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Trace Elements in Fertilizers Used in Peru

[1]Víctor Gómez, [2]Mauricio Molina-Roco, [3]Elder Chichipe and [4]Eli Morales Rojas

[1]*Department of Science for Sustainable Development, Graduate School, National University Toribio Rodriguez de Mendoza, Chachapoyas (UNTRM), Amazonas, Peru*
[2]*Department of Aquaculture and Agri-Food Resources, Universidad de Los Lagos, Osorno 5311157, Chile*
[3]*Department of Soil and Water Research Laboratory (LABISAG), Research Institute for the Sustainable Development of Ceja de Selva (INDES-CES), Universidad Nacional Toribio Rodríguez de Mendoza, Chachapoyas, Amazonas, Peru*
[4]*Department of Natural and Applied Sciences, Universidad Nacional Intercultural Fabiola Salazar Leguía de Bagua (UNIFSLB), Bagua 01721, Perú*

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Corresponding Author:
Eli Morales Rojas
Department of Natural and Applied Sciences, Universidad Nacional Intercultural Fabiola Salazar Leguía de Bagua (UNIFSLB), Bagua 01721, Perú
Email: emorales@unibagua.edu.pe

Abstract: Fertilizers provide essential nutrients for crops, however, they may also inadvertently provide trace elements that in certain concentrations represent a risk to the soil and the food chain. The objective of this research was to determine the concentrations of Cd, Pb, Cu, and Zn in the most common fertilizers used in Peruvian agriculture. Twenty-one products including inorganic, organic mineral, and organic fertilizers were analyzed by microwave plasma atomic emission spectrometry. The results were compared against international standards. The content of the trace elements was variable and related to the type of fertilizer. Phosphorus fertilizers showed higher concentrations of Cd (3.3-42.3 $mgkg^{-1}$), most of which exceeded the regulatory limits of several countries. In the case of Pb, none of the fertilizers analyzed exceeded the regulatory limits. Chicken manure showed the highest concentrations of Cu and Zn. The concentration of trace elements such as Cd in fertilizers should be monitored in Peru, since the prolonged and intensive use of phosphorus fertilizers in doses that do not consider technical recommendations, could represent an accumulation of these elements in soils, with the consequent risk to human health and the environment.

Keywords: Fertilizers, Trace Metals, Soil Contamination, Fertilizer Quality

Introduction

Fertilizers are a source of essential nutrients for agriculture and their use has increased in recent years (Savci, 2012). These compounds provide elements such as Nitrogen (N), Phosphorus (P), Potassium (K), Sulfur (S), and Magnesium (Mg) among others, that are essential for crop development (Leye Samuel and Omotayo Ebenezer, 2014). According to specialists in Peru, the most commonly used fertilizers are Urea, ammonium nitrate, diammonium phosphate, and potassium sulfate (Lluzar Martí, 2019). However, fertilizers can also be an important source of Trace Elements (TEs) such as Cadmium (Cd), lead (Pb), Copper (Cu), and Zinc (Zn) (Jiao *et al*., 2012).

Cd and Pb are non-essential metals for plants and can cause damage to living organisms if certain concentration thresholds are exceeded (Pierzynski *et al*., 2005), while Cu and Zn are essential for biological functions but also trigger toxic effects at high concentrations. Several studies have shown that the application of fertilizers and amendments is one of the main causes of TEs accumulation in the soil (Mann *et al*., 2002; Nicholson *et al*., 2003; Xiaobing *et al*., 2020). In this sense, there is concern about possible long-term adverse effects, since these elements are not biodegradable and their bioaccumulation in soils, their uptake by plants, and their passage into the food chain may represent a serious risk (Hu *et al*., 2014). Cd has been one of the elements of greatest concern because, in general, its mobility in soil and transfer to plants is higher in comparison to other TEs. In addition, Cd is found in higher concentrations in phosphorus fertilizers (Latifi and Jalali, 2018; Molina *et al*., 2009; Nziguheba and Smolders, 2008). This fertilizer is derived from phosphate rock as raw material, a mineral whose TEs concentration is generally higher than those found in the earth's crust (Kratz and Schnug, 2006; McBride and Spiers, 2001).

Cd can seriously affect health if the tolerable weekly intake (2.5 µg/kg body weight) is exceeded, as this element is associated with an increased risk of lung, endometrial, bladder, and breast cancer (EFSA, 2012a). On the other hand, Pb accumulates in the body and can severely affect the central nervous system in the developmental stages of children (Flora *et al*., 2012). For this element, there is no recommended tolerable intake level as there is no evidence of thresholds for a range of critical health effects (EFSA, 2012b). The presence of TEs is an environmental concern in fertilizers; however, in Peru, there is still no related research, nor have regulatory limits been established for these agricultural inputs.

