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Full Length Research Paper

Carbamate pesticide residues analysis of potato tuber samples using high-performance liquid chromatography (HPLC)

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Monitoring of pesticides is conducted globally to assess the environmental load of their residues. However, carbamate pesticides are among the most common used pesticides in potato protection in Egypt. Here, nine potato tuber samples were randomly collected from Assiut, Elminia, Kalubia, Cairo, and Giza cities in Egypt to detect the contamination of some carbamate residues using quick, easy, cheap, effective, rugged, and safe (QuEChERS) method. Three methods were used in this study, viz., washed, washed with salted, and peeled. The results indicated that concentrations of kresoxim and thiodicarb residues were higher in all potato samples than other pesticides studied. However, the highest significant value was found in Assiut city (Alzahraa market) for both pesticides (0.18 and 0.038 mg/kg, respectively). Furthermore, peeled method was found to be the most effective method in reducing the carbamate pesticide residues. Thus, further investigation should be done to figure out the potential methods of reducing carbamate pesticide residues in vegetables and strict regulation should be applied in using pesticides.

Key words: Carbamate, quick, easy, cheap, effective, rugged, and safe (QuEChERS), pesticide residues, high-performance liquid chromatography (HPLC), Potato tuber.

INTRODUCTION

Vegetables are considered the most important ingredients of the human diet for health maintenance and disease prevention in developing countries. For example, the total Indian meal constitutes about 150 to 250 g of vegetables per person per day (Mukherjee and Gopal, 2003). Moreover, *Solanum tuberosum* (potato) is the largest horticultural export crop of Egypt and it is documented that in the year 2000, total value of Egyptian potato exports was $US 7.7 million (Kabeil et al., 2008). In order to meet the huge demand, pesticides are widely applied to reduce heavy pest infestation, improve quality, increase yield, and extend the storage life of crops (Fernndez-Alba and Garca-Reyes, 2008). However, the results of using heavy application of pesticides on vegetables have resulted in pesticide residues above their respective Maximum Residue Limits (MRLs) which

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Table 1. Random markets from Egypt which the samples obtained from.

Market	City
Alzahraa	Assiut
Abo-Teg	Assiut
Naela Khatoon	Assiut
Alwelidia	Assiut
Der-Mwas	Elminia
Shobra-Elkhema	Kalubia
Elmaadi	Cairo
Eldokki	Giza
Elharam	Giza

may pose health hazards to consumers (Agnihotri, 1999; Kalara, 2003; Taneja, 2005; Mukherjee and Gopal, 2003). The MRLs, limits the amount of residues that can be legally present in foods. Unfortunately, not all farmers follow the legal practice and due to the tremendous number of pesticides and crops in production, a number of analytical methods was designed to determine multiple pesticide residues (Food and Drug Administration, 1999; Luke et al., 1975). Thus, analysis of pesticide residue in foods, especially vegetable, becomes an essential requirement for consumers, producers, and food quality control authorities (Ashutosh et al., 2011).

In 2002, the Quick, Easy, Cheap, Effective, Rugged, and Safe (QuEChERS) method for pesticide residue analysis was introduced by Anastassiades et al. (2002), which provides high quality results in a fast, easy, an inexpensive approach. Follow-up studies have further validated the method for more than 229 pesticides (Lehotay et al., 2005). The most common technique in modern multi-residue target pesticide analysis is the High Performance Liquid Chromatography (HPLC). In this study, we analyze the carbamate pesticide residues in potato tuber samples from different markets in different cities in Egypt.

MATERIALS AND METHODS

Samples

A total of 9 samples were collected randomly from nine local markets in five cities in Egypt (Table 1).

