

Determination of Pesticide Residues in Most Commonly Consumed Leafy Vegetables in Riyadh Region (Al-Kharej Province)

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Abstract. A total of 81 samples of various leafy vegetables viz., Rocket (*Eruca sativa*), Lettuce (*Lactuca sativa*), Coriander (*Coriandrum sativum*), Corchorus (*Corchorus olitorius*), Parsley (*Petroselinum crispum*), Spinach (*Spinacia oleracea*), Radish (*Raphanus sativus*), Dill (*Anethum graveolens*), Mint (*Mentha sachalinensis*) and Green onions (*Allium cepa*) were collected from 20 different areas in Riyadh region of the Kingdom of Saudi Arabia. Liquid-Liquid Extraction of extraction partitioning method and confirmation by gas chromatograph-mass spectrometer for the presence of various pesticide residues was performed. Pesticide residues were detected in all the collected samples with varying concentrations. A variety of pesticides including organochlorines, organophosphates, pyrethroids, carbamates, herbicides, acaricides and insecticides have been identified in the samples. While the residue levels were low for most pesticides, higher levels of chlorpyrifos (0.123 mg/kg and resmethrin (0.1 mg/kg) were found in Rocket, carbaryl (0.92 mg/kg) in spinach and corchorus (0.116 mg/kg), permethrin (total cis + trans permethrin) in lettuce (0.103 mg/kg) and coriander (0.126 mg/kg), and cypermethrin in parsley (0.126 ppm). The highest maximum residue limit (MRL) values of pesticide residues on leafy vegetables of 0.10 mg/kg were observed for carbaryl in Spinach and Corchorus, whereas in Parsley an MRL of 0.5 mg/kg was found. The current research shows that in some leafy vegetables, higher doses of pesticides were found.

Keywords: Pesticides, residues, leafy vegetables, Gas Chromatography Mass Spect. Saudi Arabia, Riyadh.

1. Introduction

The use of various pesticides in agricultural practices has significantly enhanced the quality and quantity of vegetables and fruits worldwide. However, excessive use of pesticides has caused an increase in contamination of the environment in addition to causing many short and long term health complications some reversible and irreversible (Kaloyanova and El-Batawi, 1991). The detection of large levels of pesticide residues in vegetables and fruits is a serious threat to human and animal health (Damalas and Eleftherohorinos, 2011). Minimising of exposure

to environmental toxicants like pesticides especially in consumables remains one of the major objectives of the Ministry of Health in Saudi Arabia. Pesticides are being used worldwide to protect agricultural crops, insects at home and in horticulture. Pesticides belonging to various classes such as organophosphates, organochlorines, pyrethroids and carbamates apart from other herbicides, acaricides, insecticides are used either singly or in combinations of variable concentrations can successfully cater to controlling a wide variety of pests (Sobus *et al.*, 2015). At the same time, at

least some of the pesticides are not easily biodegradable and/or remain on food products even after processing and thus make into the food chain and ecological systems.

Therefore, measurement of pesticide residues in consumable foods is an essential prerogative in maintaining health standards and thus to minimize the harmful impact of pesticides. The current study thus evaluates the pesticide residues in most commonly used leafy vegetables in Riyadh region of Saudi Arabia. Currently the wide use of pesticides in Saudi Arabia can not only lead to extensive pollution of the environment but also constitutes a potential and/or deliberate risk to human health (El-Saeid, and Shaht 2000; EL-Saeid, and Khan (2015), EL-Saeid and Selim (2013), Selim *et al.*, (2011); Khaled *et al.*, 2011). Some investigations reported that the persistence of different pesticides left residual amounts in vegetables with different residue levels (Coats and Yamamoto, 2003; Ripley *et al.*, 2003). The analysis of pesticide residues in foods especially fresh edible fruits and vegetables is very important, because of the potential adverse health and toxic effects of these compounds due to consumption of contaminated foods depend at maximum residue limits (MRLs) and Acceptable Daily Intake (ADI) (FAO/WHO, 1998). It has been reported that pesticide residues in fruits and vegetables can cause severe impact to human health including liver damage, interfering in enzyme production, interaction with drugs and hormones, gastrointestinal intoxication, immunotoxicity and neurotoxicity through cholinesterase inhibition. Therefore, investigation of pesticide residues in vegetables and fruits have become an essential prerequisite for appropriate food quality control procedures.