The objective of this study was to determine the concentrations of Cd, Pb, Cu, and Zn in selected fertilizers commonly used in agricultural soil in Peru and to compare the results with existing international standards.

Materials and Methods

Fertilizer Sampling

During 2020, samples of one kilogram of 21 fertilizers were randomly collected from commercial agrochemical companies in the city of Chachapoyas, Amazonas region. Among the fertilizers, 17 were of inorganic nature, three of organo mineral nature, and one organic material. The most widely used fertilizers in Peru were incorporated into the sampling (Lluzar Martí, 2019). Each material was homogeneously mixed before analyses (Table 1).

Determination of Cd, Pb, Cu, and Zn

The digestion process of fertilizers samples was carried out according to method 3050B (USEPA, 1996). Briefly, fertilizers were crushed with a mortar and pestle to pass a 0.5-mm sieve and 1 g of each was weighed. 10 mL of a 1:1 v/v of concentrate Nitric Acid (HNO_3) and ultrapure water solution was added to the sample in a glass digestion tube and the samples were heated at 95±5°C for 15 min. After cooling for 10 min, 5 mL of concentrated HNO_3 was added and heated for another 30 min. After cooling for 10 min the sample was heated at 95±5°C without boiling for two hours. The was filtered and made up to 25 mL with ultrapure water. Given the presence of organic material, in the case of organic mineral and organic fertilizers, 2 mL of ultrapure water and 3 mL of Hydrogen Peroxide (H_2O_2) were added additionally during the last heating period. If necessary, the addition of H_2O_2 was repeated in 1 mL aliquots until no effervescence was recorded. After digestion of the samples, Cd and Pb concentrations were determined in a Microwave Plasma Atomic Emission Spectrometer (MP-AES) at the soil and water research laboratory (LABISAG) of the research Institute for Sustainable Development of Ceja de Selva (INDES-CES) National University Toribio Rodríguez de Mendoza of Amazonas (UNTRM).

Quality Control

All fertilizers were analyzed in triplicate calibration curves and were performed using ultrapure water and trace-quality standard reagents. Three readings of each sample were performed on the MP-AES equipment and reference solutions were analyzed for every five samples. Limits of Detection (DL) were calculated using 10 blanks. Soil samples spiked with Cd, Pb, Cu, and Zn (1 and 5 $mgkg^{-1}$ for Cd and Pb and 5 and 20 $mgkg^{-1}$ for Cu and Zn), were used as reference materials. Table 2 the, detection limits for each TEs and recovery percentages for each standard material are presented.

Table 1: Fertilizers analyzed, nature and nutrients provided according to their datasheet

N°	Fertilizer	Number of simples*	Nature	Nutrients provided
1	Potassium chloride	2	Inorganic	K
2	Ammonium nitrate	1	Inorganic	N
3	Potassium nitrate	1	Inorganic	N y K
4	Ammonium sulfate	2	Inorganic	N y S
5	Magnesium sulfate heptahydrate	1	Inorganic	Mg y S
6	Potassium sulfate	2	Inorganic	K y S
7	Potassium magnesium sulfate	1	Inorganic	K, S y Mg
8	Urea	2	Inorganic	N
9	Controlled Release Fertilizer (CRF)	1	Inorganic	N, P, K, Mg y S
10	Di-ammonium phosphate	2	Inorganic	P y N.
11	Mono ammonium phosphate	1	Inorganic	P y N
12	Triple calcium superphosphate	1	Inorganic	P y Ca
13	Phosphate rock with island manure	1	Organ mineral	P, Ca, K, S, etc.
14	Phosphate rock with organic matter	1	Organ mineral	P y Ca
15	Island manure	1	Organo mineral	N, P, K. Ca, Si y S
16	Poultry manure	1	Organic	Not specified

*2 = samples of different brands of the same fertilizer; 1 = one single fertilizer sample

Table 2: Limit of detection recovery percentages of the TEs analyzed

		Percentage of recovery for each standard		
Element	Limit of detection ($mg.kg^{-1}$)	1 $mg.kg^{-1}$ (%)	5 $mg.kg^{-1}$ (%)	20 $mg.kg^{-1}$ (%)
Cd	0.13	92.3-97.3	90.4-97.2	---
Pb	0.61	90.7-97.7	90.1-99.1	---
Cu	2.39	---	93.4-98.6	94.2-99.5
Zn	0.13	---	91.4-98.0	92.3-97.9

