the use of fossil combustion. Meanwhile, the minor elements such as Mn were found to be contributed by re-suspended soil dust, construction dust, and agricultural activities.

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