



Permissible ranges of Cadmium and Lead in organic manures used in food production

Rangos permisibles de Cadmio y Plomo en abonos orgánicos utilizados en la producción de alimentos

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ABSTRACT: In Cuba, in order to find national alternatives for agricultural production maintenance, the production and use of organic manures of diverse origins is promoted. However, it is important to maintain a systematic follow-up of their contents in heavy metals (HM), since they can contaminate the food produced with them; as well as to have permissible values of HM in organic manures, which are not currently available in the country. The objectives of this work were to evaluate the cadmium (Cd) and lead (Pb) content in organic manures used for vegetable production and to determine ranges of permissible values of these metals in organic manures. HM extraction in the samples of soils, organic manures, substrates and vegetables studied was carried out with 9 mL of HNO₃ and 3 mL of HCl using a microwave oven, according to USEPA 3051A methodology and determination by Optical Emission Spectrometry. The ranges 140-150 mg kg⁻¹ for Pb and 2-3 mg kg⁻¹ for Cd were established as permissible values for these metals in organic manures used in food production. Organic manures with high Cd and Pb contents cannot be used to prepare substrates for food production, especially vegetables, because they constitute contamination risks for human health.

Key words: heavy metals, compost, contamination.

RESUMEN: En Cuba, con el fin de encontrar alternativas nacionales para el mantenimiento de la producción agrícola, se potencia la producción y el uso de los abonos orgánicos de diversos orígenes. Sin embargo, es importante mantener un seguimiento sistemático de sus contenidos en metales pesados (MP), pues pueden contaminar los alimentos producidos con ellos; así como disponer de valores permisibles de MP en abonos orgánicos, con los que no se cuenta actualmente en el país. Los objetivos del trabajo fueron evaluar el contenido de cadmio (Cd) y plomo (Pb) en abonos orgánicos que se utilizan para la producción de hortalizas y determinar los rangos de valores permisibles de estos metales en los abonos orgánicos. La extracción de los MP en las muestras de suelos, abonos orgánicos, sustratos y hortalizas estudiadas se realizó con 9 mL de HNO₃ y 3 mL de HCl utilizando un horno microondas, según la metodología USEPA 3051A y la determinación por Espectrometría de Emisión Óptica. Se establecieron los rangos 140-150 mg kg⁻¹ para el Pb y 2-3 mg kg⁻¹ para el Cd como valores permisibles de estos metales en los abonos orgánicos que se utilizan en la producción de alimentos. Los abonos orgánicos que presenten contenidos elevados de Cd y Pb no pueden utilizarse para preparar sustratos destinados a la producción de alimentos, especialmente hortalizas, porque constituyen riesgos de contaminación para la salud humana.

Palabras clave: metales pesados, compost, contaminación.

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INTRODUCTION

The production in organoponics of Urban Agriculture is an alternative to a problem of high sensitivity for the population in underdeveloped countries, since it makes possible the supply of fresh vegetables, especially leaves and condiment plants throughout the year (1,2). In Cuba, the use of organic manures (OM) of diverse origins is considered an effective alternative as a new way for food production with low inputs (3,4). However, it is known that these organic materials could be a contaminant source of heavy metals (HM), so it is important to systematically monitor the content of these metallic contaminants, mainly cadmium (Cd) and lead (Pb). The effects caused by the addition of HM in soils and their influence on the development of plants and animals deserve man's attention because they endanger the planet survival (5,6).

Currently, there is an increasing interest in knowing the contents of HM in OM such as manure *compost* of different origins, filter cake (residue from the sugar industry), earthworm humus, chicken manure, bat guano, harvest residues, residual sludge, biosolids and *compost* obtained from various sources; as well as their risks to ecosystems and human health; which provides a measure of the potential danger for their use and management in food production (7). In this sense, the use of organic manures for the production of vegetables in agriculture requires the development of standards for their generation, collection, transport, treatment and use. There are few regulations in this regard in Cuba and there are none on the permissible values of HM in OMs.

The legislation elaboration in this regard requires a long process of research and legal basis that must be complied with. It is necessary to determine not only the permissible values in these materials, but also their maximum permissible loads, both annual and accumulated (8). However, the high demand of OM for food production in the country, mainly by the National Program of Urban, Suburban and Family Agriculture, the high toxicity of these HM (7) and that the permissible values of HM in organic manures are not yet defined in Cuba, justify the objective of the work to establish the ranges for Cd and Pb in organic manures used in food production, particularly in organoponics.

