مجلة جامعة تشرين للبحوث والدراسات العلمية _ سلسلة العلوم البيولوجية المجلد (32) العدد (5) 2010
Tishreen University Journal for Research and Scientific Studies - Biological Sciences Series Vol. (32) No. (5) 2010

Contents of Heavy Metals (Cd, Pb, Ni) in Imported Spices

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(Received 24 / 6 / 2010. Accepted 24 / 11 /2010)

□ ABSTRACT □

The concentrations of some heavy metals such as cadmium (Cd), lead (Pb) and nickel (Ni) in imported spices collected as composite samples from the custom areas of Lattakia seaport hhave been estimated using atomic absorption. The study Has shown differences in heavy metal concentrations according to the origin and kind of spices. The highest concentrations of Ni (3.21, 3.11) in mg/kg dw were recorded in cloves and cardamom, respectively, while highest concentrations of Pb were 0.85, 0.43, 0.42 and 0.35 mg/kg in coriander, nutmeg, pimento and cinnamon, respectively. Cd concentrations were generally below the permissible levels approved by WHO and FAO. The highest concentrations were 0.17 and 0.14 in cloves and black pepper, respectively. The obtained results have shown significant correlations between averages and maximum content of each heavy metal. Correlation coefficient values were 0.844, 0.786 and 0.564 for Cd, Pb and Ni, respectively. Provisional Tolerable Weekly Intake (PTWI) has been calculated for each heavy metal.

Keywords: Heavy metals, spices, atomic absorption spectroscopy, PTWI

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محتويات المعادن الثقيلة (كادميوم، رصاص، نيكل) في التوابل المستوردة

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(تاريخ الإيداع 24 / 6 / 2010. قبل للنشر في 24 / 11 / 2010)

☐ ملخّص ☐

جرى تقدير تراكيز بعض المعادن الثقيلة الموجودة في توابل مستوردة (جمعت بوصفها عينات مركبة من ضمن المنطقة التابعة لجمارك مرفأ اللاذقية) باستخدام جهاز الامتصاص الذرّي. بيّنت الدراسة تبايناً في تراكيز المعادن الثقيلة تبعاً لمنشأ التوابل ونوعها. سُجّلت التراكيز الأعلى للنيكل (3.21، 3.11 ملغ/كغ مادة جافة) في القرنفل وحبّ الهيل على التتالي، أمّا التراكيز الأعلى للرصاص فكانت 0.85، 0.43، 0.42، 0.35 في الكزبرة، جوزة الطيب، البهار والقرفة على التتالي. كانت تراكيز الكادميوم دون الحدود المسموح بها من منظمة الصحة العالمية ومنظمة الأغذية والزراعة، وقد كانت التراكيز الأعلى للكادميوم 0.17 و 0.14 في كلٍّ من القرنفل والفلفل الأسود على التتالي. أوضحت النتائج وجود علاقة ارتباطية بين المتوسطات والحدّ الأعلى للتركيز. قيم معامل الارتباط كانت 0.844، 0.786، 0.564 لكلٍّ من الكادميوم والرصاص والنيكل على التتالي. وقد جرى حساب المعدل الافتراضي للمتناول أسبوعياً PTWI لكلٍّ من المعادن الثقيلة المدروسة.

الكلمات المفتاحية: معادن ثقيلة، توابل، مطيافية الامتصاص الذرّي، PTWI (المعدل الافتراضي للمتناول أسبوعياً).

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Introduction:

In line with industrial development, pollution in the environment and consequently in agricultural raw materials is becoming a major safety concern all over the world. A frequently confronted environmental pollution, threatening food safety is due to heavy metals. As a result of soil, atmosphere, underground and surface water pollution, food and beverages are getting contaminated by heavy metals. The effect of environmental pollution on contamination of food and on their safety for human consumption is a serious global public and widely addressed issue [1,2,3].

Several researches have focused on residues of numerous heavy metals in food stuffs as natural or inherent components and also possibly present as a result of contamination or deliberate addition [4]. Accordingly the levels of heavy metals in food stuffs have been reported around the world. In many developing countries such data are not readily available[5].

Exposure of consumers to heavy metals and related health risks are usually expressed as percentage intake of provisional tolerable weekly intake (PWTI), a reference value established by WHO [5,6,7]. Accordingly, the weekly intake of metals from all sources should not exceed 0.05 and 0.075 mg.kg-1 body weight for lead and cadmium, respectively. Cadmium and lead are among the most abundant heavy metals, and are particularly toxic. The excessive content of these metals in food is associated with etiology of a number of diseases [6,7].

