Food technology

Inorganic contaminants (As, Cd, Pb) in peeled and whole potatoes and sweet potatoes

Contaminantes inorgánicos (As, Cd, Pb) en papas y boniatos pelados y enteros

Contaminantes inorgânicos (As, Cd, Pb) em batatas e batatas-doces descascadas e inteiras

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Abstract: Vegetables are a rich source of macro and micronutrients. Particularly, potatoes and sweet potatoes are widely consumed and are two of the most important food crops in the world. Therefore, contamination of these products due to their content of inorganic contaminants is of great concern.
Considering the healthy trend of consuming these tubers and roots with their skins, which are rich in fiber and other nutrients, analysis of the whole product could provide valuable information in relation to their food safety. Therefore, the presence of arsenic (As), cadmium (Cd) and lead (Pb) in peeled and whole potatoes and sweet potatoes was studied. To do so, analytical methods were optimized and validated according to the Association of Official Analytical Collaboration International (AOAC) requirements for food analysis. Although the content of As, Cd and Pb was in most cases more than three times below the maximum allowed levels for these contaminants, the presence of Cd was detected in the samples of whole sweet potato but not in the peeled ones. The same behavior was observed for Pb in most of the sweet potato samples analyzed. This information points to the need to generate information on the whole root and tuber contaminants content, and to have analytical methods available to gather data on the occurrence of these contaminants in the whole vegetable in order to perform their risk assessment according to the consumption habits.

Keywords: inorganic contaminants, potatoes, sweet potatoes, food safety, trace metals.

Resumen: Los vegetales son una fuente rica en macro y micronutrientes. En particular, la papa y el boniato se consumen ampliamente, siendo de los cultivos más importantes a nivel mundial. Por tanto, la contaminación de estos productos debido a la presencia de contaminantes inorgánicos es de gran preocupación.
Considerando la creciente tendencia de consumo saludable de estos tubérculos y raíces con su cáscara, rica en fibra y otros nutrientes, el análisis del alimento entero podría brindar información valiosa con relación a su seguridad. Debido a esto, se estudió la presencia de As, Cd y Pb en papas y boniatos pelados y

enteros. Para ello, se optimizaron y validaron métodos analíticos según los requerimientos de la *Association of Official Analytical Collaboration International* (AOAC) para análisis de alimentos. A pesar de que el contenido de arsénico (As), cadmio (Cd) y plomo (Pb) fue en la mayoría de los casos menor a un tercio de los valores máximos permitidos para estos contaminantes, la presencia de Cd se detectó en todos los boniatos enteros y no así en los pelados. El mismo comportamiento se observó para la presencia de Pb en la mayoría de las muestras de boniato analizadas. Esto señala la necesidad de generar información sobre el contenido de estos contaminantes en el alimento entero y disponer de metodologías analíticas para ello, de manera de poder realizar una evaluación de riesgo de acuerdo con los hábitos de consumo.

Palabras clave: contaminantes inorgánicos, boniatos, papa, inocuidad, metales traza.

Resumo: Os vegetais são uma rica fonte de macro e micronutrientes. Particularmente a batata e a batata-doce são amplamente consumidas sendo uma das culturas alimentares mais importantes do mundo. Portanto, a contaminação desses produtos pelo conteúdo de contaminantes inorgânicos é preocupante.

Considerando a tendência de consumo saudável desses tubérculos e raízes com suas cascas, ricas em fibras e outros nutrientes, a análise do conjunto poderia fornecer informações valiosas em relação à sua segurança. Portanto, a presença de arsênico (As), cádmio (Cd) e chumbo (Pb) em batatas descascadas e inteiras e batata-doce foi estudada. Assim, os métodos analíticos foram otimizados e validados de acordo com os requisitos da *Association of Official Analytical Collaboration International* (AOAC) para análise de alimentos. Embora o teor de As, Cd e Pb estivesse na maioria dos casos mais de três vezes abaixo dos níveis máximos permitidos estabelecidos para esses contaminantes, a presença de Cd foi detectada em todas amostras de batata-doce inteira, mas não nas descascadas. O mesmo comportamento ocorre para a presença de Pb na maioria das amostras de batata-doce analisadas. Esta informação aponta para a necessidade de gerar informação de todo o teor de contaminantes radiculares, dispor de métodos analíticos para recolher dados sobre a ocorrência destes contaminantes em toda a hortaliça realizando a sua avaliação de risco de acordo com os hábitos de consumo.

Palavras-chave: contaminantes inorgânicos, batatas, batatas doces, segurança alimentar, metais traço.