MATERIALS AND METHODS

The work was carried out from 2013 to 2018, at the Soil Institute of the Ministry of Agriculture, with support from the Basic Science and Technique Unit of the Soil Institute in Guantánamo, the Chemical Analysis Laboratory of the Center for Technological Applications and Nuclear Development (CEADEN) of the Ministry of Science Technology and Environment and the Rural Federal University of Pernambuco (UFRPE), Recife, Brazil.

Description of variants and sampling

For the study, beds 3 m long by 1 m wide were formed for each case, at the Soil Institute and at the Basic Science and Technology Unit of the Soil Institute in Guantánamo. The description of the variants is shown in Table 1.

Table 1. Variant characteristics.

Variant	Substratum composition	Crops	
		Leafy vegetables	Fruit vegetables
I	50 % soil Red Ferrallitic typical (SRFT) + 50 % organic manure (vermicompost)	Lettuce (<i>Lactuca sativa</i> , L. var. <i>BH-15</i>)	Pepper (<i>Capsicum annum</i> , L. var. <i>LPD-2</i>)
II	50 % SRFT + 50 % organic manure (compost of beef manure)	Chard (<i>Beta vulgaris</i> , L. var. <i>White Ribbed</i>)	Radish (<i>Raphanus sativus</i> , var. <i>Tropical PS-9</i>)
III	50 % SRFT + 50 % organic manure (compost of crop residues)		
IV	50 % SRFT + 50 % organic manure (compost of MSW, domestic waste).		
V	50 % soil Brown Sialitic fluffy (SBSF) + 50 % organic manure (compost of crop residues)		
VI	50 % SBSF + 50 % organic manure (compost of beef manure and vegetable residues)		
VII	50 % SBSF + 50 % organic manure (compost of beef manure)		
VIII	50 % SBSF + 50 % organic manure (compost of MSW, domestic trash**).		
IX	50 % soil Brown Sialitic calcic (SBSC) + 50 % organic manure (compost of MSW, crop residues***)		
X	50 % SBSC + 50 % organic manure (compost of beef manure)		
XI	50 % SBSC + 50 % organic fertilizers (MSW compost from household garbage**)		
XII	50 % SBSC + 50 % organic fertilizers (MSW compost from household garbage*)		

SFRT: typical red Ferrallitic soil, SBSF: fluffy Sialitic Brown soil and SBSC: calcic Sialitic Brown soil. MSW: Municipal solid waste. (*) MSW from CEPRU "Sur Isleta". (**)MSW from CEPRU "Los Cocos". (***) MSW from CEPRU "Vilonio".

Soils used to prepare variants were classified according to the Cuban Soil Classification (9) and they were taken from areas of small or secondary forests, with a history of not having been fertilized, nor used for agriculture and livestock; as well as being far from sources of contamination by HM in a period of 50 years or more, which guaranteed a minimum alteration by anthropic action. The municipal solid waste (MSW) *compost* from domestic garbage used as organic manures in variants IV, VIII, IX, XI and XII were obtained from MSW from the Municipal Solid Waste Processing Centers (CEPRU according the Spanish acronyms) "Sur Isleta" and "Los Cocos", which process classified domestic garbage before forming the donkeys for compost production, and from the CEPRU "Vilonio" which only processes harvest waste, in Guantánamo province. The cow dung compost corresponding to variant XI came from the organoponic "Las Margaritas", located in the Marianao, Havana municipality and the remaining organic manures from the Organic Fertilizer Production Center from Cotorro, in Havana.

In each substrate prepared, crops referred to in Table 1 were sown in succession in the following order (lettuce-chard and pepper-radish). Soils and organic manures were sampled at the time of substrate preparation, in a random manner. Substrate samples were taken at a depth of 0-20 cm at the beginning of sowing and vegetables at the time of harvest. Both substrates and plants were sampled in a zigzag pattern along each bed, according to the standard (10). In all cases, three composite samples of between 15 and 20 single samples each were taken. Soils, substrates and organic manures sampled were air-dried and passed through a 2 mm sieve according to the Cuban standard (11). Plant samples were washed, dried in an oven at 45 °C to constant weight and ground to 74 m size.

Extraction and determination of HM contents in soils, substrates, organic manures and plants

Extraction of the pseudo-total Cd and Pb contents in the soil, substrate, organic fertilizer and plant samples was performed using the procedure described in USEPA Standard 3051A (12). 0.50 g of each sample was weighed and added 12 mL of *aqua regia* invert, a mixture of pure nitric (HNO₃) and hydrochloric (HCl) acids for analysis (Merck PA), in a 3:1 ratio. A microwave oven was used for digestion (*Mars Xpress Microwave*), where, for soil, substrate and organic fertilizer samples, 10 minutes were set to reach a maximum temperature of 175 °C, 10 minutes for maintenance and 15 minutes for the cooling process; while for plants, 15 minutes were set to reach a maximum temperature of 180 °C, 10 minutes for maintenance and 15 minutes for cooling. An Optical Emission Spectrometer (ICP-OES/Optima 7000, Perkin Elmer) was used to determine the Cd and Pb contents.

