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ORIGINAL ARTICLE

Trace elements in commercially available Triple Super Phosphate Fertilizers in Sri Lanka

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Abstract

This study tested the trace elements in commonly used Triple Super Phosphate (TSP) fertilizers in Sri Lanka. TSP samples collected from two Fertilizer Companies (A and B) were used. Six replicates of each company fertilizers were digested following the method of the Association of Official Agricultural Chemists. The concentration of eleven trace elements (Cr, Mn, Cu, Cd, Pb, Zn, Fe, Ni, Co, Mo and As) in each TSP fertilizers were determined by ICP-OES as an analytical technique. ICP-OES analysis revealed a group of non-lethal metal species, present as impurities. The highest average concentrations were, 4229.30 mg of Fe, 469.16 mg of Mn, 195.34 mg of Zn, 8.73 mg of Ni, 7.78 mg of Cu, 2.36 mg of Co and 1.39 mg of Mo per kg^{-1} of fertilizer. Considerable amount of hazardous elements such as Pb, Cr, As and Cd were detected with average concentrations of 53.93 mg of Pb, 25.56 mg of Cr, 3.19 mg of As, 1.05 mg of Cd per kg^{-1} of fertilizer, respectively. Trace metal concentrations of samples collected from two fertilizer companies were compared using two sample t–test ($\alpha = 0.05$). It was observed that, Mn, Cu and Zn were significantly higher in company A fertilizer samples than those of the company B. Concentrations of Cr, Cd, Pb, Fe, Co and Mo were significantly lower in company A fertilizer samples compared to samples of company B. However, there was no significant difference in concentrations of Ni and As contents in fertilizer samples of company A and company B. This study suggests the future course of action for the quality of the TSP fertilizer imported and the protocol or an act to regulate the standard limits of metal impurities present in TSP fertilizers imported to Sri Lanka.

Keywords: *Arsenic, ICP-OES, Sri Lanka, Trace elements, Triple supper phosphate*

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1. Introduction

Use of inorganic fertilizers and organic manure in agricultural field has been increased significantly over the last few decades in many developing countries including Sri Lanka to fulfill the food requirement for rapidly growing population. However, fertilizer is used primarily to increase the production (Ratnayake and Navaratna 2014). Government of Sri Lanka introduced fertilizer subsidy scheme for Sri Lankan farmers in 1962 intervening in fertilizer market. In 1988, fertilizer subsidy was removed from sulphate of ammonia and rock phosphate leaving the subsidy only for urea, triple supper phosphate (TSP) and muriate of potash (MOP). Fertilizer subsidy was restricted to only urea in 1997. General paddy fertilizer, which is a blend of urea, MOP and TSP, consists of phosphorous as the most abundant nutrient (Wijewardena 2006). The usual paddy fertilizer blend has the ratio 10: 27.4: 23 of N, P and K respectively (Dissanayake and Wijayatilleka 2000). Almost half the weight of a unit of paddy fertilizer is comprised of TSP and therefore, the quantity of TSP applied in rice fields is remarkably high and fertilizer subsidy applied to phosphate fertilizer were considered in the later subsidy schemes. For instance in 2005, Urea, TSP and MOP were available for the paddy farmers at a price of Rs. 350 per 50 kg under the "Kethata Aruna" fertilizer subsidy programme. Over 850,000 paddy farmers were estimated to be benefitted by this subsidy programme in 2009 (Ministry of Finance and Planning 2009). The aim of this fertilizer subsidy scheme is to provide fertilizer for farmers of Sri Lanka at an affordable price. However, it was negatively influenced on farmers to encourage for wider usage of fertilizer to crops. For example, Herath et al. (2015) reported that the urea, TSP and MOP usage were obviously increased with the implementation of fertilizer subsidy in 2005 and fertilizer use by the farmers of the paddy sector depends on the price of fertilizer.