Author notes

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1. Introduction

Potato (*Solanum tuberosum, L.*) offers the possibility of fighting poverty and malnutrition while securing food supply in developing countries. Its total demand in South America is projected to increase to 19.6 million tons in 2030 and higher levels of average per capita consumption are anticipated[1][2].

Sweet potato (*Ipomoea batatas, L.*) is produced mainly in developing countries[3]. Its consumption decreases vitamin A deficiency and combats undernourishment, serving as an energy-dense food often in post disaster contexts[4][5].

Several sources of inorganic contamination in these vegetables are related to the environment[6]. The presence of metals such as Cd and Pb in crops can indicate environmental contamination in cultivation areas. Industrial processes release these metals into the atmosphere and watercourses, reaching the trophic chain[7][8]. Other sources are fertilizers and manures used in crops[9][10]. As is an element normally occurring in its mineral bound form in the earth's crust and it's liberated into the environment by anthropogenic activity, affecting human health[11]. Due to their toxicity[12], contamination with these elements is of great concern, and maximum allowed levels (MAL) have been established by the Codex Alimentarius and in the region by the MERCOSUR (South Common Market) for peeled vegetables[13][14]. The MAL for Cd and Pb is 0.1 mg kg^{-1}, while for As it is 0.20 mg kg^{1}. The latter is only established in MERCOSUR regulation.

Nowadays, there is a tendency to consume these vegetables with their skin. Consumption of cooked potatoes with skin improves lipid metabolism and can significantly enhance cardio protective fiber intake[15]. Even the whole cooked potato is used by the food industry to obtain potato flour[16]. The unpeeled sweet potato reduces the loss of physical and chemical properties during its processing and enhances the stability with a better nutritional value[17]. Both tuber and root are considered healthy foods and especially when they are consumed with their fiber-rich skin[18][19][20][21].

It has been reported that the metal content in the skin is higher than in the pulp[22]. As the current tendency is to consume the whole vegetable, it is relevant, then, to collect data about the content of these contaminants in the whole tuber/root to evaluate their safety. Reliable analytical methods are thus needed. Some of these analytes, e.g., As, represent a challenge, since the determination of total As in complex food matrices has suffered from difficulties regarding accuracy and precision[13].

The aim of this research was to provide suitable analytical methods for quantifying inorganic contaminants (As, Cd, and Pb) in potatoes and sweet potatoes, and to start a food safety surveillance program in both peeled and unpeeled potato/sweet potato. To our knowledge this is the first study on the matter in the southern cone of America and represents a contribution to draw attention to the necessity of collecting data on these contaminants content in the whole edible tuber and root.

2. Materials and methods

2.1 Reagents

All chemicals were of analytical reagent grade unless otherwise specified. Deionized water (ASTM type I) was obtained from a Millipore (Milestone, Sorisole, Italy) Direct-Q 5 water purifier.

Standard atomic stock solutions (Merck, Darmstadt, Germany) of 1000 mg L^{-1} of Cd and As were employed.

Pb stock solution was prepared from $Pb(NO_3)_2$ 99.99 % (Sigma Aldrich, USA).

Working standard solutions were prepared daily by dilution of the stock solution with 4:21 HNO_3: H_2O.

Palladium modifier solution (500 mg L^{-1}) was prepared by dilution with 0.1% v/v HNO_3 of palladium matrix modifier for graphite furnace, 10 g L^{-1} (Merck, Darmstadt, Germany).

Ammonium dihydrogen phosphate modifier solution (5 g L^{-1}) was prepared from $(NH_4)H_2PO_4$ matrix modifier (Merck, Darmstadt, Germany) for graphite furnace, 99.99 % in deionized water. Nickel nitrate modifier solution 10 g L^{-1} was prepared from $Ni(NO_3)_2$ (Fisher Scientific, Hampton, USA) in deionized water.

Sodium tetrahydroborate solution (3.0 % (w/v) in sodium hydroxide 0.05 mol L^{-1}) was prepared from sodium tetrahydroborate (Hydride-Generation grade Fluka, NY, USA).

Certified reference material (CRM) 1568a Rice flour was obtained from NIST.

High purity argon (Ar) (Praxair, Montevideo, Uruguay) was employed.

2.2 Instrumentation

Dried and grinded samples were digested in 100 mL high-pressure closed vessels EasyPrep Plus, using a microwave oven (Mars 6, CEM Corporation, Matthews, NC, USA).