Sample preparation

Not less than 3 g of potato tubers were taken for each sample in polyethylene bags labeled by the name of the market and city, and then transferred immediately to the laboratory. Samples were completely homogenized then divided into three portions consisting of 1 kg each. Samples homogenization was done following the guidelines of Codex Guide vol.2-section 4, Anonymous (1993) as follows:

(1) 1 kg sample of potato tuber was completely homogenized, three replicates of 100 g each were taken, two for extraction and the third was kept in a deep freezer at -20°C. Extraction was carried out as soon as possible.

(2) The samples were comminuted, and 10 g of each sample was then placed into 50 ml polyethylene tube. Samples were extracted and cleaned up immediately after sampling using QuEChERS methodology (Anastassiades et al., 2002). 15 ml of acetonitrile was added into each tube. The samples were well shaken using a vortex mixer at maximum speed. Afterwards, 6 g of anhydrous magnesium sulfate and 1.5 g of sodium chloride were added, then extracted by shaking vigorously on vortex for 5 m and centrifuged for 10 m at 4,000 rpm. An aliquot of 4 ml was transferred from the supernatant to a new clean 15 ml centrifuge tube containing 100 mg PSA and 600 mg anhydrous magnesium sulfate. The samples were again vortexed for 3 m and then centrifuged for 10 m at 4,000 rpm. Sodium Chloride saturated solution (35%) was used for washed and slated method. Peeled method was done by the use of a sharp knife; the samples were taken through the whole samples preparation mentioned above. In general, each sample was conducted in three replicates.

Table 2. The average recovery of carbamate pesticides in potato tuber samples.

Pesticide	Average recovery (%)
Kresoxim-ethyl	98.11
Furathiocarb	84.35
Mexacrbate	89.26
Fenoxycarb	95.21
Vernolate	91.23
Aldicarb	88.21
Thiodicarb	87.65
Methomyl	88.65
Propoxur	78.49
Bendiocarb	89.30
Carbofurn	84.16
Ethofumesate	91.24
Chlorufum	95.24
Methiocarb	92.13
Pirimicarb	88.46
Carbaryl	87.23
promocarb	84.23

Pesticides detected

Seventeen carbamate pesticides were studied for identification and quantification, the detected residues include: Kresoxim-ethyl, Furathiocarb, Mexacrbate, Fenoxycarb, Vernolate, Aldicarb, Thiodicarb, Methomyl, Propoxur, Bendiocarb, Carbofurn, Ethofumesate, Chlorufum, Methiocarb, Pirimicarb, Carbaryl and promocarb. The average recovery percentage of pesticides for 3 spiked levels (0.05, 0.01, and 0.001mg/kg) in Potato tuber samples were conducted (Table 2).

HPLC

An Agilent technology 1260 HPLC UV-DAD (Diode Array Detector) system was used. It contained a binary pump, a degasser, column

Table 3. Carbamate pesticides residue analysis in potato tuber samples.

Market	Pesticide found	MRL (mg/kg)	Unwashed	Washed	Washed and salted	Peeled
Alzahraa	Kresoxim	0.05	0.18	0.04	0.01	0.007
	Thiodicarb	0.02	0.038	0.012	0.008	ND
Abo-Teg	Kresoxim	0.05	0.02	0.007	0.002	ND
Niela Khatoon	Thiodicarb	0.02	0.008	ND	ND	ND