Phosphorus is one of the major plant nutrients, which is deficient in many agricultural soils in Sri Lanka where, continuous crop removal could be suspected as the main reason for the depletion. Granular form of Triple Super Phosphate is one of the popular Phosphate fertilizers imported to Sri Lanka and it is mainly used for vegetables and paddy. In 2012, the total amount of TSP imported to Sri Lanka was 1.08×10^6 kg (Jayasumana et al. 2015). *Eppawala* Rock Phosphate (ERP) fertilizer is Sri Lankan origin and mined from the *Eppawala* rock phosphate deposit, contained apatite ($Ca[PO_4]_3.OH,F,Cl$) as the major constituent mineral. Most fertilizer company in Sri Lanka utilizes ERP for their long term plantation crop fertilizer blends. Reasons for the importation of TSP fertilizer into Sri Lanka were high water solubility of it compared to the solubility of ERP and it is considered to be useful only on long term plantation crops.

Compared to the other fertilizers which are usually synthetic in nature, trace metals are naturally associated with the phosphate fertilizers and, they are incorporated into the soil unwittingly (Dissanayake and Chandrajith 2009). These trace elements may persist in the soil for long time depending on the nature of the trace elements and soil properties. Among the trace elements, Cd, As, Pb and Cr are considered as a potentially toxic elements to plant and human health. An acidic condition of soil increases the bioavailability of trace metals which may create potential risk of accumulation of trace elements in plants and human body through the food chain (Ratnayake and Navaratna 2014). Heavy elements such as As, Cd and Pb are shown to cause Kidney diseases. It is hypothesized that these heavy elements accumulated in the human body causes Chronic Kidney Disease of Unknown etiology (CKDu), which is prevailing mainly in the dry and intermediate zone in Sri Lanka (Jayatilake et al. 2013; Jayasumana et al. 2013). TSP is produced by chemical reaction with phosphoric acid and rock phosphate as raw materials. Different elements are associated with TSP fertilizers and it can be derived from natural ore contain impurities could remain in TSP after the acid dilution process (Ratnayake and Navaratna 2014) or contamination of TSP by elements may occur during the processing of fertilizer (Dissanayake and Chandrajith 2009; Ratnayake and Navaratna 2014; Mehmood et al. 2009). Among the inorganic fertilizers, phosphate fertilizers are the major source of contaminants as they may contain different trace metals such as As, Cd, Cr, Hg, Pb, Fe, Mn, Cu, Ni, Co, Mo, Zn etc. (Allowy 1990; Al-Attar et al. 2012; Khan et al. 2018; Thomas et al. 2012; Raven et al. 1998). Because of phosphate fertilizer is produced mainly through mining as oppose to the other synthetic fertilizers produced through chemical reactions. There were few studies conducted relevant to metal impurities present in TSP fertilizers available in Sri Lanka (Rathnayaka and Nawarathna 2014). Therefore, it is worth to test the trace elements contained in TSP fertilizers with different origins. This study focused on determining the presence and quantification of the trace elements used TSP fertilizers in Anuradhapura area of Sri Lanka.

2. Methodology

2.1Reagents

Analytical grade reagents and deionized water was used for the preparation of all reagents and to maintain the calibration standards. External calibration was prepared from multi-element stock solution (Cr, Mn, Cd, Cu, Pb, Zn, Co, Ni, Mo) of 10 mg L^{-1}, 100 mg L^{-1} of Fe solution and 1000 mg L^{-1} stocks solution of As. The calibration curves were prepared in range of concentration from 0.5 mg L^{-1} to 5.0 mg L^{-1} for Cr, Mn, Cu, Cd, Pb, Zn, Ni, Co and Mo, and from 5.0 mg L^{-1} to 50.0 mg L^{-1} for Fe and As.