The analyses of pesticide residues are routinely carried out by means of multi-residue methods based on homogenization of the sample with an appropriate solvent, separation

of the liquid portion of the sample from the insoluble material, purification and cleansing by florisil column, followed by the final chromatographic determination procedure (Torres *et al.*, 1996; Albero *et al.*, 2001; Tadeo *et al.* 2000). The organic solvents commonly used to extract pesticide residues in fresh fruit and vegetables are acetonitrile, Petroleum Ether (PE) and diethyl ether. An extensive clean-up of organic extraction is necessary to reduce the adverse effects related to the quantification of residues, such as the masking of residue peaks by co-eluted matrix component, the occurrence of false positives and/or inaccurate quantitation (Colume *et al.*, 2000; Navarro *et al.*, 2000). To detect the pesticide accurately with high specificity and precision, we have used a modified method for sample preparation and extraction (Schenck and Hobbs, 2004) followed by detection using gas chromatograph-mass spectrophotometer (Fernandez *et al.*, 2000; Rosa *et al.*, 2009; Miguel *et al.*, 2011). The aim of the current study was to monitor the levels of various pesticide residues in some commonly used fresh leafy vegetables collected from various regions of Riyadh, Kingdom of Saudi Arabia. In addition, the Maximum Residue limits (MRL) were analysed in order to compare with the International MRLs.

2. Materials and Methods

2.1 Sample Collection

Samples of leafy vegetables were collected in sterile polythene bags from some farms in selected areas in Riyadh Region (Al-Kharej Province). These samples were transported to the laboratory where they were analysed immediately or stored at 4°C and analysed within 24 hours.

2.2 Reagents and Equipment

All the following pesticides standards were obtained from (Riedel de Haen and Supelco). 1mg/ml stock solution of each was

prepared by dissolving 20mg of the pure analytical standard in acetone. A single composite standard solution was prepared by diluting with acetone according to limit of detection (LOD). All standard solutions were stored in glass-stoppered flasks at 4°C. Mixed compound calibration solutions were prepared in acetone and they were used as spiking solution. Solvents (residue analysis grade) used were acetone, acetonitrile, petroleum ether and other reagents such as sodium chloride and anhydrous sodium sulphate, florisil 60-100 mesh for residue analysis were also purchased from (Fluka). The florisil and anhydrous sodium sulphate was activated at 100 °C overnight and stored in 500 ml glass flask with glass stoppers and stored in oven at 100 °C. The equipment used were a high-speed blender with a stainless steel jar (Waring, USA), a separation funnel shaker (GFL, Germany), a rotavapor, R 215 with cooler circulator chiller B-740 (Buchii, Switzerland), Buchner funnel and chromatographic tubes with Teflon stopcocks and coarse fritted glass (Agilent, USA) and syringes (Hamilton Bonadus AG, Switzerland). All glassware were rinsed thoroughly using deionization water, then washed with acetone and dried in oven (100-130 °C) over night.

2.3 Extraction and Partitioning Method

The pieces leafy vegetable samples (100g) were placed in a stainless steel 1L jar

and 200ml of acetonitrile and 10g of celite will be extracted. The mixture was filtered through Buchner funnel fitted with shark-skin filter paper into a 500ml suction flask. An aliquot of organic matter was transferred to a 1L separator funnel and 100ml of PE was added. The mixture was vigorously shaken for 1 – 2 minutes and then 100ml of saturated NaCl solution and 600ml water was added. The mixture was vigorously mixed and the separator funnel was held in a horizontal position for a few minutes. The aqueous layer was discarded and the solvent layer was washed with twice the amount of distilled water and this washed layer was transferred into a 100ml beaker and washed with 15g anhydrous sodium sulphate. Finally, the extract was concentrated to 5ml volume and transferred directly to a florisil column (Selim *et al.*, 2011).

2.4 Recovery Experiment

For recovery studies, 100 g homogenized sample was spiked prior to determination procedure by addition of a mixture of standard pesticides solution 0.1µg/ml of each compound. Spiked samples were left to stand for 30 min. to allow pesticide to absorb into the sample. Samples were extracted according to the method described above. The recoveries % (Table 1) and limit of detection (LOD) (Table 2) data for tested pesticides are calculated.