Cadmium compounds are used commonly as color pigment, in re-chargeable nickel-cadmium batteries, coatings, electroplating steel and cast iron, plastic stabilizers constituent of low melting or easily fusible alloys, electronic and optics and solder for aluminum, reactor control rods, hardener for copper and catalysts. Cadmium (in form of powder) is also used in dentistry, as an amalgam (1 Cd: 4 Hg). Moreover, it is present as a contaminant in phosphatic fertilizer and sewage sludge and is dispersed by mining activities [8,9]. Cadmium exposure may cause kidney damage and/or skeletal damage [6].

Lead is present in the environment because of air, soil and water pollution. Major sources of lead are exhaust fumes from vehicles, industrial gases and liquid effluents, some phosphate fertilizers and pesticides. Airborne lead can be deposited on soil, water and plants thus reaching humans via the food chain. Lead is accumulated in the skeleton, especially in bone marrow and causes renal tubular damage and may also give rise to kidney damage (WHO, 1995). International Agency for Research on Cancer (IARC) classified cadmium and lead as human carcinogen.

Nickel is a very abundant element, and it is combined with other metals to form alloys. These alloys are used in the making of metal coins and jewelry and in industry for making metal items. Nickel compounds are also used for nickel plating, to color ceramics, to make some batteries, and as catalysts that increase the rate of chemical reactions. Major sources of exposure are: tobacco smoke, auto exhaust, fertilizers, superphosphate, food processing, hydrogenated-fat-oils, industrial waste, stainless steel cookware, testing of nuclear devices, baking powder, combustion of fuel oil, dental work and bridges. The most common adverse health effect of nickel in humans is an allergic reaction [1].

Spices and condiments are added to food in small amounts but they make important contribution to the sensory qualities due to presence of volatile and fixed oils [10]. Spices and condiments are dried parts of plants. They consist of rhizomes, barks, leaves, fruits, seeds, and other parts of the plant. Most of these are fragrant, aromatic and pungent. The bulk of the dry material of spices and condiments contains carbohydrates, and organic compounds having diverse functional groups. Addition of spices and condiments –that may be contaminated with heavy metals- to food as a habit (or regular ingredient) may contribute to the accumulation of these metals in human organs and lead to different health troubles [11,12]. Therefore strict periodic surveillance of these heavy metals (contaminants) is advisable [13], whereas such research is not available in Syria until now.

Objectives and Research Importance:

Syria imports spices and condiments among a lot of foodstuff from several countries. These spices and condiments may be subject to contamination by one way or another as described above, especially these days, where the release of toxic wastes in the environment has increased.

The objective of this work is to estimate the levels of some heavy metals i.e. lead, cadmium, and nickel in imported spices and condiments which are shipped through the main sea entryway port, Lattakia. Moreover, the investigation of the levels of heavy metals is recommended by the International Organizations (FAO and WHO).

Materials And Methods:

Sample Collection

Spices' samples were collected as composite samples from the custom areas of Lattakia sea port. Representative sampling was done at random from different containers and boxes within these custom areas. A total of eight kinds of spices including coriander, nutmeg, cardamom, black pepper, Pimento (Jamaica pepper), cloves, cinnamon, and mustard, were collected in pairs (2 x 1 kg, for each kind) from each of these custom areas. Collected samples recognized and classified according to their English name, Arabic name, scientific name, and the used part of the plant (Table 1).

Table 1 : Scientific and common names of studied spices

Common name	Arabic name	Scientific name	Family	Used part
Coriander	كزبرة	*Coriandium sativum*	Umbellifeae or Apiaceae	Seeds
Nutmeg	جوزة الطيب	*Myristica fragrance*	Myristicaceae	Nuts
Cardamom	هيل	*Elettaria cardamonum*	Zingiberaceae	Seeds
Black pepper	فلفل أسود	*Capsicum nigrum*	Piperaceae	Seeds
Pimento (Jamaica pepper)	بهار حلو	*Pimenta dioica*	Myrtaceae	unripe Fruits
Cloves	كبش قرنفل	*Syzygium aromaticum*	Myrtaceae	Flower buds
Cinnamon	قرفة	*Cinnamonum zylanicum*	Lauraceae	Bark
Mustard	خردل	*Brassica nigra*	Brassicaceae	Seeds

Sampling was done periodically throughout months between October of 2007 and June of 2009. The sampling period covered the total amount of shipped spices and condiments (Table 2). Sample origin is not specified.