Cd and Pb determinations both in potato and sweet potato samples, and As determination in sweet potato samples were performed in a longitudinal heated, electro-thermal atomic absorption spectrometer (Perkin Elmer HGA 900, Shelton, USA) fitted with deuterium background corrector. Atomization took place on solid pyrolytic graphite L'vov platform inserted in standard pyrocoated graphite tubes. Cd electrodeless discharge lamp (210 mA, 228.8 nm, Perkin Elmer precisely, Shelton, CT USA), As electrodeless discharge lamp (400 mA, 193.7 nm, Perkin Elmer precisely, Shelton, CT USA) or Pb hollow cathode lamp (10 mA, 283.3 nm, Perkin Elmer Lumina, Singapore) were used as appropriate.

For As determination in potatoes a nitrogen microwave induced plasma atomic emission spectrometer 4210 (MIP OES, Agilent Technologies, Santa Clara, USA) equipped with a standard torch and a multimode spray chamber (MSIS, Agilent) was used. Nitrogen (99.5 %) was supplied by a nitrogen generator model 4107 (Agilent Technologies, Santa Clara, USA) coupled with an air compressor model KK70 TA-200 K (DürrTechnik, Bietigheim-Bissingen, Germany).

2.3 Sampling and sample preparation

Sampling was carried out at Metropolitan Agrifood Market[23] (UAM, formerly Mercado Modelo of Montevideo). The UAM is the main wholesale logistic/distribution center for fruit and vegetables trade and distribution in Uruguay, supplying more than 60% of the vegetables consumed in the country[24]. The fresh vegetables enter to the market directly from the producers and from there they are distributed to greengroceries, warehouses, self-services, supermarkets, hospitals, etc. Sampling was performed with randomly selected traders in coordination with UAM management before the opening of daily operations (between 4:00-6:00 AM). In this way, the completely random sampling was representative of the distribution of the products to the country. This allowed the monitoring of contaminants in vegetables that will be offered in a wide range of retail sales and human consumption points. Approximately 1 kg (5 pieces) of each vegetable were placed in plastic bags, sealed and labeled. Samples were transported to the processing lab within 1 hour of collection. Samples were collected between 2018 and 2020 at two times of the year, in February and between July and August. The sweet potatoes were from Salto and Canelones departments, while the potatoes were from San José and Canelones.

Potatoes and sweet potatoes were chopped with a ceramic knife and dried in an air-forced oven, Model DN93 (Yamato, Tokyo, Japan) set at 70 °C. After 96 hours, the weight was recorded for water content

calculations and samples were grinded in a Model 4 Wiley mill (Thomas Scientific, NJ, USA), with a 1 mm sieve, and later placed in 50 mL Falcon tubes and kept in dried conditions until analysis. Three pooled samples of whole potato and sweet potato, 5 pooled samples of peeled potato and 11 pooled samples of peeled sweet potato, 2 of them of orange pulp, were obtained to be analyzed. All the samples of potato collected were of white/cream pulp and red/purple skin.

The number of pooled samples obtained was determined by the availability of each product on the sampling date. The traceability of the samples was assured in order to analyze the individual samples if a result above the MAL was obtained.

Prior to analytical measurements, 0.5 g of dried and grinded samples were treated with 8 mL of HNO_3 1:1, let stand for 10 min and digested with microwave assistance. The heating program consisted of a 20 min ramp up to 120 °C, holding at that temperature for 20 min, followed by a 20 min ramp from 120 °C to 170 °C and remaining 15 min at that temperature. The remaining solution was diluted with ultrapure water to 25 g. For each element, the determinations (including reagent blanks) were carried out by triplicate.

2.4 Analytical determinations

The determination of Cd and Pb in potatoes and sweet potatoes and of As in sweet potatoes was performed by electrothermal atomic absorption spectrometry (ETAAS).

Working conditions are described in Table 1.

TABLE 1
Temperature programs for As Cd and Pb determinations by ETAAS

Stage	Temperature (°C)	Hold time (s)	Ramp rate (°C s-1)	Internal Ar flow (mL min-1)
Drying (1)	110 (As, Cd, Pb)	30 (As, Cd, Pb)	1 (As, Pb) / 30 (Cd)	250 (As, Cd, Pb)
Drying (2)	130 (As, Cd, Pb)	20 (As, Cd, Pb)	15 (As, Cd, Pb)	250 (As, Cd, Pb)
Pyrolysis	1200(As) /500(Cd) / 800(Pb)	30(As)/40(Cd, Pb)	10 (As, Cd, Pb)	250 (As, Cd, Pb)
Atomization	2500(As)/1400(Cd)/1300(Pb)	5 (As, Pb /4(Cd)	0 (As, Cd, Pb)	0 (As, Cd, Pb)
Cleaning	2600(Cd) /2500(As, Pb)	5 (As, Cd, Pb)	1 (As, Cd, Pb)	250 (As, Cd, Pb)

µL digest / µg matrix modifier: As, 20/ 100 Ni(NO3)2; Cd, 10/ 5 Pd; Pb, 20/ 50 NH4H2PO4 (a cool down step until 200 °C holding for 30 s was added).