Table 1. Recovery % and limits of detection (LOD µg/kg) for tested pesticides in leafy Vegetable Samples.

Pesticides	Spiking level (µg/g).	LOD (µg/g)	Recovery %
Mevinphos	0.10	0.090	89.6
Ethoprophos	0.10	0.050	93.4
Lindan	0.10	0.010	101.3
Iprobenfos	0.10	0.040	104.7
Chlorpyrifos	0.10	0.030	98.6
Carbaryl	0.10	0.030	98.5
Linuron	0.10	0.040	93.8

Endosulfan	0.10	0.090	48.9
Resmethrin	0.10	0.030	88.3
Phosmet	0.10	0.030	88.6
Fenoxycarb	0.10	0.070	89.8
Tetramethrin	0.10	0.020	85.2
Amitraz	0.10	0.030	92.9
Azenophos-ethyl	0.10	0.030	94.6
Permethrin	0.10	0.020	98.8
Cyfluthrin	0.10	0.040	89.7
Cypermethrin	0.10	0.030	93.4
Deltamethrin	0.10	0.050	101.3

Table 2. Averages of pesticide residues, Standards Deviation and number of samples of leafy vegetables. Scientific Name.

Common Name	Scientific Name	No. of Samples	Pesticide Name	Averages ($\mu\text{g/g}$)	St. D
Rocket	<i>Eruca sativa</i>	8	Cyfluthrin	0.013	0.002
			Lindane	0.001	0.001
			Iprobenfos	0.023	0.002
			Chloropyrifos	0.123	0.003
			Resmethrin	0.101	0.001
Lettuce	<i>Lactuca sativa</i>	9	Mevinphos	0.001	0.002
			Deltamethrin	0.048	0.004
			Tetramethrin	0.024	0.003
			Azinophos-ethyl	0.008	0.001
			Permethrin	0.104	0.006
Coriander	<i>Coriandrum sativum</i>	8	Resmethrin	0.014	0.006
			Permethrin	0.126	0.019
			Lindane	0.005	0.003
			Linuron	0.064	0.018
Corchorus	<i>Corchorus olitorius</i>	8	Endosulfan	0.013	0.001
			Carbaryl	0.117	0.002
			Permethrin	0.003	0.001
			Chloropyrifos	0.006	0.004
Parsley	<i>Petroselinum crispum</i>	8	Amitraz	0.002	0.002
			Cyfluthrin	0.009	0.002
			Cypermethrin	0.168	0.038
			Ethoprophos	0.001	0.001
Spinach	<i>Spinacia oleracea</i>	8	Carbaryl	0.920	0.048
			Endosulfan	0.003	0.002
			Linuron	0.005	0.004
			Phosmet	0.012	0.001
Mint	<i>Mentha sachalinensis</i>	8	Fenoxycarb	0.001	0.001
			Permethrin	0.075	0.004
Dill	<i>Anethum graveolens</i>	8	Cyfluthrin	0.008	0.001
			Resmethrin	0.003	0.002
Radish	<i>Raphanus sativus</i>	8	Endosulfan	0.004	0.002
Onion	<i>Allium cepa</i>	8	Permethrin	0.002	0.002

2.5 Pesticide Residues Measurement by Chromatographic Analysis

Gas chromatograph-mass spectrometer (Aglient model 6890N) coupled with quadrupole mass spectrometer (model 5975B) with a GC column HP-5MS 5% phenyl - 95% methyl siloxane, 30m x 0.25mm id x 0.25 µm film thickness was used. GC operating conditions were splitless injection, injector temperature 250 °C, helium carrier gas (99.9999 purity) at flow rate 0.9 ml/min with column head pressure 7.4 psi, oven temperature from 70 °C (2 min hold), then raised to 130 °C at the rate (25 °C/min) afterwards raised to 220 °C at (2 °C/min) and then raised to 280 °C at (10 °C/min) and eventually (4.6 min hold). The sample was injected in splitless modes. The MS system was routinely set in selective ion monitoring (SIM) mode and each compound was quantified based on peak area using one target and one or two qualifier ion. Mass spectrometer parameter was set as follows: electron impact ionization mode with 70 eV electron energy, scan mass range 100-400 at 0.62 sec/cycle. Ion source temperature 230 °C, MS quad temperature 150 °C, EM voltage 1450 and solvent delay 4 min. An appropriate aliquot (2µl) of samples was injected in Gas Chromatography Mass spectrometry under the mentioned conditions and then pesticides residues were identified by comparison of retention time values with reference standard. Confirmation of results was performed using selected ion monitoring (SIM) mode, one is the quantifier and the qualifying ion (Selim *et al.*, 2011; Tuija *et al.*, 2007).