Table 2: Quantities of Imported Spices and Shipped between October 2007 and June 2009 to Latakia Sea Port

Imported Spices	Coriander	Nutmeg	Cardamom	Black pepper	Pimento	Cloves	Cinnamon	Mustard
Quantity in kg	2440	20869	3611970	36000	780200	315481	1414128	3234
Number of sample	3	5	5	3	4	4	5	3
Number of replicate	3	3	3	3	3	3	3	3

Reagents and solutions

The standard references for the given elements were procured as it was described in the guidebook of Atomic Absorption Spectrophotometer apparatus. The working solutions were prepared by diluting the stock solutions to appropriate volumes. The nitric acid 65% and hydrochloric acid 35% used were of ultra pure grade, purchased from Merck. All reagents were of analytical-reagent grade and all solutions were prepared using double distilled water.

Sample Preparation and Digestion

Sub-samples (50 g, each) were taken at random from the composite sample (1 kg) and were processed for analysis by the dry-ashing method. Samples were first dried in oven at 105 °C to a constant weight. Each oven-dried sample was ground in a mortar until it could pass through a 60 mesh sieve. The ground samples (5g each) were placed in crucibles and few drops of concentrated nitric acid were added to the sample as an ashing aid. Dry-ashing process was carried out in a muffle furnace by stepwise increase of the temperature up to 550 °C and then left to ash at this temperature for 4 hrs [14,15,16]. The ash was left to cool and then rinsed with 1M hydrochloric acid. The ash suspension was filtered with 0.45 μm pore size cellulose nitrate membrane filter paper and the filtrate made up to the volume of 50 ml with 1 M hydrochloric acid. The samples were stored in clean, dry, high density polyethylene bottles of 100 ml capacity with screw caps. Bottles were prewashed with nitric acid, rinsed with double distilled water, dried and tested for contamination by leaching with 5% nitric acid. The bottles contained no metal liners that could contaminate the samples. Bottles were stored until flame atomic absorption spectrophotometry was performed. All measurements were carried out in duplicates.

Sample Analysis of Heavy Metal Content

A Buck Scientific, Inc. type Atomic Absorption Spectrophotometer (AAS) model 210 VGP with Air-C_2H_2 flame type was used. Flame conditions for these elements are given in Table3.

Table 3: Flame Atomic Absorption Concentration Ranges

Metal	Direct Aspiration Detection limit (mg/L)	Sensitivity (mg/L)	Linear Working Range (mg/L)	Flame Type	Wavelength	Band pass	Detection Limit (mg/L)
Cadmium	0.005	0.025	0.2 to 2.0	A-A	228.8	0.7	0.01
Lead	0.1	0.5	5.0 to 25	A-A	217.0 383.3	0.7 0.7	0.09 0.10
Nickel	0.04	0.15	1.0 to 7.5	A-A	232	0.2	0.02
A-A means Air-Acetylene (Air-C_2H_2)							

The standard references for the given elements were procured the AAS-apparatus producing company. Calibration curves for various elements obtained from these standards were linear in the working range.

Quality Assurance and Quality control

Appropriate quality assurance procedures and precautions were carried out to ensure reliability of the results. Standard reference materials (SRM) obtained from the AAS-apparatus producing company were used for validation of the analytical procedure. The results of measurements are summarized in Table 3. Blank and drift standards were run after twenty determinations to maintain instrument calibration. For validation of the analytical procedure, standard procedure was carried out by spiking and homogenizing several already analyzed samples with varied amounts of standard solutions of the metals. Result is summarized in table 4.

Table 4: Recovery (%) of Cd, Pb and Ni in analyzed spices

Heavy metal	Spiked concentration mg/kg	Recovery concentration mg/kg	Recovery %	
Cd	0.025	0.022	89	92
	0.05	0.046	92	
	0.1	0.096	96	
Pb	0.025	0.021	85	85
	0.05	0.042	83	
	0.1	0.088	88	
Ni	0.025	0.023	91	94
	0.05	0.047	94	
	0.1	0.097	97	
Data are mean of three sample of three replicate				