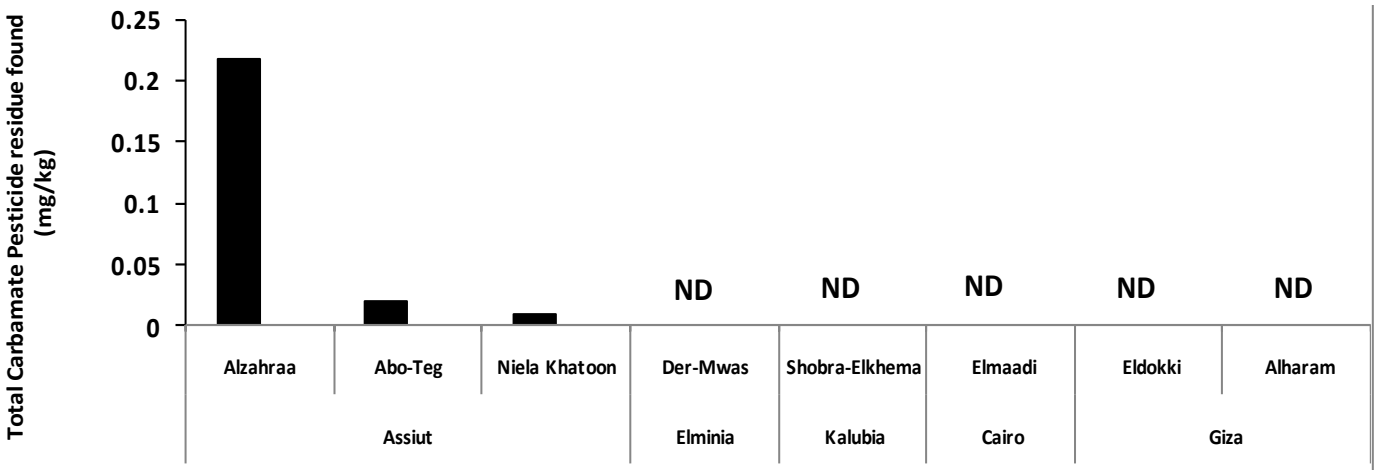

Figure 1. Total carbamate pesticides residue found in random markets.

thermostat and an autosampler. A reverse-phase Agilent Zorbax SB-C18 analytical column of 250 × 4.6 mm internal diameter (ID) and 5 µm particle size. Deionized water containing 0.1% formic acid (mobile phase component A) and acetonitrile (component B) were employed for the gradient program, which started with 20% B for 3 m and was linearly increased to 100% B in 27 m (held for 3 m). The column was then re-equilibrated for 12 m back to 20% B. Thus, the total run time took 45 m. The flow rate was constant at 0.6 ml/m, and injection volume was 10 µl.

All organic solvents were of HPLC grade and supplied by Merck, USA. Primary and secondary amine (PSA, 40 lm Bondesil) was purchased from Supelco (Supelco, Bellefonte, PA, USA). Anhydrous magnesium sulfate was of analytical grade, purchased from Merck, USA, and was activated by heating at 250°C for 4 h in the oven before use and kept in desiccators. A stock standard solution (100 lg/ml) was prepared with methanol and stored at -20°C. The standard working solutions were prepared from stock solution by serial dilution with methanol at 0.01, 0.05, 0.1, 0.2, 0.5, 1.0, and 2.0 lg/ml and were stored at 4°C before use.

RESULTS AND DISCUSSION

The carbamate pesticide residues analysis found in Potato tuber samples results are shown in Table 3. The highest total carbamate pesticides residue found in Assiut city whereas was not detectable (ND) at the rest of the cities (Figure 1). However, Alzahraa market was considered the highest carbamate pesticides residue found (Figure 1). Two carbamate pesticides, Kresoxim and Thiodicarb were found in Alzahraa market and the values were 0.18 and 0.038 mg/kg respectively, which is higher than the MRL. Peeled method was considered the best method in reducing the two carbamate pesticides by 96% and not detectable respectively (Figure 2). Furthermore, Abo-Teg market was found to have the second highest carbamate pesticide residue (Figure 1), one carbamate was found, Kresoxim, and the value was 0.02 mg/kg.

However, after using three different methods to reduce the Kresoxim, peeled method was the best method in reducing the pesticide (Figure 3). Niela Khatoon market had the lowest carbamate pesticide residue found in Assiut city (Figure 1), Thiodicarb value was 0.008 mg/kg and washed with salted water, and peeled methods were the best methods in reducing the residue of the pesticide. In total, the pesticide residues that are found in vegetables become a global phenomenon. A similar study reported that, the residue of carbamate pesticides in Potato tuber samples in Egypt and abroad, agrees with this study (Abd El Rahman, 2005; Mansour et al., 2009; Wang et al., 2008; Quinetro et al., 2008).