3. Results

3.1 Concentration of Pesticide Residues in Leafy Vegetables

Pesticide residues in this study were identified in all the collected samples with varying concentrations. Mixtures of pesticides

were identified in most leafy vegetables except Radish and green onion where only endosulfan and permethrin were used respectively. Pesticides belonging to all major categories such as organochlorines, organophosphates, pyrethroids, carbamates, herbicides, acaricides and insecticides have been identified in the samples. The residue levels for most leafy vegetables was less (<0.05 mg/kg) but higher levels of chlorpyrifos (0.123 mg/kg) and resmethrin (0.1 mg/kg) were found in Rocket. Carbaryl (0.92 mg/kg) in spinach and corchorus (0.116 mg/kg), permethrin in lettuce (0.103 mg/kg) and coriander (0.126 mg/kg), and cypermethrin in parsley (0.126 mg/kg) (Fig. 1 and 2).

When the percentage of pesticide use was evaluated, it has been found that approximately 50% pesticides used belonged to carbamate class (carbaryl) followed by 36% pyrethroids and 14% others including organophosphates, acaricides, herbicides, and insecticides (Fig. 3).

3.2 Limit of Detection of Pesticide Residues Identified in the Current Study

The limit of detection (LOD) for the pesticides used in the current study was evaluated. The results showed that the highest LOD was for Cypermethrin (0.1) followed by Mevinphos (0.09) and Endosulfan (0.09). An LOD value of 0.07 was found for Fenoxycarb, 0.05 mg/kg for Deltamethrin and Ethoprophos. The LOD values for Cyfluthrin, Iprobenfos and Linuron was 0.04. Chlorpyrifos, Resmethrin, Azinophos-ethyl, Carbaryl, Amitraz and Phosmet had LOD values of 0.03. Rest of the pesticides had LOD values below 0.02 (Fig. 4).

3.3 Maximum Residue Limit of Pesticides Found in Leafy Vegetables

The highest maximum residue limit (MRL) values of pesticide residues on leafy vegetables of 10 were observed for carbaryl in Spinach and Corchorus, whereas in Parsley an MRL of 5 was

found for Cypermethrin. The MRL values 2 were found for Deltamethrin, Permethrin and

Endosulfan. The MRL values of most other pesticides were found to be 0.5 mg/kg (Fig. 5).