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Statistical Analysis

Descriptive statistics (mean, median, standard deviation, coefficient of variation) were performed for the Cd, Pb, Cu, and Zn content of the fertilizers; the data were analyzed in the Minitab 19 statistical software. For each TE and each type of fertilizer, the fertilizer with the highest concentration of these contaminants was identified. Fertilizers were separated into three groups according to their nature: Inorganic phosphorus, inorganic non-phosphorus, and organo mineral (this group included chicken manure, the only organic fertilizer analyzed). In the case of Cd for phosphorous fertilizers, the values of concentration per kg of fertilizer (Cd) were concentration per kg of fertilizer (mgkg^{-1}) were converted to concentration per unit of P (mgkg^{-1} of P_2O_5) as required by the regulations of the various countries studied.

Results

The TEs concentration varied markedly, depending on the type of fertilizer. Tables 3-5 show the range, mean, median, standard deviation, and coefficient of variation of TEs concentration in the 21 fertilizers analyzed, grouped according to their nature. Table 3 shows the results for the non-phosphorous inorganic fertilizers. The highest concentration of Pb was evidenced in urea with a mean of 2.9 mgkg^{-1}. These fertilizers present very low mean concentrations of TEs, with various fertilizers showing elemental contents below the limits of detection.

Table 4, shows the results for the phosphorous inorganic fertilizers. It was evidenced the highest mean was 117 mgkg-1 for Zn, belonging to diammonium phosphate fertilizer (2), while the lowest mean was Cd a mean of 6.7 mgkg-1 in the same diammonium phosphate (2).

Table 5 shows that the highest concentration in the organic mineral fertilizers was for Zn with 144 mgkg^{-1} corresponding to the poultry manure fertilizer. However, the organo mineral fertilizers differed in that they did not show values for Pb.

Table 6 shows the percentages of phosphorus heavy metals (inorganic and organo mineral). Where Cd was compared with cadmium in 13 countries, the highest cadmium values with respect to the Maximum Permissible Limit (MPL) were found in Austria, France, Belgium, and Australia. However, cadmium values in organic and organo mineral fertilizers exceeded the MPL for phosphate rock with island guano with a value of 269.6 mgkg^{-1} P_2O_5. The results for lead did not exceed the MPL of the countries consulted.

Table 3: Concentration range of non-phosphorus inorganic fertilizers (n = 12)

Trace element	Range* (mg kg^{-1})	Media (mg kg^{-1})	Median (mg kg^{-1})	SD (mg kg^{-1})	CV (%)	Fertilizer with higher concentration
Cd	< D.L	--	--	--	--	--
Pb	< D.L.- 4.5	2.9	0.7	4.4	152	Urea
Cu	< D.L.-5.8	<D.L.	< D.L	1.6	336	Ammonium nitrate
Zn	< D.L.-8.4	1.6	< D.L	2.6	164	Urea

*For the calculation of the statistics, concentrations below the D.L were considered zero

Table 4: Concentration of heavy metals in inorganic phosphorous fertilizers (n = 5)

Trace element	Range* (mg kg^{-1})	Media (mg kg^{-1})	Median (mg kg^{-1})	SD (mg kg^{-1})	CV (%)	Fertilizer with higher concentration
Cd	<D.L.-18.3000	6.7	3.3	6.4	96.7	Diammonium phosphate (2)
Pb	<D.L.-11.1000	6.7	7.7	4.3	63.8	Diammonium phosphate (1)
Cu	<D.L.-229000	58.9	36.6	85.1	144.0	FLC
Zn	44.81-259.96	117.0	74.0	81.9	69.9	Diammonium phosphate (2)

*For the calculation of the statistics, concentrations below the D.L were considered as zero; 2 and 1 refer to the type of fertilizer

Table 5: Heavy metal concentrations in organo-mineral fertilizers

Trace element	Range* (mg kg^{-1})	Media (mg kg^{-1})	Median (mg kg^{-1})	SD (mg kg^{-1})	CV (%)	Fertilizer with higher concentration
Cd	<D.L-42.3	16.3	11.8	16.3	99.6	Phosphate rock with island manure
Pb	<D.L.	-	-	-	-	-
Cu	17.7-46.9	36.5	41.4	11.2	30.8	Poultry manure
Zn	44.6-326	144.0	110.0	107.5	73.8	Poultry manure