However, the highest value of the carbamate pesticide residue was found in the samples regardless of the heavy use of the pesticides and the persistence of these

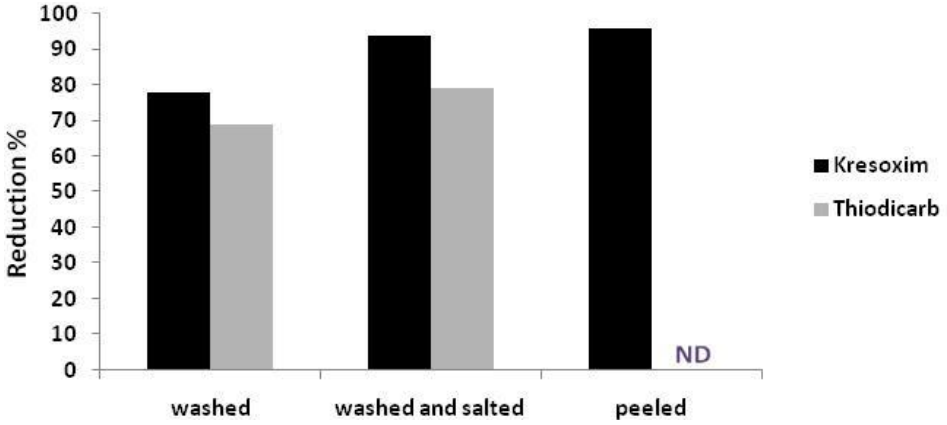

Figure 2. Reduction percentage of two carbamate pesticides residue found after the three methods were used in Alzahraa, Assiut city.

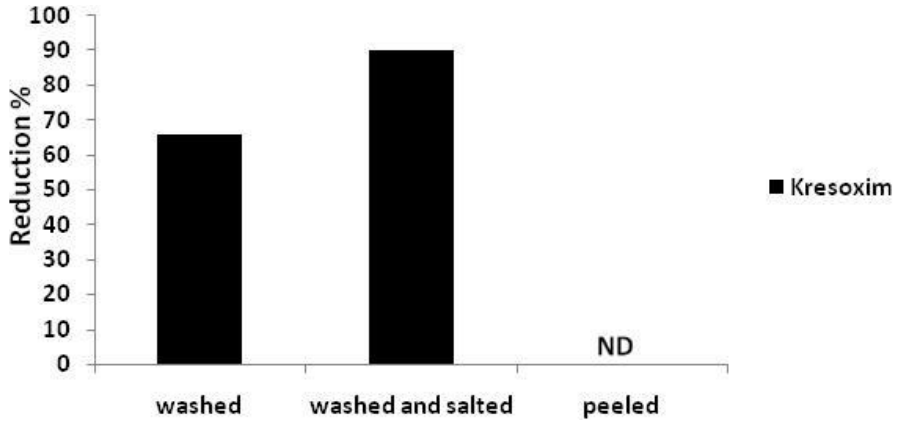

Figure 3. Reduction percentage of one carbamate pesticide residue found after used the three methods in Abo-Teg, Assiut city.

pesticides which indicated that not all farmers follow the legal practices and due to tremendous number of pesticides and crops in production, not all farmers send the production to analyze the residues using appropriate analytical methods which had been designed to determine multiple pesticide residues.

However, the results show no product can be consumed right after it is purchased from the market and should be processed using suitable method to clean up from the pesticide residues. Furthermore, a periodical monitoring of carbamate pesticides residue in vegetables and other foods are the recent need for the consumers as well as authorities of food quality control not only in Egypt but all over the world.

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Conflict of Interests

The author(s) have not declared any conflict of interests.

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