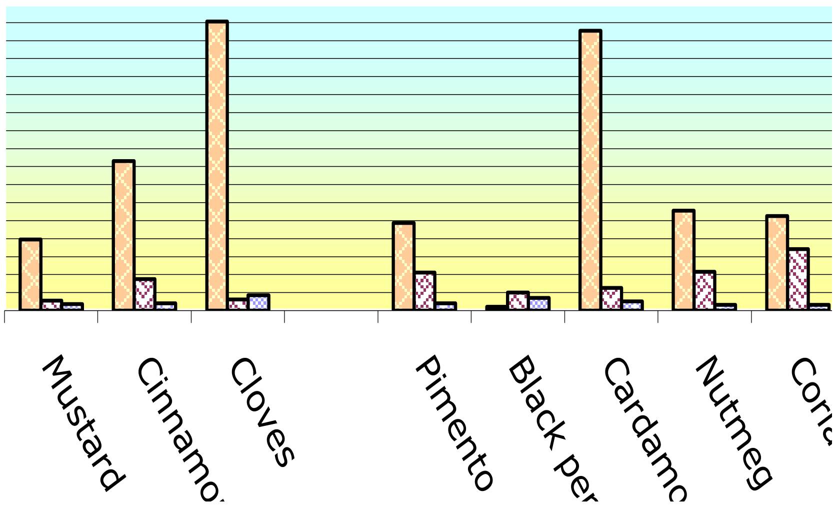
The coefficient of variation of replicate analyses was determined for the measurements to calculate analytical precision.

Results And Discussion:

Averages of 3 heavy metal (Cd, Pb, Ni) concentrations and ranges found in the studied spices are summarized in Table 5, and presented graphically in Figure 1.

Table 5: Content of Heavy Metals in Shipped & Controlled Spices (mg/kg dry wt).

	Cd			Pb			Ni		
Spices	**Max.**	**Average**	**Min.**	**Max.**	**Average**	**Min.**	**Max.**	**Average**	**Min.**
Coriander	0.08	0.06	0.08	1.78	0.68	0.59	5.24	1.05	nd
Nutmeg	0.11	0.06	0.05	0.54	0.43	0.42	1.32	1.11	0.10
Cardamom	0.12	0.1	0.07	0.33	0.25	0.23	3.49	3.11	1.39
Black pepper	0.18	0.14	0.03	0.29	0.20	nd	0.74	0.04	nd
Pimento (Jamaica pepper)	0.14	0.08	0.07	0.67	0.42	0.27	2.21	0.97	0.71
Cloves	0.61	0.17	0.16	0.61	0.12	nd	3.85	3.21	3.06
Cinnamon	0.10	0.08	0.05	0.37	0.35	nd	3.24	1.66	1.23
Mustard	0.10	0.07	0.04	0.30	0.11	nd	1.39	0.79	0.71
nd = Not detected or below detection limit									



The ranges of concentration averages were 0.06-0.17 for cadmium, 0.12-0.68 for lead, and 0.04-3.2 for nickel. It means that the spices are contaminated from origin source. The profile of the heavy metals content of spices was found to be in the order Ni > Pb > Cd as figure 1 shows. Coriander had the highest content of Ni (5.24 mg/kg dw), and the highst content of Pb (1.78 mg/kg dw) as well. This indicates that the main quantity of imported Coriander was delivered from polluted areas.

The contamination levels of Ni and Pb shown on (Table 5) were generally higher than that of Cd. The elevated concentrations of Nickel in plant tissue reflect man-made pollution [3]. The Ni as well as Pb contents in the imported spices indicate that the spices had been cultivated (production media) in industrial and /or urban areas.

Table 6:Correlations between data of analysed samples

Correlation between Averages			
	Cd	Pb	Ni
Cd	1	-0.533	0.359
Pb	-0.533	1	-0.192
Ni	0.359	-0.1927	1
Correlation between Max.			
	Cd	Pb	Ni
Cd	1	-0.103	0.196
Pb	-0.103	1	0.702
Ni	0.196	0.702	1
Correlation between Max. and Average as well as Min. and Average			
	Cd	Pb	Ni
Max. / Average	0.844	0.786	0.564
Min. / Average	0.719	0.862	0.847

On the other hand, table 6 demonstrates correlation between results data in table 5. The correlation between average and maximum content (for each heavy metal) was for the

3 heavy metals significant within inspected (studied) spices. Correlation coefficient values were 0.844, 0.786 and 0.564 for Cd, Pb and Ni respectively. Correlation between average and minimum was also significant with coefficient values of 0.7194, 0.8622 and 0.8465 for Cd, Pb and Ni respectively. Correlation coefficient values were higher between average and minimum for Pb and Ni, and between average and maximum for Cd. Additionally, correlation coefficient values between averages were not so high, and the highest value was -0.538 between Pb and Cd.

Abou-Arab & Abou Donia [17] and Murphy et al. [18] affirmed that the heavy metals contents in spices varied depending on the country of origin, environmental pollution levels, plant part and technological processes.

It is generally believed that a combination of more than one factor including the use of polluted water, bad practice in post harvesting and handling of the spices (as products) with disregard to the food safety guidelines, and the physical market environments in locations surrounded by a heavy urban pollution deposition may have exacerbated contamination levels of these spices. Products with such specifications have no chance to enter the market in developed countries. Only markets of developing countries seem to be the final destination for consumption, whereby food legislations as well as control are in weak situation [19,20,21].