For data analysis, the IBM-SPSS 20 statistical package was used with simple descriptive statistics parameters, the population mean and the standard deviation as a dispersion measure. The permissible values for each element, in fresh mass, according to the Food Codex (13) were used as a

comparative reference for leafy vegetables and fleshy fruits and roots. For soils, the permissible limits described (14) were used, and for substrates and OMs, those appearing in the Standards were used (15).

Determination of permissible values for Cd and Pb in organic manures

In order to determine the permissible ranges of Cd and Pb in the OMs used for vegetable production, an adaptation of the methodology for obtaining critical values of microelements in soils, described by Trierweiler and Lindsay in 1969, was used (16). The adaptation of this methodology consisted of separating statistically and graphically the OMs that contaminated the vegetables used as indicator plants for Cd and Pb contamination from the OMs that did not contaminate. A relationship was established between two pairs of quantitative variables using descriptive statistics with the IBM-SPSS 20 statistical package. One variable was the content of Cd and Pb in the organic manures, which was related to the content of these metals in leafy vegetables (lettuce and chard) and subsequently to the content in vegetables with fleshy fruits and roots (bell pepper and radish), always using the permissible values of the Food Codex (13).

RESULTS AND DISCUSSION

PM content in organic manures, soils and substrates

Table 2 shows the Cd and Pb content in organic manures used in each variant and their comparison with the maximum permissible limits corresponding to the standard (15). The values of Cd and Pb in the organic manures, with the exception of the compost obtained from MSW from household garbage, are below the permissible limits, which affirms that these organic manures, from the point of view of their HM contents, can be used in the production of vegetables in organoponics, without causing harm to human health.

In the case of MSW compost from household garbage, the Cd and Pb contents are above the established limits because they contain products that are sources of HM; for this reason, these organic materials should not be used for food production. However, depending on their HM content, they can be used in nurseries, forestry and ornamental plants. Similar results were found in other research (17), in which it is reported that Pb contents in the earthworm humus obtained from MSW are higher than in those obtained from cow dung and cattle manure, a phenomenon that is related to materials present in MSW that are polluting sources of HM. The Cd and Pb contents in the SRFT, SBSF and SBSC soils used to prepare variants are below the maximum permissible limits, so they are not contaminated.

Table 3 shows variants studied, as well as the corresponding permissible limits. The variants where the organic fertilizer used is contaminated (Table 2), the substrate also presents contents above the maximum permissible limits established for substrates (15).

Table 2. Cd and Pb contents in the organic manures.

Variant	Organic manure, soils	HM (X ± s) (mg kg ⁻¹)	
		Cd	Pb
I	Vermicompost	1,42 ± 0,01	88,63 ± 2,23
II	Compost of beef manure	1,89 ± 0,06	98,24 ± 6,08
III	Compost of crop residues	1,94 ± 0,10	100,10 ± 1,03
IV	Compost of MSW, domestic waste (*)	7,80 ± 0,28	465,83 ± 9,01
V	Compost of crop residues	1,96 ± 0,07	137,60 ± 2,10
VI	Compost of beef manure and vegetable residues	1,92 ± 0,01	134,93 ± 5,09
VII	Compost of beef manure	3,02 ± 0,05	150,24 ± 4,30
VIII	Compost of MSW, domestic trash (**)	6,15 ± 0,13	460,80 ± 9,33
IX	Compost of MSW, crop residues (***)	1,91 ± 0,09	139,10 ± 2,96
X	Compost of beef manure	3,04 ± 0,23	151,51 ± 5,03
XI	Compost of MSW, domestic waste (*)	3,63 ± 0,35	151,74 ± 1,06
XII	Compost of MSW, domestic waste (**)	4,60 ± 0,29	437,52 ± 8,01
SRFT		<1,00	24,35 ± 3,02
SBSF		<1,00	36,95 ± 2,95
SBSC		<1,00	46,09 ± 3,05
PML ^a		3	180
PML ^b		3	150

MSW: Municipal solid waste. (*) MSW from CEPRU "Sur Isleta". (**) MSW from CEPRU "Los Cocos". (***) MSW from CEPRU "Vilonio". SFRT: soil typical red Ferrallitic; SBSC: Sialitic Calcic Brown soil; SBSF: soil Sialitic Brown Fluffy (8). PML^a: maximum permissible limit for substrates (15). PML^b: permissible limit for soil (14)

Table 3. Cd and Pb contents in the substrates prepared for each variant.