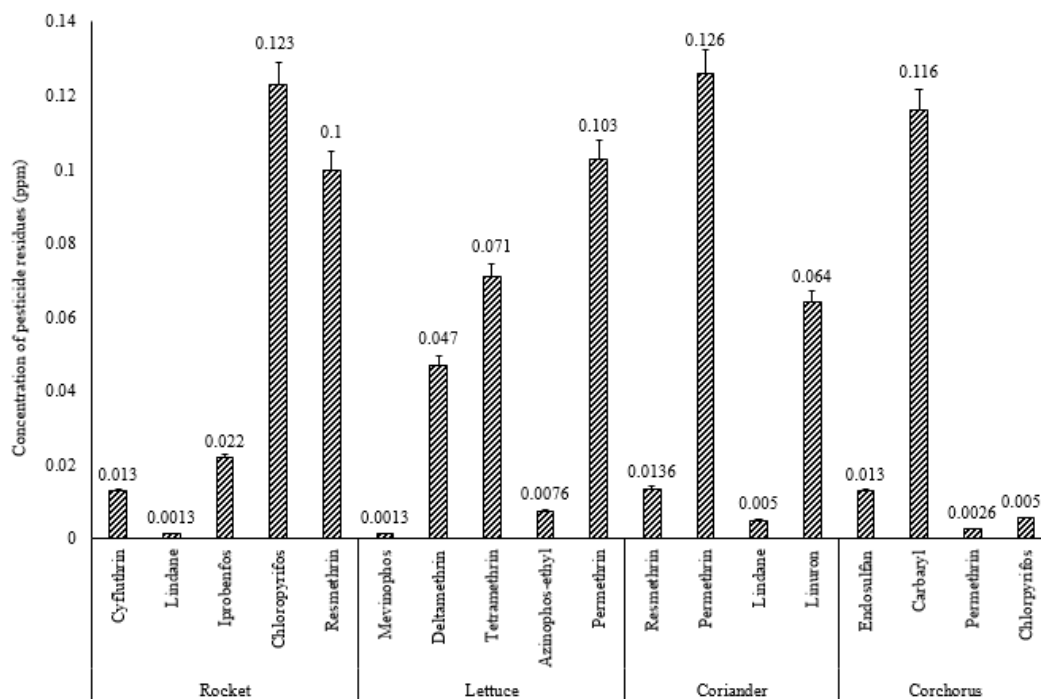


Fig. 1. Concentrations of various pesticide residues in Rocket, Lettuce, Coriander and Corchorus. Data are presented as mean \pm SD. All values are in ppm.

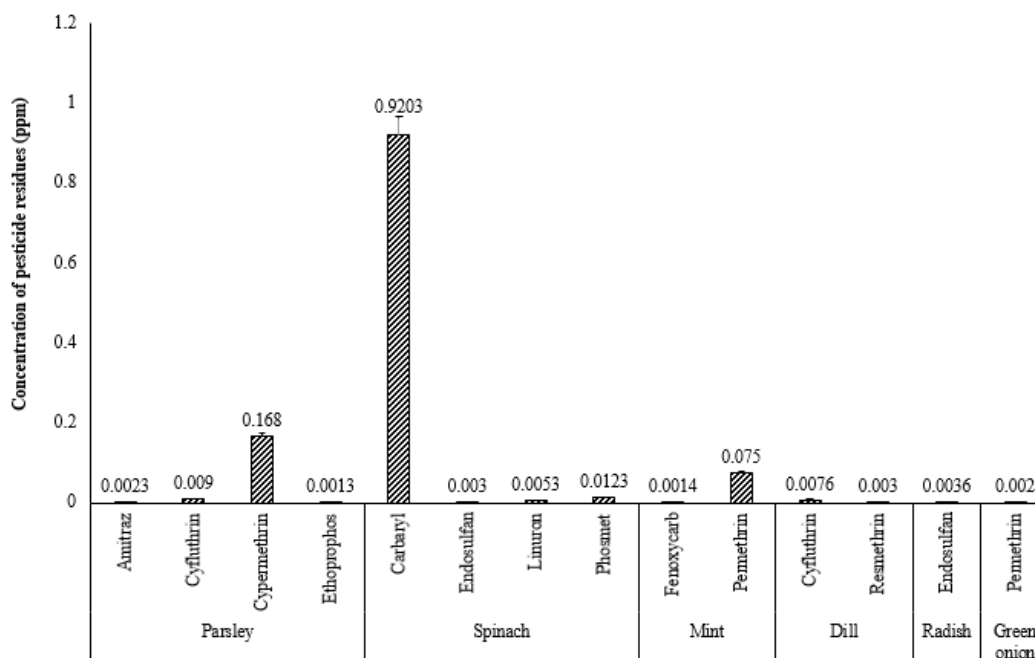


Fig. 2. Concentrations of various pesticide residues in Parsley, Spinach, Mint, Dill, Radish, and Green onion. Data are presented as mean \pm SD. All values are in ppm.