The As determination in potatoes was performed by hydride generation nitrogen microwave induced plasma atomic emission spectrometry (HG – MIP OES). Prior to the determination, a pre-reduction step with 20 % (w/v) KI and 37 % HCl in the ratio, digested sample, or standard (10): HCl (1): KI (1) at room temperature for 1 hour had to be carried out. Both the reduced digested sample and sodium tetraborate entered to the multimode spray chamber at 0.90 mL min^{-1} (pump speed 30 rpm). After 20 s of stabilization of the system, the hydridegenerated was carried towards the atomizer by a 0.7 L min^{-1}N. flow. The determination was performed at 193.695 nm with automatic background correction. The viewing position was 10 (respect to the centre of the torch) and the reading time was 10 s (read by duplicate).

Analytical methods were validated according to the Commission Decision 2002/657/EC criteria and their figures of merit were compared to the AOAC requirements[25][26].

2.5 Validation

The following figures of merit were evaluated: linear range, precision under repeatability conditions, trueness, and detection (LOD) and quantification limits (LOQ).

Linearity was evaluated by means of a 6-point calibration curve. Quality of the linear fit was verified by both visual inspection and assessment of the value of the coefficient of determination (R^2)[26]. Precision (repeatability) was evaluated, for As and Cd determination, by analyzing a certified reference material (CRM NIST 1568a Rice flour; n=6). For Pb it was assessed by measuring a sample previously spiked with Pb, at the MAL level of 0.1 mg kg^{-1} (n=6).

Detection and quantification limits (3.3 s and 10 s criteria, respectively) were estimated by measuring 10 blanks[27]. Trueness was evaluated by analyzing a certified reference material (CRM) NIST 1568a Rice flour for As and Cd, and as this CRM did not inform a certified value for Pb concentration, trueness in this case was evaluated with 6 samples previously spiked at the 0.1 mg kg^{-1} level.

3. Results and discussion

The figures of merit for the validated analytical methods for the determination of As, Cd and Pb are presented/summarized in Table 2. Determination coefficient (R^2) for all calibration curves was > 0.99 (data not shown).

Samples of peeled and whole potatoes and sweet potatoes were analyzed by the developed methods within the framework of a health surveillance program. The obtained results are presented in Tables 3 and 4.

TABLE 2
Figures of merit of the methods developed to determine As, Cd and Pb levels

	Potato				Sweet potato			
	LOD	LOQ	Trueness** (R%)	RSD %	LOD	LOQ	Trueness** (R%)	RSD %
As*	0.0033	0.011	115 ± 9	7.6	0.0043	0.011	69 ± 9	7.1
Cd	0.00043	0.0013	90 ± 11	12	0.00043	0.0013	90 ± 11	12
Pb	0.0060	0.018	82 ± 2	2.8	0.0060	0.018	93± 2	2.3

All determinations were performed using ETAAS except for As* in potatoes, which was by HG-MIP OES. LOD and LOQ are expressed in mg kg-1 of fresh weight. ** Mean recovery ± standard deviation (%) (n = 6).

TABLE 3
As Cd and Pb content for whole potatoes and sweet potatoes

Sample	Element	Whole potato	Whole sweet potato
Pool 1	As	ND	ND
	Cd	0.0090 ± 0.0004	0.0027 ± 0.0002
	Pb	ND	
Pool 2	As	ND	ND
	Cd	0.0022 ± 0.0002	0.0022 ± 0.0002
	Pb		
Pool 3	As	ND	ND
	Cd	0.034 ± 0.001	0.0023 ± 0.0001
	Pb	ND	

Results are expressed in mg kg-1 of fresh weight (mean ± standard deviation; n=3); ND: not detected;

TABLE 4
Content of As Cd and Pb in peeled potatoes and sweet potatoes

	Sweet potatoes pooled samples											Potatoes pooled samples				
Element	1	2	3	4	5	6	7	8	9	10	11	1	2	3	4	5
As	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND		
Cd	ND	ND		ND		ND	ND	ND	ND		ND	0.0097 ± 0.0005	0.0022 ± 0.0001	0.010 ± 0.001	0.016 ± 0.001	0.009 ± 0.002
Pb	ND	ND			0.067 ± 0.001		ND	ND	ND	ND	ND	0.0333 ± 0.0005	0.041 ± 0.003	0.029 ± 0.005	ND	ND