*For the calculation of the statistics, concentrations below the DL. Were considered zero

Table 6: Cd concentration in phosphorus fertilizers (inorganic and organo-mineral) compared to Cd limits in phosphorus fertilizers and Pb limits in fertilizers in different countries

N° Fertilizer	1 Di-ammonium phosphate 1	2 Di-ammonium phosphate 2	3 Mono-ammonium phosphate	4 Triple-calcium superphosphate	5 Phosphate rock with island manure	6 Phosphate rock with organic matter	7 Island manure
Cd (mg kg^{-1})	3.0	17.5.0	3.3	9.6	41.8	14.5	9.1
% P_2O_5	46.0	46.0.0	52.0	46.0	15.5	18.7	10.0
Cd (mg kg^{-1} P_2O_5)	6.7	37.9.0	6.3	20.9	270	77.5	90.7
Country	Limit for Pb (mg kg^{-1})	Limit for Cd (mg Cd kg^{-1} P_2O_5)			Reference		
Hungary	---	20.0			European Parliament (2017)		
Finland	100	22.0			European Commission (2016)		
The Netherlands	---	31.3			European Parliament (2017)		
Sweden	100	44.0			European Commission (2016)		
Denmark	---	48.0			European Parliament (2017)		
Germany	150	50.0			DüMV (2008)		
Italy	---	50.0			European Parliament (2017)		
Czech Republic	30	50.0			European Commission (2016)		
Greece	---	60.0			European Parliament (2017)		
Austria	---	75.0			European Parliament (2017)		
France	150	90.0			European Commission (2016)		
Belgium	---	90.0			European Parliament (2017)		
Australia	500	131.0			JADE (2015)		

Discussion

There is growing concerned about TEs entering soils through fertilizers (Latifi and Jalali, 2018). Establishing regulatory limits for TEs in these products of intensive use in agriculture would be a way to minimize risks. In Peru, regulatory limits for fertilizers have not yet been established, thus in this study, the comparisons were made using international regulations. In most countries, the limit for Cd is expressed by a unit of P_2O_5 given P fertilizer applications are calculated according to the P_2O_5 content (%). If the most demanding regulatory limit for Cd (Hungary: 20 mg Cd kg^{-1} P_2O_5) were considered, only two of the seven phosphorus fertilizers analyzed would comply with the regulations. On the contrary, if the reference were the least demanding regulation for Cd (Australia: 131 mg Cd kg^{-1} P_2O_5), six of the fertilizers analyzed would be within the required limits. One diammonium phosphate and triple superphosphate showed the highest Cd concentration per unit of P_2O_5. In the case of phosphate rock with island manure, the regulatory limits of all the countries for this element are exceeded, making it necessary to pay attention to the use of this product in agriculture.

In the case of Pb, the concentrations in all types of fertilizers analyzed did not exceed the limits proposed in the regulations of the countries consulted. For this element, the most demanding legislation establishes a concentration of 30 mgkg-1 (Czech Republic) and the maximum concentration reported was 14.5 mg.kg-1 in urea. In general, the limits established for Pb are higher than those for Cd because the former is less mobile in the soil (Pierzynski *et al.*, 2005), so there is less risk of being uptaken by plants and therefore entering into the food chain.

As observed in Tables 3-5, the variability of TEs concentration, as expressed as the coefficient of variations, is high, indicating variability between each fertilization type within groups and showing that distribution companies may use different suppliers with different origins of the materials. This may be the case for the two diammonium phosphate fertilizers analyzed (Table 6).

The fertilizers analyzed have variable concentrations of Cu and Zn, in the same way as for Cd and Pb, these elements were generally present in higher concentrations in the phosphorus fertilizers. However, two non-phosphorus fertilizers individually had the highest concentrations of these elements. Cu was present in higher concentration in the controlled release fertilizer with an average of 220 while mgkg^{-1} in the case of Zn the poultry manure (organic fertilizer) presented an average concentration of 311 mgkg^{-1}. Unlike Cd and Pb, both Cu and Zn are classified as micronutrients in agriculture, since they are essential for plants. However, high concentrations of these could also cause certain problems. High concentrations of Cu can be toxic to organisms (Huseen and Mohammed, 2019).