Variant	Composición del sustrato	HP (X ± s) (mg kg ⁻¹)	
		Cd	Pb
I	50 % SFRT + 50 % organic fertilizer (earthworm humus)	<1,00	66,46 ± 6,85
II	50 % SFRT + 50 % organic fertilizer (cow manure compost)	<1,00	98,06 ± 8,37
III	50 % SFRT + 50 % organic fertilizer (compost from harvest residues)	<1,00	65,36 ± 6,12
IV	50 % SFRT + 50 % organic fertilizer (MSW compost from household garbage*).	9,61 ± 0,63	1 501,50 ± 10,60
V	50 % SBSF + 50 % organic manure (MSW compost from harvest residue)	<1,00	132,16 ± 8,07
VI	50 % SBSF + 50 % organic fertilizer (cattle manure and vegetable waste compost).	<1,00	76,56 ± 7,78
VII	50 % SPSM + 50 % organic fertilizer (cattle manure compost)	2,60 ± 0,10	251,72 ± 28,00
VIII	50 % SBSF + 50 % organic fertilizer (MSW compost from household garbage**)	9,15 ± 0,59	352,00 ± 5,02
IX	50 % SBSC + 50 % organic fertilizer (MSW compost from crop residues***)	1,95 ± 0,93	145,00 ± 9,62
X	50 % SBSC + 50 % organic fertilizer (cattle manure compost***)	5,48 ± 0,95	399,60 ± 10,69
XI	50 % SBSC + 50 % organic fertilizers (MSW compost from household garbage***)	5,86 ± 0,32	305,90 ± 8,61
XII	50 % SBSC + 50 % organic fertilizers (MSW compost from household garbage*)	5,61 ± 0,63	1 301,5 ± 21,07
PML*		8	300

PML*: permissible limit for substrates (15). SFRT: typical red Ferrallitic soil, SBSF: fluffy Sialitic Brown soil and SBSC: calcic Sialitic Brown soil. MSW: Municipal solid waste. (*) MSW from CEPRU "Sur Isleta". (**)MSW from CEPRU "Los Cocos". (***)MSW from CEPRU "Vilonio".

From the above, it is derived that the contamination of the substrates is due to the organic manures, since the soils are below the LPM. HM concentration in substrates was variable due to the heterogeneity of materials. Variability is an important element to consider because the real values, in some points, could be higher than those obtained (18).

The result allows affirming that these contaminated substrates cannot be used in food production because HM can be translocated to vegetables produced with them and directly affect living organisms. HM, especially Cd and Pb, affect various organs and tissues, producing chronic renal insufficiency, increased cholesterol, bone, testicles, placenta, liver, lungs, heart, and the central and peripheral nervous system; they can even cause cancer and finally death (19-21).

Permissible values of Cd and Pb in organic manures

Figures 1 and 2 show the relationship between Cd contents in OMs and its content in leafy and fleshy fruit and root vegetables grown with the application of them. When the Cd concentration in the organic fertilizer is equal to or higher than 3 mg kg⁻¹, its content in both leafy and fleshy fruit or root vegetables grown with the use of this organic fertilizer exceeds the maximum limit established by the Food Codex used as an evaluation criterion (13), which makes them unfit for human consumption; on the contrary, when the Cd content in the organic fertilizer is lower than 2 mg kg⁻¹ it does not constitute a risk of contamination to vegetables.

For this reason, 2-3 mg kg⁻¹ of Cd in organic manures can be considered as the permissible range of the metal in these products, which indicates that only OMs with Cd concentrations below the lower value of the range can be used in vegetable production.

In the Pb case (Figure 3), concentrations equal to or higher than 150 mg kg⁻¹ in compost contaminate leafy vegetables, in accordance with the maximum permissible limits for PM in fresh vegetables. However, when the Pb value in compost is lower than 140 mg kg⁻¹, no contamination of crops occurs. Thus, it can be stated that 140-150 mg kg⁻¹ Pb in OMs is the Pb permissible range in these organic products, which indicates that only OMs with Pb contents in OMs below the lower value of the range can be used in the production of leafy vegetables.

When the Pb content in fruit vegetables is analyzed in relation to the Pb content in the organic fertilizer used for their production (Figure 4), a behavior similar to that found in leaf vegetables is observed, i.e., OMs with values below 140 mg kg⁻¹ can be used to produce vegetables with low risk to human health; whereas, a concentration equal to or higher than 150 mg kg⁻¹ can contaminate vegetables consumed by humans and cause serious damage to their health (22).