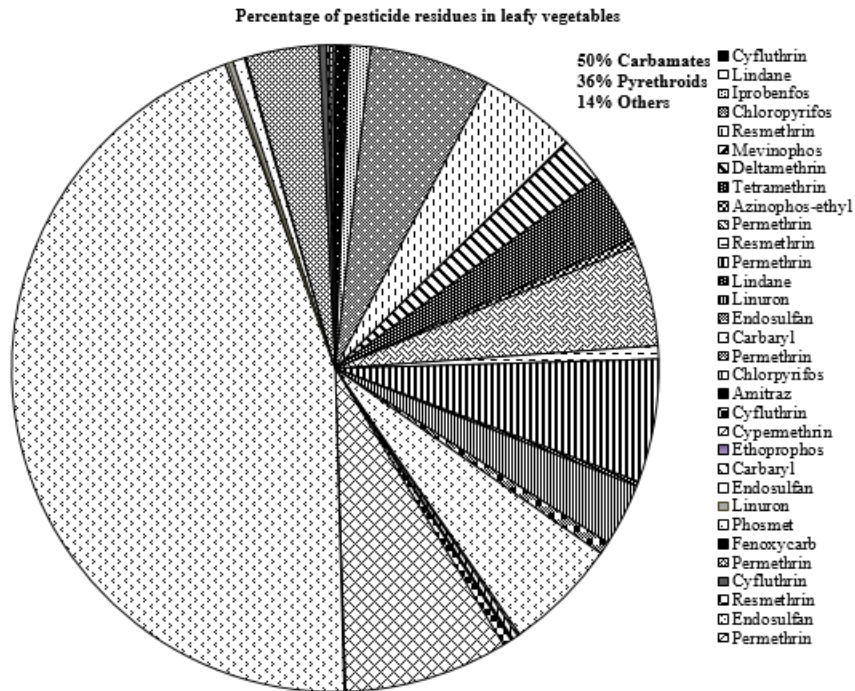


Fig. 3. Percentages of various pesticide residues in leafy vegetables investigated in the current study. The order of pesticide usage was carbamates (50%)>pyrethroids (36%)>others (14%). Others include organophosphate pesticides, insecticides, acaricides, and herbicides.

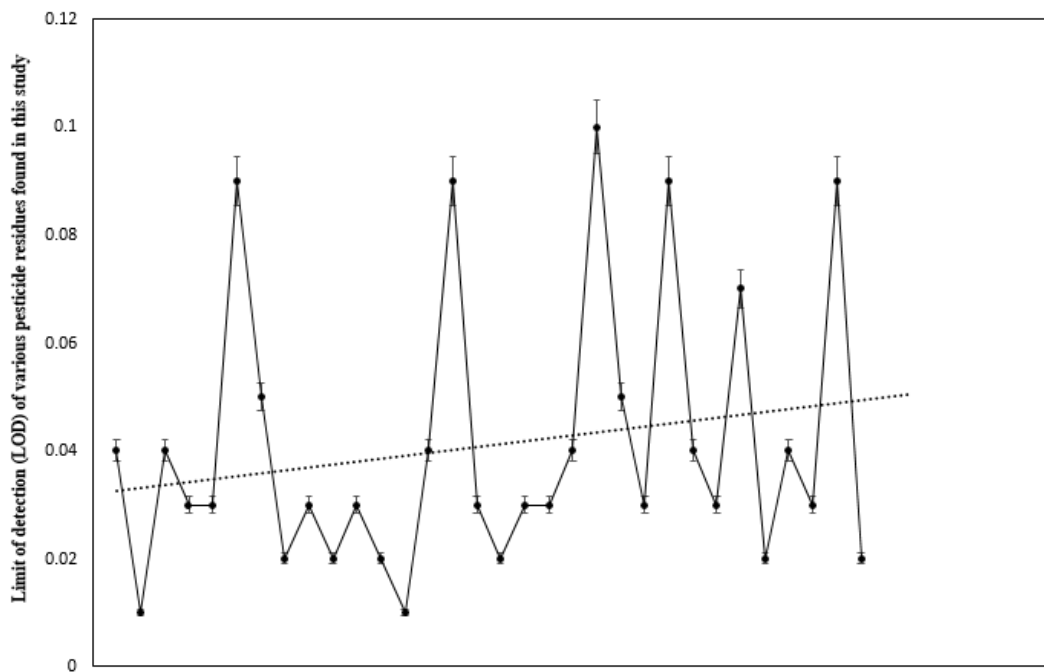


Fig. 4. The limit of detection (LOD) of various pesticide residues in leafy vegetables in the current study.