2.2 Preparation of TSP fertilizer samples

Six samples of TSP fertilizers from each company A and B weighing 2 kg in same batch from different packs were randomly collected from local market of Anuradhapura district, Sri Lanka. These two companies distribute nearly 65% of total demand of TSP in the area. Identity of the two companies were kept undisclosed, hence they are denoted as A and B. The fertilizers were digested according to the methods recommended by the Association of Official Agricultural Chemists (AOAC) (Williams 1984). Two fertilizer samples (samples of company A and B) were well ground into fine powder and six replicates weighing 5 g each were dissolved in 10 ml of conc. HCl by in a 100 ml beaker and covered with a watch glass. Then the contents were boiled for approximately 30 min by using a hotplate and evaporated to near dryness. After cooling, 20 ml of 0.1 M HCl was added and, the contents were quantitatively transferred into 100 ml volumetric flask by filtering through Whatman No. 2 filter paper. The residues were thoroughly washed with 0.1 M HCl and the volume was adjusted with the same solution to 100 ml. Each 10 ml of filtered fertilizer stock solutions were added into labeled ICP-OES plastic tubes. The amount of 50 µl of conc. HNO_3 was added each sample before analysis.

2.3 Instrumentation

Inductively couple plasma optical emission spectrometer (ICP - OES, iCAP 7000, United States of America) with axial and radial view was used to analyze the trace metals concentration in TSP fertilizer samples. The specifications of the operating conditions were as follows. Purge Gas Flow is normal and Radio frequency power, Auxiliary Gas Flow, Coolant Gas Flow, Nebulizer Gas Flow and Pump Speed were 1150 W, 0.50 l/min, 12 l/min, 0.5 l/min, 50 rpm respectively.

2.4 Data analysis

Minitab statistical software V.18 was used for data analysis. Two sample t–test with $\alpha = 0.05$ was used for the comparisons.

3. Results and discussions

Table 1 exhibits the maximum, minimum and average trace elements concentrations of the products. The results show that TSP fertilizers were comprised of essential plant micro nutrients such as Fe, Mn, Zn, Ni, Cu, Co, Mo and harmful hazardous elements such as Pb, Cr, As, Cd. Results also show that the average concentrations of individual trace metals varied considerably within the samples and between samples. Fe, Mn, Zn, Ni, Cu, Co, Mo, Pb, Cr, Cd were consistent throughout the series and As levels showed considerable variability within the sample series (Figure 1). Souza et al. (2014); Santos et al. (2013) and Momen et al. (2007) reported that ICP-OES technique is not usually applicable for the determination of As, Cd and Pb in low concentrations, but depending on the concentration of the analysts in the sample and the optimization of the digestion procedure, minimizing the dilution, it is possible to perform the determination with sufficient accuracy. Metal concentrations of Mn, Cu and Zn were significantly higher in company A fertilizer samples than those of the company B. Metal concentrations of Cr, Cd, Pb, Fe, Co and Mo were significantly lower in company A fertilizer samples than those of the company B fertilizer samples. However, there was no significant difference in the concentration of Ni and As contents in both products (Table 2). Fertilizer A is better for environment than fertilizer B due to contamination of fewer amounts of impurities. These trace elements concentrations do not exceed the maximum allowed regulatory limits in USA and Canada (Molina et al. 2009). However, Sri Lanka is yet to set regulatory upper limits for the trace elements present in fertilizers. Fe was the most abundant trace metal in both fertilizers: 3719.58 mg kg^{-1} in A and 4739.02 mg kg^{-1} in B fertilizers. Overall average Fe concentration was 4229.30 mg kg^{-1} and this value was lower than TSP used in Chile country (6003 mg kg^{-1} of fertilizer) (Molina et al. 2009). All phosphorous fertilizers had high Fe contents because Fe is one of the most abundant elements in the earth's crust (Molina et al. 2009). In terms of environment, Fe is considered as a less important element, because it is found in high concentrations in soil. However, it is considered as a micronutrient for plants. TSP is kind of rock phosphates obtained from reaction with phosphoric acid and it incorporated with Fe containing minerals such as haematite (Fe_2O_3) and magnetite

(Fe_3O_4) or Fe rich top soil with naturally (Ratnayake and Navaratna 2014).

Table 1: Maximum, minimum and mean trace element concentrations (mg kg^{-1}) in TSP samples.