Because of the absence in Syria of such directive which sets maximum levels of lead, nickel, and cadmium in spices, one could take the directive (S.N.S. 575/2009) which sets the maximum level for heavy metals content in seeds and legumes (similar food group). The maximum level of lead and cadmium is 0.2 and 0.1 mg/kg respectively [22], but there is no setting for nickel. Directive 2001/22/ of the European Community also sets the maximum levels of lead and cadmium for cereals, legumes and pulses 0.2 and 0.1mg/kg wet weight respectively[23]. Such data for nickel do not seem to be present.

Exposure of consumers to health risks is usually expressed as provisional tolerable daily intake (PTDI), a reference value established by the FAO/WHO [23,24,6,7]. The daily intake (μg/kg*day) was calculated based on these assumptions:

1) The human weight is 50 kg (some references set 60 kg) and

2) The human intake from spices per day is 20 g.

The daily intake (μg/kg*day) = heavy metal concentration in spice × 20/1000 /50.

Table 7: Averages of (Cd, Pb, Ni) concentrations (mg/kg) found in collected samples, calculated Daily Intake and Provisional Tolerable Weekly Intake μg/kg body weight

	Cd				Pb				Ni			
	Average	DI	WI	% from PTWI	Average	DI	WI	% from PTWI	Average	DI	WI	% from PTWI
Coriander	0.06	0.03	0.18	2.5	0.68	0.27	1.89	7.56	1.05	0.42	2.93	8.36
Nutmeg	0.06	0.02	0.15	2.2	0.43	0.17	1.20	4.78	1.11	0.44	3.11	8.88
Cardamom	0.10	0.04	0.27	3.8	0.25	0.10	0.70	2.79	3.11	1.24	8.71	24.87
Black pepper	0.14	0.06	0.39	5.6	0.20	0.08	0.56	2.24	0.04	0.02	0.11	0.31
Pimento (Jamaica pepper)	0.08	0.03	0.23	3.3	0.42	0.17	1.17	4.68	0.97	0.39	2.70	7.73
Cloves	0.17	0.07	0.47	6.8	0.12	0.05	0.35	1.39	3.21	1.29	9.00	25.71
Cinnamon	0.08	0.03	0.23	3.3	0.35	0.14	0.97	3.90	1.66	0.67	4.66	13.31
Mustard	0.07	0.03	0.20	2.9	0.02	0.01	0.04	0.17	0.79	0.32	2.22	6.34
Average= mg/kg, DI=Daily Intake (μg/kg*day), WI= Weekly Intake μg/kg body weight, PTWI= Provisional Tolerable Weekly Intake μg/kg body weight												
PTWI for Pb is 25 μg/kg, PTWI for Cd is 7 μg/kg, PTWI for Ni is 35 μg/kg.												

Table 7 shows an estimate for each heavy metal controlled in the studied samples. The estimated daily intake of Pb, Cd and Ni in this study is below that recommended by the JECFA-Committee (Joint Expert Committee for Food Additives and Contaminants FAO/WHO) which has set a limit for heavy metal intake based on body weight for an average adult (60 kg body weight). Based on the above concentrations, if a person consumes cardamom or cloves from the studied spices, he/she will then ingest an amount of nickel about 25% of PTWI (Provisional Tolerable Weekly Intake). Cinnamon has the second array with 13 % of PTWI. Other spices have less than 10 %. For cadmium and lead, the estimated PTWI was under 10 %.

The Consumption of spices with foods may be in a part of 1-2 %. In this case 2 percent of food contributes to up to 25 percent of PTWI. These amounts may be hazardous if the other food component resources are not below the permissible levels. Thus, it could be concluded that importing of these spices is likely to be a health hazard to the consumer, and many arrangements may be taken to control the origin and resources of imported foods.

Conclusion:

The present study provides useful guide in monitoring and controlling imported foodstuffs (such as spices) which must be taken into consideration to avoid the heavy metal contamination and its toxic effects. It is therefore suggested that regular monitoring of heavy metal levels in imported foodstuffs is essential in order to evaluate the potential risk associated with their consumption by humans.

On the other hand, care should be taken to select carefully the origin of our foodstuffs; enhance better coordination in controlling the trading system to improve food safety standards; increase awareness of consumers and policy makers to the dangers of heavy metal contamination in the food intake. Moreover, preventive measures should be enforced by the Syrian Custom Guard. These results can also be used to estimate PTDI as well as PTWI.

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