Results are expressed in mg kg-1 of fresh weight (mean ± standard deviation; n=3); ND: not detected;

3.1 Analytical determinations

The AOAC requirements for an analytical method to be consider a reference method for heavy metals monitoring purposes establishes that LOQ should be ≤ 0.010 mg kg^{-1}[25]. This requirement was not fulfilled in the case of As (0.011 mg kg^{-1}) and Pb (0.018 mg kg^{-1}). Nonetheless, it was considered that the limits of quantification achieved widely satisfy the needs for their application in potatoes and sweet potatoes. The LOQ´s are more than 5 times below the MAL for Pb and almost 20 times below the MAL for As, and this was considered appropriate. Besides this, the obtained LOQ values are also better than those reported in several published studies[28][29][30].

In the case of As determination, when it was determined by ETAAS, matrix incidence in the response was different for potatoes and sweet potatoes. Interference abatement was unsuccessful in potato samples, thus for As determination in that matrix, HG-MIP OES was used instead, reaching suitable limits of detection and quantification.

With regard to the other figures of merit, all of them fulfilled the performance requirements established by the AOAC, as it can be observed in Table 2.

Finally, the simple instrumentation required by these methods is an important advantage that makes the wide application of them possible. Moreover, the use of MIP OES does not need expensive gases such us argon or acetylene as other atomic spectrometric techniques, making it a suitable option for monitoring purposes.

Following the fully described methods, any laboratory with the adequate instrumentation could implement a reliable analytical platform for food safety monitoring taking advantage of the suggested aspects, that are alternatives to standard methods, which make them simpler, more economical and in accordance with the principles of green chemistry. These characteristics make them a powerful tool for monitoring the presence of these contaminants in developing countries where these products are increasingly important.

3.2 Inorganic contaminants concentration

The inorganic contaminants content was in most of the cases more than three times below the MALs.

As was not quantified in any sample; Pb was found both in peeled and whole samples, and, as it was mentioned before, it could be related to environmental contamination, and Cd was present in all potato samples. Cd accumulation in potato has been reported due to the use of phosphate fertilizers[31]. This kind of fertilizers is widely used worldwide and their misuse may be one of the main causes of the presence of Cd.

However, in sweet potatoes, Cd and Pb are present in the whole samples analyzed but not in the peeled ones, and the same behavior appears in most of the analyzed samples, indicating that the determination in the whole product could be a better indicator of their safety. As it was mentioned before, the revalorization of the skin of these vegetables contributes to the tendency of consuming them unpeeled. Due to this fact,

we consider that the analysis of the whole food gives valuable information that needs to be considered to perform a risk assessment which contemplates the trend of consumption of these foods unpeeled.

Regarding the sampling plan, despite our study involved few samples, each pooled sample represents a random sampling of several producers in the country. We propose that pooling samples from wholesales market site, when traceability is assured (site of production, irrigation conditions, soil type), could be used as an alternative way to sampling in farms or retail shops. In this way, if any contaminant is found above the MAL in the pool, the individual samples could be analyzed, and using survey information more tests could be performed at production site (soil and irrigation water).

4. Conclusions

Potatoes and sweet potatoes are foods widely consumed and so their safety must be guaranteed in terms of their content of trace metals such as As, Cd and Pb. The concentration of these contaminants found in the tested samples was in accordance with the Codex Alimentarius and the MERCOSUR recommendations. Despite this, in some cases the presence of these contaminants was found in the whole samples, but they were not detected in the peeled ones.

Given that there is an increasing trend to consume vegetables with their peel, gathering data of the contaminants contents in the whole sample is needed to perform a risk assessment when they are consumed in this condition. For this purpose, analytical methods were developed that are accurate to perform the determinations of these elements in both peeled and whole samples. Cd was present in almost all samples of potato, although the content was below the MAL; this could be a warning sign about the need for future actions for surveillance of food safety.

For the first time, a study considering both peeled and whole samples of potato and sweet potato from the southern region of South America was carried out. This first prospective study about the presence of these contaminants in peeled and whole products shows the need of gathering more information regarding their content in the whole tuber, for future discussions about the definition of maximum allowed limits according to the type of food and the habits of consumption, as well as the levels in different crop regions and harvest season.

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