Company A	**Cr**	**Mn**	**Cu**	**Cd**	**Pb**	**Zn**	**Fe**	**Ni**	**Co**	**Mo**	**As**
Maximum	23.94	541.72	8.58	0.84	41.46	207.12	4040.18	8.84	2.34	1.02	3.29
Minimum	23.4	533.82	8.38	0.8	38.78	202.1	3490.46	8.66	2.28	1	3.04
Mean	23.69	538.53	8.46	0.82	40.45	205.09	3719.58	8.73	2.32	1.02	3.16
Company B	**Cr**	**Mn**	**Cu**	**Cd**	**Pb**	**Zn**	**Fe**	**Ni**	**Co**	**Mo**	**As**
Maximum	27.68	402.18	7.18	1.3	68.72	187.9	4920.62	8.88	2.42	1.8	3.3
Minimum	27.24	397.44	7.04	1.26	66.3	184.16	4614.12	8.6	2.38	1.7	3.11
Mean	27.43	399.79	7.1	1.28	67.41	185.58	4739.02	8.72	2.39	1.75	3.21
Overall of company A and B	**Cr**	**Mn**	**Cu**	**Cd**	**Pb**	**Zn**	**Fe**	**Ni**	**Co**	**Mo**	**As**
Mean	25.56	469.16	7.78	1.05	53.93	195.34	4229.3	8.73	2.36	1.39	3.19

Overall average Cd concentration was recorded as a 1.05 mg kg^{-1} and it was the lowest concentration among the elements tested. Cd levels ranged between 0.8 – 1.3 mg kg^{-1}. This value was higher than the values (0.6 – 0.8 mg kg^{-1}) reported by Rathnayake and Nawarathna (2014) who used atomic absorption spectrometry (AAS) as the analytical method. The maximum acceptable levels of Cd in phosphate fertilizer is 4 mg kg^{-1} and the results of this study showed an average tested value (1.05 mg kg^{-1}) was below the acceptable level (Bandara et al. 2010; Jayatilake et al. 2013). Cd naturally occurs in soils at levels up to about 20 ppm (Ratnayake and Navaratna 2014). The overall average Cr concentration was recorded as a 25.56 mg kg^{-1} and series were ranging between 23.4 – 27.68 mg kg^{-1}. These trace heavy metals such as Cd and Cr present in fertilizers are influenced to biota than those amount bound to soil particles (Sager, 1997). Sri Lankan farmers usually apply the fertilizer (especially phosphate fertilizer) in bulk quantities and, over application of fertilizer is common (Ratnayake and Navaratna 2014; Dissanayake and Chandrajith 2009). Excessive application of phosphate fertilizer may increase the trace metals contents in the soil. This may lead to environmental issues and pose threats to human health. For example, annual average TSP usage for production of high-yield corn (*Zea mays* L.) in Chile was around 65 kg P ha^{-1} and 17.5 g Cd ha^{-1} released to the soil as an input (Molina et al. 2009). Total TSP usage for paddy cultivation in *Yala* season in Sri Lanka was around 23.4 kg P ha^{-1} under fertilizer in-kind subsidy in 2018 (Total cultivated extent was 332,971 ha and Total distributed TSP fertilizer quantity was 17199 Mt) and 0.055 g Cd ha^{-1} released to soil as an input. Furthermore, 1.3 g Cr ha^{-1} released into the paddy soil under same subsidy scheme in *Yala* season (Ministry of Finance and Planning 2018). The World Health Organization (WHO) has announced Cd as a major contributor for CKDu, prevalent in the North Central Province in Sri Lanka. According to the WHO tolerance limits, Cd and Cr in drinking water are 3 and 50 ppb (World Health Organization 2011). The average total Cd content of cultivated soil in Europe is 0.5 mg/kg (Finnish Environment Institute 2000) and permissible levels of Cd and Pb in rice grains are 0.4 mg kg^{-1} and 0.2 mg kg^{-1} respectively (World Health Organization 2011; World Health Organization 2016). The application of highly soluble TSP is common in paddy cultivated area in Sri Lanka and addition of Cd into the soil may cause emergence of diseases with agriculture communities in the country (Ratnayake and Navaratna 2014). For instance, high number of kidney disease affected patients have been reported in Sri Lanka where paddy is continuously grown and P based fertilizers are commonly applied (Jayasumana et al. 2013; Wimalawansa 2014; The Ministry of Health 2009). Heavy metals concentration is very low in organic manure widely used in Sri Lanka. Therefore, application of organic manure to the soil will reduce the incorporation of heavy metals in to the soil. The government of Sri