The concentration of most TEs found in this research is related to the ranges found in other research from other countries for phosphorus fertilizers (Franklin *et al.*, 2005; Molina *et al.*, 2009; Nziguheba and Smolders, 2008; Vieira da Silva *et al.*, 2017). The results show that Cd concentrations are generally higher in fertilizers of a phosphorus nature than in other types of P fertilizers, these results are in agreement with other research (Latifi and Jalali, 2018; Molina *et al.*, 2009; Nziguheba and Smolders, 2008; Verbeeck *et al.*, 2020). This type of fertilizer comes from phosphate rock as raw material (Rauf *et al.*, 2002), a mineral in which TEs concentrations are generally higher than those found in the earth's crust (Kratz and Schnug, 2006; McBride and Spiers, 2001). In addition, about 85% of phosphate rocks for phosphorus fertilizer production are sedimentary in nature, these contain higher TEs content compared to those of igneous nature (Kratz *et al*., 2016). Therefore, regulation for TEs in phosphorus fertilizers especially for Cd has been of greater interest from an environmental perspective.

Cd concentrations in the analyzed inorganic phosphorus fertilizers were on average very similar to those reported by Franklin *et al*. (2005); Kratz *et al*. (2016); Latifi and Jalali (2018); Nziguheba and Smolders (2008); Rauf *et al*. (2002); Raven and Loeppert (1997); Verbeeck *et al*. (2020). Furthermore, the concentrations were on average lower than those reported by Molina *et al*. (2009). The phosphorus fertilizer with the highest Cd concentration was di-ammonium phosphate, a result similar to that reported by Raven and Loeppert (1997) in which diammonium phosphate is also one of the fertilizers with the highest Cd content.

Pb concentrations found in phosphorus inorganic fertilizers were on average similar to those reported by Latifi and Jalali (2018); Rauf *et al*. (2002); Raven and Loeppert (1997); lower than those reported by Molina *et al*. (2009) and higher than those reported by Franklin *et al*. (2005); Kratz *et al*. (2016), Nziguheba and Smolders (2008); Verbeeck *et al*. (2020). It is also important to take into account the evaluation of Pb content in fertilizers and its input to soils. Since Pb is considered a silent pollutant with a large number of toxic effects on human health (Karrari *et al*., 2012).

Cu concentrations found in phosphorus inorganic fertilizers were on average lower than those reported by Molina *et al*. (2009) but higher than those reported by Kratz *et al*. (2016); Latifi and Jalali (2018); Rauf *et al*. (2002); Raven and Loeppert (1997); Verbeeck *et al*. (2020). For Zn the concentrations found in phosphorus inorganic fertilizers were on average lower than those reported by Kratz *et al*. (2016); Latifi and Jalali (2018); Molina *et al*. (2009); Nziguheba and Smolders (2008); Rauf *et al*. (2002); Raven and Loeppert (1997); Verbeeck *et al*. (2020). Zn concentration was higher in diammonium phosphate, Raven and Loeppert (1997) also reported a higher concentration of this element in diammonium phosphate.

The results for Cd and Pb for non-phosphorus inorganic fertilizers were on average lower than those reported by Latifi and Jalali (2018); Rauf *et al*. (2002); Raven and Loeppert (1997). For Cu and Zn also the results in this type of fertilizers, were below the concentrations reported by Latifi and Jalali (2018). Only for Zn on average, the concentrations reported were lower than those found by Raven and Loeppert (1997).

Conclusion

The fertilizers analyzed concentrations of Cd, Pb, Cu, and Zn, with the phosphorus fertilizers having, in general, the four TEs. The concentrations of Cd in the phosphorus fertilizers in many cases exceeded the established limits. In the case of Pb, all the fertilizers presented concentrations below the suggested international standards. The concentration of TEs in fertilizers should be monitored in Peru, since the prolonged and intensive use of phosphorus fertilizers could represent a possible accumulation of these in soils, generating contamination, with the consequent risk to human health and the environment after they enter the food chain. In general, small and medium scale producers do not use the techniques recommended doses of fertilizers, and thus do not take into account the soil fertility analysis and plant needs. This fact may lead to an over-application of P fertilizers, increasing the risk of TEs soil buildup in the long term, altering the health and biological and chemical processes. The information obtained in this study can be used to calculate Cd, Pb, Cu, and Zn inputs in agrosystems where these fertilizers are used.

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Author’s Contributions

Víctor Gómez, Mauricio Molina-Roco and Elder Chichipe: Conceptualization, drafted and reviewed the final version.

Eli Morales Rojas: Manuscript written, data collection, analysis, and project management. Finally, all authors have read and accepted the final version of the manuscript.

Ethics

This article is original and contains unpublished material. The corresponding author confirms that all authors have read and approved the manuscript and that there are no ethical issues.

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