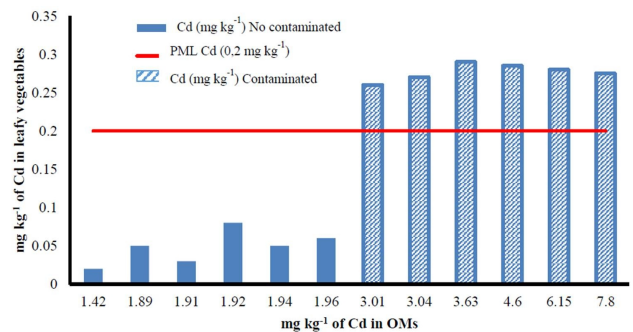
Corresponding to the above, the values 2-3 mg kg⁻¹ for Cd and 140-150 mg kg⁻¹ for Pb are established as maximum permissible limit ranges for these metals for OMs used in food production, especially vegetables. This proposal coincides with what is referred to in the Spanish regulation RD 824/2005 for class B and class C *compost*, with the Ecolabel Decision 2001/688 and the resolution 2nd Draft B.T/2001 for second category *compost* of the European Community and with the Brazilian Normative Instruction for organic manures (15,23). It is necessary to deepen these studies in order to obtain a final proposal of these maximum permissible limits of Cd and Pb.

CONCLUSIONS

- The permissible ranges 2-3 mg kg⁻¹ Cd and 140-150 mg kg⁻¹ Pb in organic manures guarantee the production of food, especially vegetables, free of these contaminants.
- Organic manures with Cd and Pb concentrations above the maximum permissible limits cannot be used to prepare substrates for food production because they constitute contamination risks for plants and animals, as well as for human health.

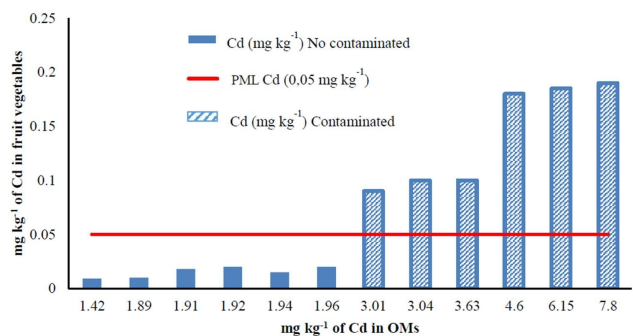
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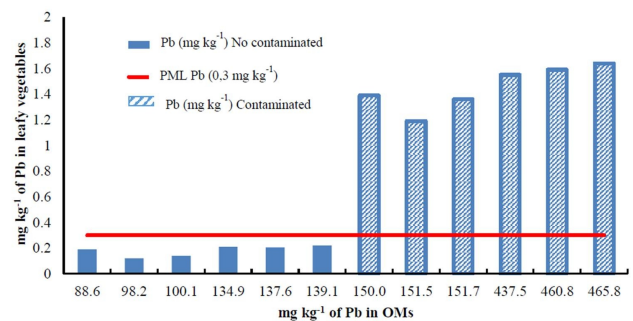
PML Cd: limit value for leafy vegetables (13)

Figure 1. Relationship between Cd contents in leafy vegetables with the content in the Oms.



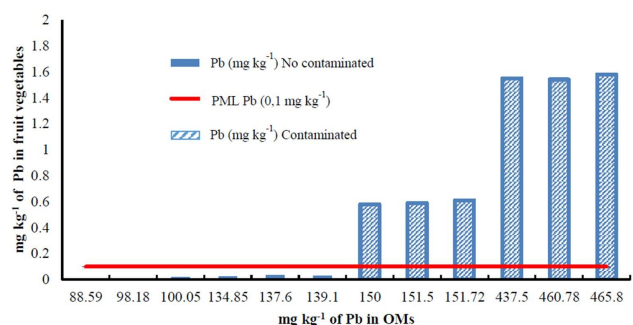
PML Cd: limit value for fleshy fruit and root vegetables (13)

Figure 2. Relationship between Cd contents in fleshy fruit and root vegetables with the content in the OMs.



PML Pb: limit value for leafy vegetables (13)

Figure 3. Relationship between Pb contents in leafy vegetables and their content in the OMs.



PML Pb limit value for fleshy fruit and root vegetables (13)

Figure 4. Relationship between Pb contents in fleshy fruit and root vegetables and their content in the OMs.

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