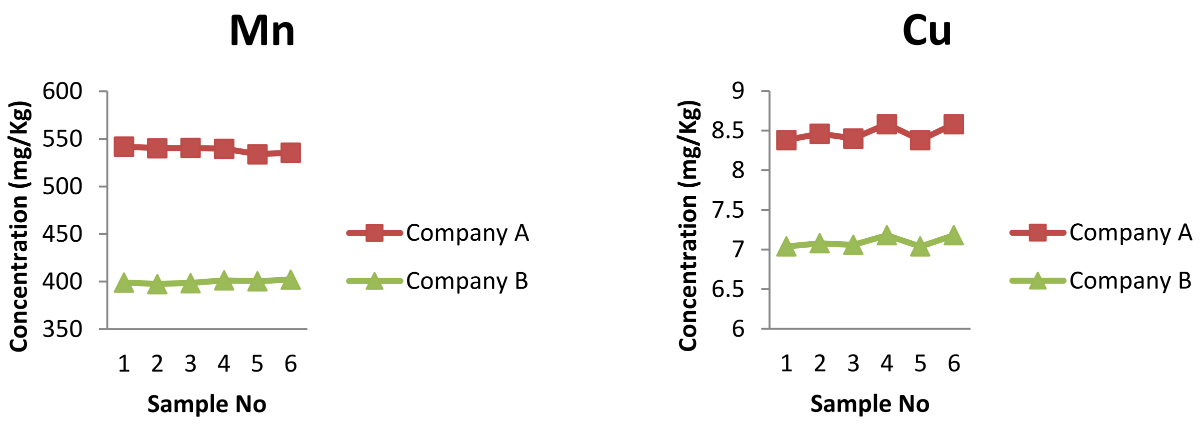
Lanka and the Department of Agriculture in Sri Lanka introduced several steps to ensure sustainable P management in agriculture. The major steps included introduction of fertilizer act to regulate fertilizer use including phosphorous based fertilizer, methods to control fertilizer adulteration, the installation of the National Fertilizer Secretariat and introducing major role as a sustainable phosphorous management strategies, the establishment of the Fertilizer Advisory Committee, upgrade of straight fertilizer use, introduction of soil testing-based fertilizer recommendations, periodic change of P fertilizer recommendations, research attempts to replace TSP by ERP for all possible crops and the introduction of P application in alternate seasons at a pilot scale for rice (Wijewardena 2006; Sirisena et al. 2013). Despite all these steps being taken, farmers will not adopt them at the field level and they continue to apply high rates of P than recommended amounts (Kendaragama et al. 2001; Sirisena et al. 2013). Therefore, it is worth to empower farmers by upgrading their knowledge and change their decision making process.