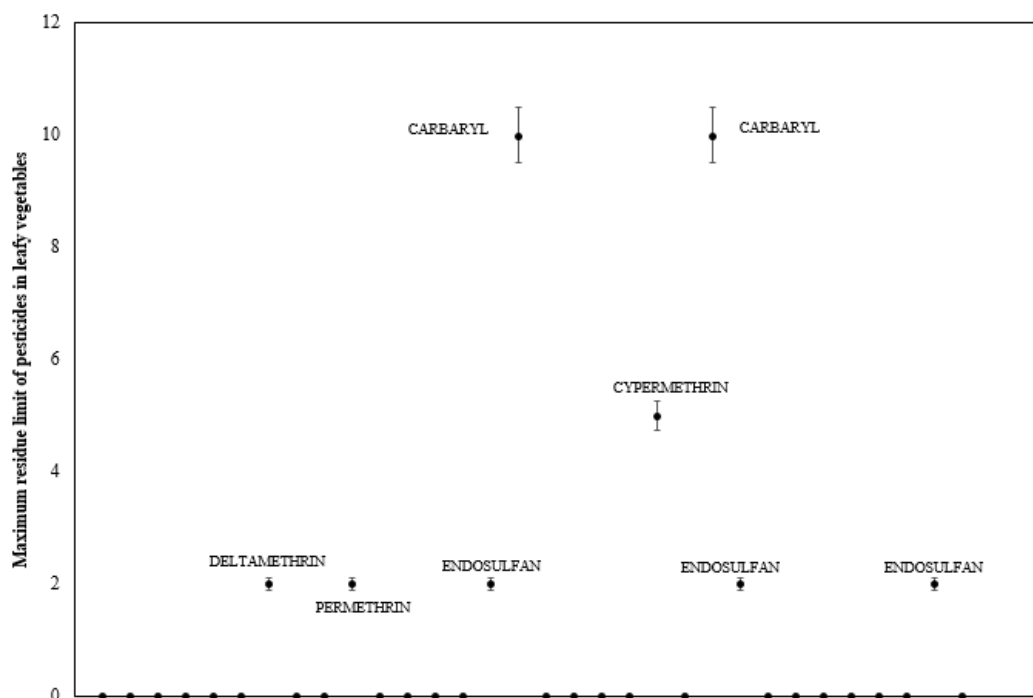


Fig. 5. Maximum residue level of various pesticide residues found in leafy vegetables in the current study. Carbaryl showed the highest MRL, followed by Cypermethrin, Deltamethrin, Endosulfan and Permethrin.

4. Discussion

A total 81 samples of leafy vegetables (Rocket, Lettuce, Coriander, Corchorus, Parsley, Spinach, Radish, Dill, Mint and Green onions) were collected from 20 different regions of Riyadh. All samples were processed and analysed using the GCMS system. Pesticide residues in this study were interestingly found in all the collected leafy vegetables. Almost in all cases, the farmers used a mixture of pesticides belonging to various chemical classes except in vegetables like Radish and green onion.

Health-based recommendations or guidelines are based on data obtained from toxicological studies on experimental animals, and sometimes on observations in man. In order to evaluate the acceptability of a proposed MRL, it is necessary to compare the dietary intake of pesticide residues, which is calculated on the basis of MRL, with the ADI.

The dietary intake is calculated by multiplying each MRL with the quantity of the corresponding diet component, followed by summation of the residue quantities obtained. It should be noted that the use of the MRL in the calculation of total intake might lead to a higher value than the actual intake, since the actual residue levels will often be lower than the recommended MRLs (Van Leeuwen, 1997).

Farmers used a mixture of organochlorines, organophosphates, pyrethroids, carbamates, herbicides, acaricides and insecticides for cultivation. However, most of the leafy vegetables showed pesticide residues of <0.05 ppm. In certain leafy vegetables very high levels of carbaryl, chlorpyrifos, resmethrin, permethrin and cypermethrin were found. Interestingly, pesticides which have been categorized as restricted use by the Ministry of Agriculture, Kingdom of Saudi Arabia eg: carbaryl, chlorpyrifos and endosulfan were

used on leafy vegetables. Majorly pesticides of the carbamate group were used (50%) followed by pyrethroids (36%) and others (14%). Most of the pesticides showed MRL values of 0, while, the highest MRL values observed were 10 for carbaryl and 5 for Cypermethrin. MRL values 2 were found for Deltamethrin, Permethrin and Endosulfan.