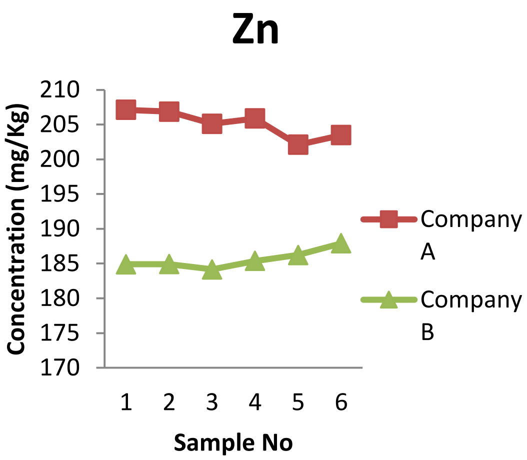
Studies on the assessment of As in fertilizer is rare in Sri Lanka probably due to the presence in small quantities in fertilizer as an impurity and hard to detect by some analytical instruments. The average As concentration in TSP that was tested in the study was 3.19 mg kg^{-1}. A study by Jayasumana et al. (2015) reports it as 31 mg kg^{-1}, which is a large value compared to the results of this study. According to the same study, when compared the As content of the synthetic and natural fertilizers available in Sri Lanka, the highest As content was detected in TSP fertilizer and TSP was the rich source of other heavy metals as well. The amount of As content in rice grains produced in Sri Lanka ranged from around 0.012 – 0.54 mg kg^{-1} and average contained 0.12 mg kg^{-1} (n = 154). According to the World Health Organization (2011 and 2016) data, permissible level of As in rice grain is 0.2 mg kg^{-1} and As content in certain rice grains samples were exceeded the permissible level (Rowell et al. 2014; Jayasumana et al. 2015b; Suriyagoda et al. 2018). When consider the country Chile, 6.4 g ha^{-1} of As is released into the soil as a source of TSP fertilizer (~65 kg P ha^{-1}) (Molina et al. 2009). However, most developed counties in the world were maintaining the standards to limit the entry of trace metals into their soils. For example, Canada has been maintained maximum content of As, Cd, Co, Ni, Mo, Pb and Zn in fertilizers and maximum amounts of these trace metals in cultivated soils over a period of 45 years (Canadian Food Inspection Agency 1997). Moreover, the American Association of Plant and Food Control Officials (AAPFCO 2007) also set standards for the concentration of nine trace metals in P fertilizers. According to published literature, there is no standard or limit of impurities defined for the imported TSP in Sri Lanka. Therefore, it is suggested to form a standard and also close monitoring of the impurities of all other TSP sources brought into Sri Lanka. This study tested only two have more impurities.

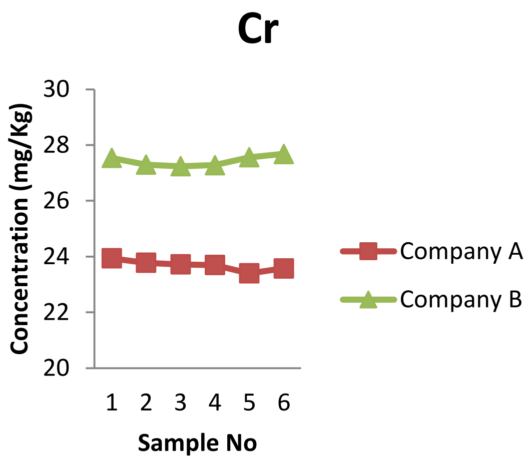
Table 2: Comparison of mean trace elements concentrations (mg kg-1) of TSP samples. Significant differences are indicated by * for *P*-value<0.05

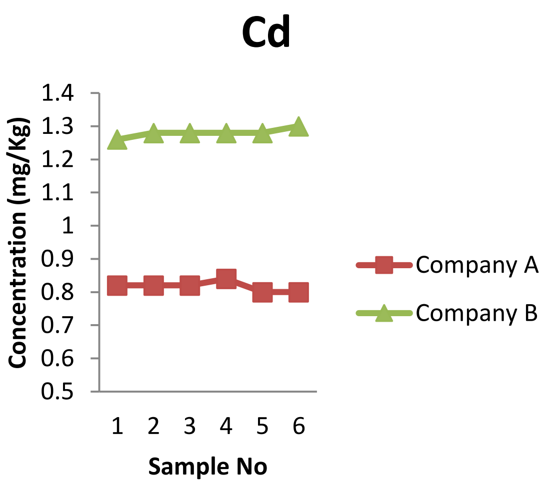
Element	Metal concentration (mg kg-1) Mean (± SEM)		P value
	Company A	Company B	
Mn	538.53(1.3)	399.79(0.74)	0*
Cu	8.46(0.039)	7.1(0.027)	0*
Zn	205.09(0.8)	185.58(0.54)	0*
Cr	23.69(0.075)	27.43(0.075)	0*
Cd	0.82(0.0061)	1.28(0.0052)	0*
Pb	40.45(0.42)	67.41(0.37)	0*
Fe	3719.58(88)	4739.02(53)	0*
Co	2.32(0.0095)	2.39(0.0067)	0*
Mo	1.02(0.0033)	1.75(0.014)	0*
Ni	8.73(0.028)	8.72(0.045)	0.808
As	3.16(0.04)	3.21(0.031)	0.283

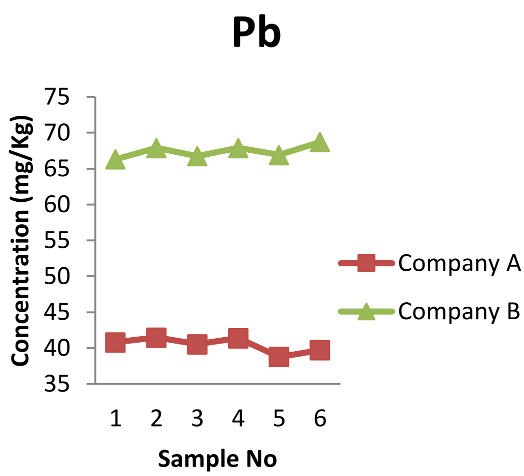
*$P < 0.05$, significant.

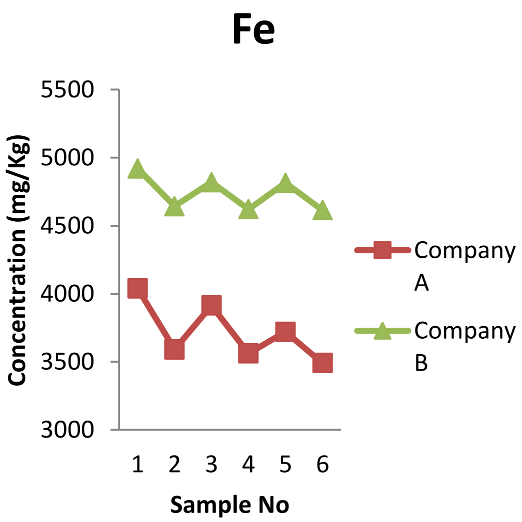


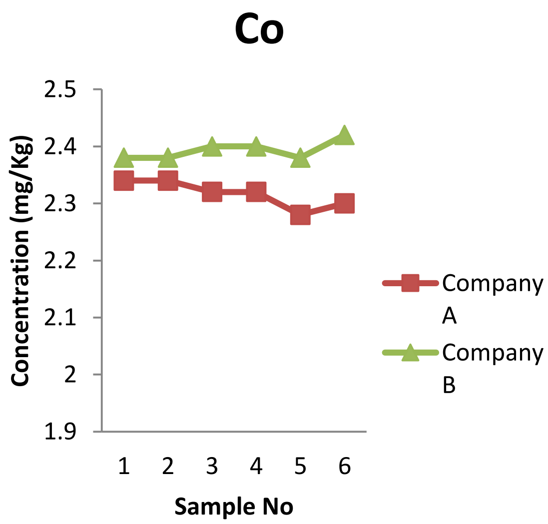

Zn
Concentration (mg/Kg)
210
205
200
195
190
185
180
175
170
1 2 3 4 5 6
Sample No
Company A
Company B