In the analysed samples 20 different pesticides were found at various concentrations. A combination of 5 pesticides were used for cultivation of Rocket and Lettuce. Four pesticides were used for cultivation of coriander, corchorus, parsley and spinach. The combination of two pesticides was used in dill and mint while only one pesticide each was used for cultivation of radish and green onions. The present study showed a high incidence of pesticide residues (mostly carbamates, organochlorines and pyrethroids) in analysed leafy vegetable samples.

In the current study we also used the Liquid-Liquid Extraction and GCMS method which helped in easy, simple and rapid extraction method in addition to high sensitivity detection reliable analysis for all tested pesticide residues (Yang *et al.*, 2012; Subhash *et al.*, 2014; Soparat *et al.*, 2015). The results obtained were in accordance to quality control requirements and all experiments were validated. These methods helped in quick analysis within a shorter time and allowed for efficient pesticide monitoring. The LOD values obtained were found to be highest for Cypermethrin (0.1), Mevinphos (0.09) and Endosulfan (0.09) followed by Fenoxycarb (0.07) and 0.05 mg/kg for Deltamethrin and Ethoprophos.

In the present study, the Liquid-Liquid Extraction method was used for sample preparation and GCMS analysis was established. This combination helped in

achieving highly sensitive results that are easily reproducible. The contamination levels of pesticide residues observed in this study on leafy vegetables can pose a serious threat to public health if proper washing or processing conditions are not applied. The high MRL values observed for carbaryl, pyrethroids and organochlorines observed in this study indicate that these pesticides may pose a possible public health problem. The results highlight the necessity for consistent monitoring for pesticide residues for these vegetables before being marketed to the public.

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تحديد متبقيات المبيدات الحشرية في الخضروات الورقية الأكثر استخدامًا في منطقة الرياض (محافظة الخرج)

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المستخلص. تم جمع ٨١ عينة من الخضروات الورقية المختلفة من ٢٠ موقعًا من منطقة الرياض بالمملكة العربية السعودية، وهي: جرجير، وخس، وكزبرة، وملوخية، وبقدونس، وسبانخ، وفجل، وشبت، ونعناع، والبصل الأخضر. تم إجراء الكشف لوجود متبقيات مختلفة من مبيدات الآفات بطريقة استخلاص السائل والتأكد بواسطة مطياف الكتلة الكروماتوجرافية الغازية. وجدت بقايا المبيدات في جميع العينات التي تم جمعها بتركيزات مختلفة، وقد تم تحديد مجموعة متنوعة من المبيدات الحشرية في العينات، وهي: الكلور العضوي، والفوسفات العضوي، والبيرثرويدات، والكاربامات، ومبيدات الأعشاب، والمبيدات الحشرية. ففي حين أن مستويات المتبقيات كانت منخفضة بالنسبة لمعظم المبيدات الحشرية، كانت المستويات الأعلى للكلوربيريفوس (٠,١٢٣، ملجم/كجم وريسيميثرين (٠,١ ملجم/كجم)) في الجرجير، والكارباريل (٠,٩٢ ملجم/كجم) في السبانخ و (٠,١١٦ ملجم/كجم) في الملوخية، والبيرميثرين (مجموع Cis + بيرميثرين غير المشبع) في الخس (٠,١٠٣ ملجم/كجم) والكزبرة (٠,١٢٦ ملجم/كجم)، والسايبرميثرين في البقدونس (٠,١٢٦ جزء في المليون). وقد لوحظت قيم الحد الأقصى لمتبقيات المبيدات (MRL) على الخضروات الورقية، والبالغ قدرها ٠,١٠ ملجم/كجم للكارباريل في السبانخ والملوخية، بينما وجد في البقدونس حد أقصى قدره ٠,٥ ملجم/كجم. ويظهر البحث الحالي وجود جرعات عالية من المبيدات الحشرية في بعض الخضروات الورقية.

كلمات مفتاحية: المبيدات الحشرية، المتبقيات، الخضروات الورقية، تحليل الغاز الكروماتوجرافي، السعودية، الرياض.