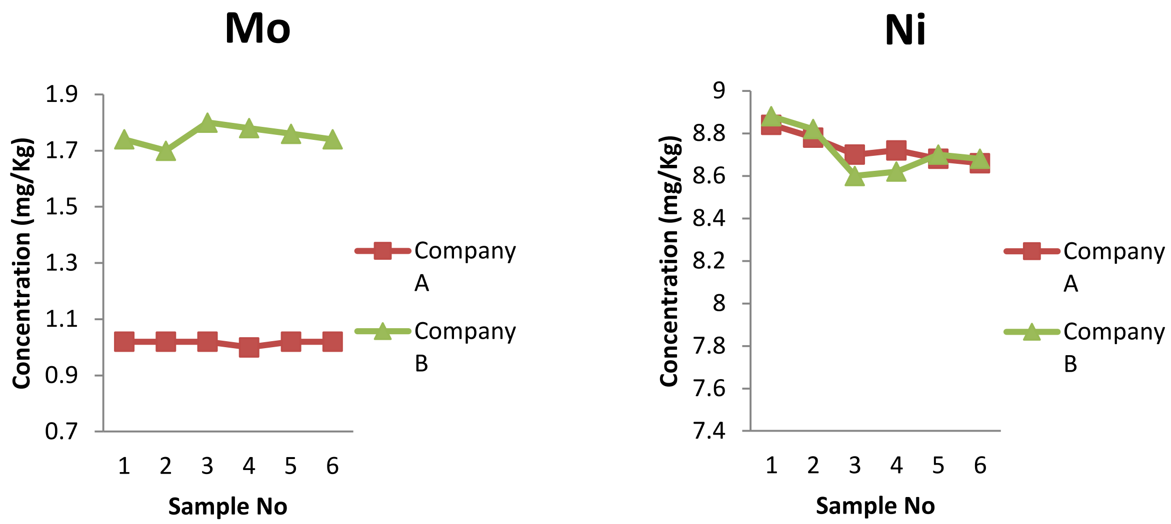

Cr
Concentration (mg/Kg)
30
28
26
24
22
20
1 2 3 4 5 6
Sample No
Company A
Company B


Cd
Concentration (mg/Kg)
1.4
1.3
1.2
1.1
1
0.9
0.8
0.7
0.6
0.5
1 2 3 4 5 6
Sample No
Company A
Company B


Pb
Concentration (mg/Kg)
75
70
65
60
55
50
45
40
35
1 2 3 4 5 6
Sample No
Company A
Company B


Fe
Concentration (mg/Kg)
5500
5000
4500
4000
3500
3000
1 2 3 4 5 6
Sample No
Company A
Company B


Co
Concentration (mg/Kg)
2.5
2.4
2.3
2.2
2.1
2
1.9
1 2 3 4 5 6
Sample No
Company A
Company B



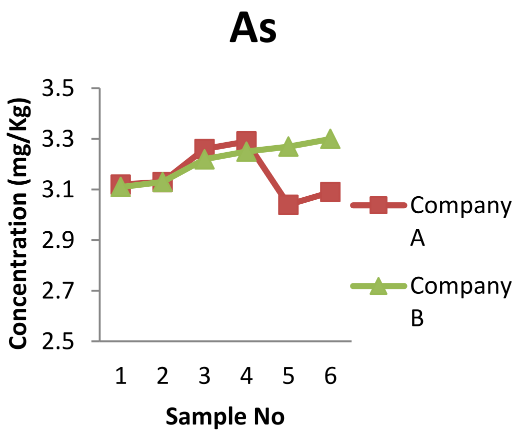


Figure 1: Uneven distribution of trace elements in TSP fertilizer samples of company A and B.

4. Conclusions

Triple Super Phosphate is one of the major phosphorus fertilizers imported to Sri Lanka and incorporated with different trace elements such as Cr, Mn, Cu, Cd, Pb, Zn, Fe, Ni, Co, Mo, and As. However, Cu, Zn, Fe, Co and Mo are trace elements essential for plant growth. Therefore, TSP provides some trace elements other than P for plant growth. Fe concentration in the tested two fertilizer products showed the highest metal concentration in TSP fertilizer. Analysis through ICP-OES revealed a group of hazardous elements such as Pb, Cr, As and Cd are present in the fertilizer. Metal concentrations of Mn, Cu and Zn were significantly higher in company A fertilizer samples than those of the company B fertilizer samples. Metal concentrations of Cr, Cd, Pb, Fe, Co and Mo were significantly lower in company A fertilizer samples than those of the company B fertilizer samples. This study suggests the future course of action for the quality of the TSP fertilizer imported and protocol or an act to regulate the standard limits of metal impurities present in TSP fertilizers imported to Sri Lanka.

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